# Lubricants and Lubrication

*Edited by Theo Mang and Wilfried Dresel*

*Lubricants and Lubrication. 2nd Ed.* Edited by Th. Mang and W. Dresel

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# Lubricants and Lubrication

*Edited by*
*Theo Mang and Wilfried Dresel*

Second, Completely Revised and Extended Edition

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## Preface, Foreword 2nd edition

Six years after the publication of “Lubricants and Lubrication”, its high acceptancy motivated the publisher and the editors to realize a second edition. The result is this largely revised and extended version.

The use of lubricants is as old as the history of mankind but the scientific analysis of lubrication, friction and wear as an aspect of tribology is relatively new. The reduction of friction along with the reduction or even avoidance of wear by the use of lubricants and lubrication technologies results in energy savings, the protection of resources and also fewer emissions. These benefits describe the economic and ecological importance of this field of work.

Only recently have lubricants begun to be viewed as functional elements in engineering and this group of substances are also attracting increasing attention from engineers.

This book offers chemists and engineers a clear inter-disciplinary introduction and orientation to all major lubricant applications. The book focuses not just on the various products but also on specific application engineering criteria.

The authors are internationally-recognized experts. All can draw on many years of experience in lubricant development and application.

This book offers the following readers a quick introduction to this field of work: the laboratory technician who has to monitor and evaluate lubricants; plant maintenance people for whom lubricants are an element in process technology; research and development people who have to deal with friction and wear; engineers who view lubricants as functional elements and as media which influence service life and increasingly safety and environmental protection officers who are responsible for workplace safety, an acceptable use of resources along with the reduction or avoidance of emissions and wastes.

Mannheim, December 2006

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## Abbreviations

| | |
|---|---|
| AAS | Atomic Absorption Spectrometry |
| ACEA | Association des Constructeurs Européens d'Automobiles<br>Association of the European Car Manufacturers |
| AD | Ashless Dispersant |
| AENOR | Asociación Espanola de Normalización y Certificación<br>Spanish Association for Normalization and Certification |
| AFNOR | Association Francaise de Normation<br>French Association for Normalization |
| AGMA | American Gear Manufacturers Association (USA) |
| API | American Petroleum Institute (USA) |
| ASTM | American Society of Testing Materials (USA) |
| ASTME | American Society of Tool and Manufacturing Engineers (USA) |
| ATF | Automatic Transmission Fluid |
| ATIEL | Association Technique de L'Industrie Européenne des Lubrifiants<br>Technical Association of the European Lubricant Industry |
| AW | Anti-Wear |
| AWT | Almen-Wieland-Test |
| BAM | Bundesanstalt für Materialprüfung (D)<br>Federal Institute for Materials Research and Testing |
| BOD | Biological Oxygen Demand |
| CAFE | Californian Act for Fuel Emission (USA) |
| CCEL | China Certification Committee for Environmental Labelling of products |
| CCMC | Committee of Common Market Automobile Constructors (EU) |
| CCS | Cold Cranking Simulator |
| CEC | Coordinating European Council for the development of performance tests for lubricants and engine fuels |
| CETOP | Comité Européen des Transmissions Oleohydrauliques et Pneumatiques European Oil Hydraulic and Pneumatic Committee |
| CFMS | Closed-Field Magnetron Sputtering |
| COD | Chemical Oxygen Demand |
| CONCAWE | The Oil Companies' Organization for the Conservation of Clean Air and Water in Europe |

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| | |
|---|---|
| CRC | Coordinating Research Council (EU) |
| CVD | Chemical Vapor Deposition |
| CVT | Constantly Variable Transmission |
| DD | Detergent and Dispersant |
| DGMK | Deutsche Wissenschaftliche Gesellschaft für Erdöl, Erdgas und Kohle German Scientific Society for Mineral Oil, Natural Gas and Coal |
| DIN | Deutsches Institut für Normung<br>German Institute for Normalization |
| DKA | Deutscher Koordinierungsausschuß im CEC<br>German Committee for Coordination in the CEC |
| DLC | Diamond-like amorphous Carbon |
| DSC | Differential Scanning Calorimetry |
| EBT | Electron Beam Texturing |
| ECP | Environmental Choice Program (CDN) |
| EHD | Elastohydrodynamic |
| EHEDG | European Hygienic Equipment Design Group |
| ELGI | European Lubricating Grease Institute |
| EN | European Norm |
| EP | Extreme Pressure |
| FCC | Fuel Catalytic Cracker |
| FDA | Food and Drug Administration (USA) |
| FM | Friction Modifier |
| FTMS | Federal Test Methods Standardization (USA) |
| FVA | Forschungsvereinigung Antriebstechnik (D)<br>Research Association for Drive Technology |
| FZG | Forschungsstelle für Zahnräder und Getriebebau (D)<br>Research Center for Toothed Wheel and Gearing Engineering |
| GC | Gas Chromatography |
| GfT | Gesellschaft für Tribologie (D)<br>Society for Tribology |
| GOST | Gossudarstwenny Obschtschessojusny Standart (former USSR)<br>State Standard (Governmental Union Standard) |
| HC | Hydrocracked |
| HD | Heavy Duty |
| HLB | Hydrophilic-Lyophilic Balance |
| HPDSC | High Pressure Differential Scanning Calorimetry |
| HPLC | High Pressure Liquid Chromatography |
| HRC | Rockwell C Hardness |
| HTHS | High Temperature High Shear |
| HVI | High Viscosity Index |
| IARC | International Agency for Research on Cancer |
| IBAD | Ion-Beam-Assisted Deposition |
| ICP | Inductively Coupled Plasma Atomic Emission |

| | |
|---|---|
| IFP | Institute Francais de Pétrole<br>French Institute of Petroleum |
| ILMA | Independent Lubricant Manufacturers Association (USA) |
| ILSAC | International Lubricant Standardization and Approval Committee |
| IP | Institute of Petroleum (UK) |
| ISO | International Standard Organisation |
| ISO VG | ISO Viscosity Grade |
| JEA | Japanese Environmental Association |
| JIS | Japanese Industrial Standard |
| LDF | Long Drain Field Test |
| LIMS | Laboratory Information and Management System |
| LVFA | Low Velocity Friction Apparatus |
| LVI | Low Viscosity Index |
| MAK | Maximale Arbeitsplatzkonzentration<br>Maximum Workplace Concentration |
| MIL | Military Standard (USA) |
| MLDW | Mobil Lube Dewaxing |
| MQL | Minimum Quantity Lubrication |
| MRV | Mini Rotary Viscosimeter |
| MS | Mass Spectroscopy |
| MSDW | Mobil Selective Dewaxing |
| N-D-M | (Refractive Index) n-Density-Molecular Weight |
| NLGI | National Lubricating Grease Institute (USA) |
| NMR | Nuclear Magnetic Resonance |
| OECD | Organization for Economic Cooperation and Development (EU) |
| OEM | Original Equipment Manufacturer |
| PCMO | Passenger Car Motor Oil |
| PEP | Passive Extreme Pressure |
| PPD | Pour Point Depressant |
| PTFE | Polytetrafluorethylene |
| PVD | Physical Vapor Deposition |
| RAL UZ | Reichsausschuß für Lieferbedingungen und Gütesicherung, Umweltzeichen (D)<br>Imperial Committee for Quality Control and Labelling, Environmental Symbol |
| RNT | Radionuclide Technique |
| RVT | Reichert-Verschleiß-Test<br>Reichert-Wear-Test |
| SAE | Society of Automotive Engineers (USA) |
| SCDSC | Sealed Capsule Differential Scanning Calorimetry |
| SHPD | Super High Performance Diesel |
| SRE | Standard Reference Elastomer |
| SRV | Schwing-Reibverschleiß Gerät (translatorisches Oszillations-Prüfgerät)<br>Translatory oscillation apparatus |

| | |
|---|---|
| STLE | Society of Tribologists and Lubrication Engineers |
| STOU | Super Tractor Oil Universal |
| SUS | Saybolt Universal Seconds |
| TAN | Total Acid Number |
| TBN | Total Base Number |
| TDA | Thermal Deasphalting |
| TEI | Thailand Environment Institute |
| TEWL | Transepidermal Water Loss |
| TG | Thermogravimetry |
| TGL | Technische Normen, Gütevorschriften und Lieferbedingungen (former DDR)<br>Technical Standards, Quality Specifications and Terms of Delivery |
| TLV | Threshold Limit Value |
| TOST | Turbine Oil Oxidation Stabilty Test |
| TRIP | Transformation Induced Plasticity |
| TRK | Technische Richtkonzentration (D)<br>Technical Guideline Concentration |
| TSSI | Temporary Shear Stability Index |
| UEIL | Union Européenne des Indépendents en Lubrifiants<br>European Union of Independent Lubricant Manufacturers |
| UHPD | Ultra High Performance Diesel |
| UHVI | Ultra High Viscosity Index |
| UNITI | Bundesverband Mittelständischer Mineralölunternehmen (D)<br>Federal Association of the Middle-class Oil Companies |
| USDA | United States Department for Agriculture |
| UTTO | Universal Tractor Transmission Oil |
| VAMIL | Regeling Willekeurig Afschrijving Milieu-Investeringen (NL)<br>Regulation of discretion deductions for investments into the environment |
| VCI | Vapor Phase (Volatile) Corrosion Inhibitor |
| VDA | Verband der Automobilindustrie (D)<br>Automobile Industry Association |
| VDI | Verein Deutscher Ingenieure<br>Association of German Engineers |
| VDMA | Verband Deutscher Maschinen- und Anlagenbau<br>German Engineering Federation |
| VDS | Volvo Drain Specification |
| VGB | Technische Vereinigung der Großkraftwerkbetreiber (D)<br>Technical Association of Large Power Plant Operators |
| VGO | Vacuum Gas Oil |
| VHVI | Very High Viscosity Index |
| VI | Viscosity Index |
| VIE | Viscosity Index Extended |
| VII | Viscosity Index Improver |

| | |
|---|---|
| VKA | Vier-Kugel-Apparat<br>Four-Ball-Apparatus |
| VOC | Volatile Organic Compounds |
| Vp | Viscosity-pressure |
| VPI | Vapor Phase Inhibitor |
| VT | Viscosity-Temperature |
| VTC | Viscosity-Temperature-Coefficient |
| WGK | Wassergefährdungsklasse<br>Water Hazardous Class (Water Pollution Class) |
| XHVI | Extra High Viscosity Index |
| XRF | X-ray Fluorescence spectrometry |
| ZAF | Zinc and Ashes Free |

# 1
# Lubricants and their Market

*Theo Mang*

## 1.1
## Preface

The most important function of lubricants is the reduction of friction and wear and in some cases, the relative movement of two bearing surfaces is only possible if a lubricant is present. In times when saving energy and resources and cutting emission have become central environmental matters, lubricants are increasingly attracting public awareness. Scientific research has shown that 0.4 % of gross domestic product could be saved in terms of energy in Western industrialized countries if current tribological knowledge, i.e. the science of friction, wear and lubrication, was just applied to lubricated processes.

Apart from important applications in internal combustion engines, vehicle and industrial gearboxes, compressors, turbines or hydraulic systems, there are a vast number of other applications which mostly require specifically tailored lubricants. This is illustrated by the numerous types of greases or the different lubricants for chip-forming and chip-free metalworking operations which are available. Between 5000 and 10 000 different lubricant formulations are necessary to satisfy more than 90 % of all lubricant applications.

If one thinks of lubricants today, the first type to come to mind are mineral oil-based. Mineral oil components continue to form the quantitatively most important foundation of lubricants. Petrochemical components and increasingly derivatives of natural, harvestable raw materials from the oleo-chemical industry are finding increasing acceptance because of their environmental compatibility and some technical advantages.

On average, lubricating oils, which quantitatively account for about 90 % of lubricant consumption, consist of about 93 % base oils and 7 % chemical additives and other components (between 0.5 and 40 %).

The development of lubricants is closely linked to the specific applications and application methods. As a simple description of materials in this field makes little sense, the following sections will consider both lubricants and their application.

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## 1.2
## Lubricant Sales

Lubricants today are classified into two major groups: automotive lubricants and industrial lubricants. Industrial lubricants can be sub-divided into industrial oils and industrial specialties. Specialties in this case are principally greases, metalworking lubricants and solid lubricant films.

Process oils are often included in lubricant statistics. These are oils which are included as raw materials in processes, but above all as plasticizers for the rubber industry. Process oil's only link with lubricants is that they are mineral oil products resulting from the refining of base oils but they often distort lubricant consumption figures. They will not be covered in this book. However, to provide a degree of comparison, they have been included in the following lubricant statistics.

In 2004, 37.4 million tons of lubricants were consumed worldwide (53 % automotive lubricants, 32 % industrial lubricants, including related specialties, 5 % marine oils, and 10 % process oils). Of total industrial lubricants, 37 % were hydraulic oils, 7 % industrial gear oils, 31 % other industrial oils, 16 % metalworking fluids (including temporary corrosion preventives whose multipurpose function often includes lubrication), and 9 % greases.

**Tab. 1.1** Regional breakdown of world demand for lubricants (without marine lubricants).

| **Region** | **Amount (kT)** | |
|---|---|---|
| | **2004** | **2005[a]** |
| North America | 8,170 | 8,130 |
| Latin America | 3,215 | 3,295 |
| Western Europe | 4,705 | 4,635 |
| Central/Eastern Europe | 4,835 | 4,905 |
| Near/Middle East | 1,775 | 1,795 |
| Africa | 1,850 | 1,870 |
| Asia-Pacific | 11,580 | 11,870 |
| World | 36,130 | 36,500 |

a Estimated [1.4, 1.5]

Table 1.2 shows the 2004 per-capita consumption for various regions. North America and Western Europe make up 35.9 % of world lubricant consumption even though these regions only account for 9.4 % of the world's population [1.1, 1.4, 1.5].

**Tab. 1.2** Per-capita consumption of lubricants in 2005 [1.4, 1.5].

| **Region** | **Amount (kg p.a.)** |
|---|---|
| North America | 24.9 |
| Central/Eastern Europe | 12.1 |
| Western Europe | 11.8 |
| Near/Middle East | 9.4 |
| Latin America | 5.7 |
| Asia-Pacific | 3.1 |
| Africa | 2.1 |
| **World** | **5.6** |

Global per-capita consumption decreased from 8.8 to 5.6 kg per year between 1970 and 2004, i.e. the increase in lubricant demand (+ 12.3 %) did not keep up with the worldwide growth in population (+ 72.5 %) during this period.

Since 1975, quantitative lubricant demand has significantly detached itself from gross national product and also from the number of registered vehicles. This quantitative view, which at first glance shows a continuous decline in lubricant volumes, gives an inadequate impression of the significance of the lubricants business today. In almost all areas, products now have a longer life and offer greater performance, i.e. specific lubricant consumption has declined but specific revenues have increased noticeably. This is also confirmed by the volumetrically very important group of engine oils: the doubling of requirements with extended oil change intervals in recent years have quadrupled the cost of such oils. The efforts to increase the life of lubricants are not based on the wish to reduce lubricant costs. Much more important is the reduction of service and maintenance costs which result from periodic oil changing or regreasing.

As about 50 % of the lubricants sold worldwide end in and thus pollute the environment, every effort is made to minimize spillages and evaporation. An example is diesel engine particulate emissions, about a third of which are caused by engine oil evaporation. These high lubricant losses into the environment were behind the development of environmentally friendly lubricants which are thoroughly covered in this book.

A further incentive to reduce specific consumption is the ever-increasing cost of disposal or recycling of used lubricants. But this again creates new demands on lubricants because reduced leakage losses means less topping-up and less refreshing of the used oil. The new oils must therefore display good aging stability.

Bearing in mind the growth potential in Asia where per-capita consumption in some areas is extremely low (2004: India 1.1 kg, China 2.9 kg) and a continuing reduction in volumes or stagnation in Western industrialized countries, overall a modest global growth is forecast. This has been estimated to be 0.5 % p.a. between 2005 and 2010 [1.2, 1.4, 1.5]. The growth in value will be more pronounced because the rapid globalization of technologies will promote high-value products even in the new lubricant markets such as China, India or Indonesia and the machines and plant used in these countries will be similar or identical to those used in the developed industrialized countries.

## 1.3
## The Lubricants Industry

Worldwide, there are 1380 lubricant manufacturers ranging from large to small. On one hand there are vertically-integrated petroleum companies whose main business objective is the discovery, extraction and refining of crude oil. Lubricants account for only a very small part of their oil business. At present, there are about 180 such national and multinational oil companies engaged in manufacturing lubricants. The 1200 independent lubricant companies mainly concentrate on the manufacturing and marketing of lubricants and view lubricants as their core business. While the large, integrated companies focus on high-volume lubricants such as engine, gear and hydraulic oils, many independent lube companies concentrate on specialties and niche business, where apart from some tailor-made lubricants, comprehensive and expert customer service is part of the package.

The top 1 % of the world's manufacturers of finished lubricants (of which there are more than 1380) account for more than 60 % of global sales; the other 99 % share less than 40 %. The world's largest manufacturers of finished lubricants are listed in Tables 1.3 and 1.4.

**Tab. 1.3** World ranking of the largest manufacturers of lubricants (2005) [1.3, 1.4, 1.5].

| | | |
|---|---|---|
| 1 | Shell | Great Britain/The Netherlands |
| 2 | Exxon Mobil | USA |
| 3 | BP | UK |
| 4 | Petrochina/Sinopec | China |
| 5 | Chevron | USA |
| 6 | Lukoil | Russia |
| 7 | Fuchs | Germany |
| 8 | Nippon Oil | Japan |
| 9 | Valvoline | USA |
| 10 | Idemitsu | Japan |
| 11 | Conoco Phillips | USA |
| 12 | CPC | Taiwan |
| 13 | PDVSA | Venezuela |
| 14 | Repsol | Spain |
| 15 | Indian Oil | India |
| 16 | Agip | Italy |
| 17 | Yukos | Russia |

**Tab. 1.4** World ranking of the largest manufacturers of industrial lubricants (2005) [1.3, 1.4, 1.5].

| | | |
|---|---|---|
| 1 | Exxon Mobil | USA |
| 2 | Shell | Great Britain/Netherlands |
| 2 | Petrochina/Sinopec | China |
| 4 | Chevron | USA |
| 5 | BP | UK |
| 6 | Fuchs | Germany |
| 7 | Nippon Oil | Japan |
| 8 | Idemitsu | Japan |
| 9 | Total | France |
| 10 | Lukoil | Russia |
| 11 | PDVSA | Venezuela |
| 12 | Yukos | Russia |
| 13 | Repsol | Spain |
| 14 | Agip | Italy |
| 15 | Valvoline | USA |
| 16 | Conoco Phillips | USA |

The independent lubricant manufacturers also generally purchase raw materials on the open market and they rarely operate base oil refineries. They buy their raw materials from the chemical and oleo-chemical industry and their mineral base oils from the large petroleum companies.

The production of simple lubricants normally involves blending processes but specialties often require the use of chemical processes such as saponification (in the case of greases), esterification (when manufacturing ester base oils or additives) or amidation (when manufacturing components for metalworking lubricants). Further manufacturing processes include drying, filtration, homogenizing, dispersion or distillation.

Depending on their field of activity, lubricant manufacturers invest between 1 and 5 % of their sales in research and development.

Towards the end of the nineties, the petroleum industry was affected by a wave of mergers which will continue. These created new and larger lubricant structures at the merged companies. The principal reasons for these mergers were economic factors in crude oil extraction and refining which resulted in lower refining margins.

Independent lube companies also experienced a continuing concentration with technological, safety-at-work and ecological considerations along with the globalization of lubricant consumers playing an important role. Critical mass is becoming increasingly important in company strategies.

## 1.4
## Lubricant Systems

Apart from the most common lube oils, the many thousands of lubricant applications necessitate a diverse number of systems which is seldom equaled in other product groups.

The group next to oils are emulsions, which as oil-in-water emulsions are central to water-miscible cutting fluids (Chapter 14), rolling emulsions and fire-resistant HFA hydraulic fluids (Chapter 11). In these cases, the lubricant manufacturer normally supplies a concentrate which is mixed with water locally to form an emulsion. The concentration of these emulsions with water are generally between 1 and 10 %. The annual consumption of such emulsions in industrialized countries is about the same as all other lubricants together. From this point of view, the volumetric proportion of these products (as concentrates) is significantly under-rated in lubricant statistics with regard to the application engineering problems they create and their economic significance.

The next group of lubricant systems are water-in-oil emulsions. Their most important application is in metal forming. These product are supplied ready-to-use or as dilutable concentrates. Fire resistant HFB fluids are designed as water-in-oil emulsions too (invert emulsions).

In some special cases, oil-in-oil emulsions are developed as lubricants and these are primarily used in the field of metalworking.

Water-based solutions in the form of non-dispersed systems are sometimes used in chip-forming metalworking operations.

Greases (Chapter 16) are complex systems consisting of base oils and thickeners based on soaps or other organic or inorganic substances. They are available in semiliquid form (semifluid greases) through to solid blocks (block greases). Special equipment is required for their production (grease-making plants). A group of products closely related to greases are pastes.

Solid lubricant suspensions normally contain solid lubricants in stable suspension in a fluid such as water or oil. These products are often used in forging and extrusion as well as other metalworking processes. Solid lubricant films can also be applied as suspensions in a carrier fluid which evaporates before the lubricant has to function.

Solid lubricant powders can be applied directly to specially-prepared surfaces.

In the case of dry-film lubricants (Chapter 17), solid lubricants are dispersed in resin matrices. Dry-film lubricants are formed when the solvent (principally water or hydrocarbons) evaporates.

Molten salts or glass powder are used for hot forming processes such as extrusion. These are normally supplied as dry powders and develop lubricity when they melt on the hot surface of the metal.

Polymer films are used when special surface protection is required in addition to lubricity (e.g. the pressing of stainless steel panels). Together with greases, these products are also used to some extent in the construction industry.

An intermediary field between materials and lubrication technology is the wide area of surface treatment to reduce friction and wear. While the previously mentioned dry-film lubricants are an accepted activity of the lubricants industry, chemical coatings are somewhat controversial. These coatings are chemically bonded to the surface of the metal. They include oxalation and phosphating (zinc, iron and manganese). In cases when such coatings adopt the carrier function of an organic lubricant, the entire system could be supplied by the lubricant manufacturer. If the chemical coating is not designed to be supplemented with an additional lubricant coating (e.g. dry film on phosphatized gear), it will probably be supplied by a company which specializes in surface degreasing and cleaning.

Even more different from traditional lubricants are metallic or ceramic coatings which are applied with CVD (chemical vapor deposition) or PVD (physical vapor deposition) processes. They also sometimes replace the EP functions of the lubricants (Chapter 6). Such coatings are increasingly being used together with lubricants to guarantee improved wear protection in extreme conditions and over long periods of time.

# 2
# Lubricants in the Tribological System

*Theo Mang*

The development of lubricants has become an integral part of the development of machinery and its corresponding technologies. It is irrevocably and interdisciplinarily linked to numerous fields of expertise and without this interdisciplinary aspect, lubricant developments and applications would fail to achieve success.

## 2.1
## Lubricants as Part of Tribological Research

Tribology (derived from the Greek tribein, or tribos meaning rubbing) is the science of friction, wear, and lubrication. Although the use of lubricants is as old as mankind, scientific focus on lubricants and lubrication technology is relatively new. The term tribology was first introduced in 1966 and has been used globally to describe this far-reaching field of activity since 1985. Even though efforts had been made since the 16th century to describe the whole phenomenon of friction scientifically (Leonardo da Vinci, Amontons, Coulomb), the work always concentrated on single aspects and lubricants were not even considered. Some research work performed up to the early nineteen seventies totally ignored the chemical processes which take place in lubricated friction processes.

Tribology, with all its facets, is only sporadically researched. Fundamental scientific tribological research takes place at universities which have engineering or materials-testing departments. Naturally, lubricant manufacturers also perform research. The advantage of tribological research by engineering departments is the dominant focus on application engineering. The most common disadvantage is the lack of interdisciplinary links to other fields of expertise. Joint research projects which combine the disciplines of engineering, materials, chemistry, health and safety and the work conducted by lubricant manufacturers themselves therefore offer the best prospects of practical results.

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## 2.2
## The Tribological System

The tribological system (commonly referred to as the tribosystem, Fig. 2.1) consists of four elements: the one contacting partner, the opposing contacting partner (material pair), the interface between the two and the medium in the interface and the environment [2.1]. In lubricated bearings the lubricant is located in this gap. In plain bearings, the material pair are the shaft and the bearing shells; in combustion engines they are the piston rings and the cylinder wall or the camshaft lobes and the tappets and in metalworking, the tool and the workpiece.

The variables are the type of movement, the forces involved, temperature, speed, and duration of the stress. Tribometric parameters – such as friction, wear, and temperature data – can be gathered from the stress area. Tribological stress is the result of numerous criteria of surface and contact geometry, surface loading, or lubricant thickness. Tribological processes can occur in the contact area between two friction partners – which can be physical, physical–chemical (e.g. adsorption, desorption), or chemical in nature (tribochemistry).

## 2.3
## Friction

The description of friction as the cause of wear and energy losses has always posed significant problems because of the complexity of the tribological systems. There is also no internationally-recognized nomenclature. In this discussion friction is described according to its type, and by the combination of friction and lubrication conditions, in line with the view taken by most experts.

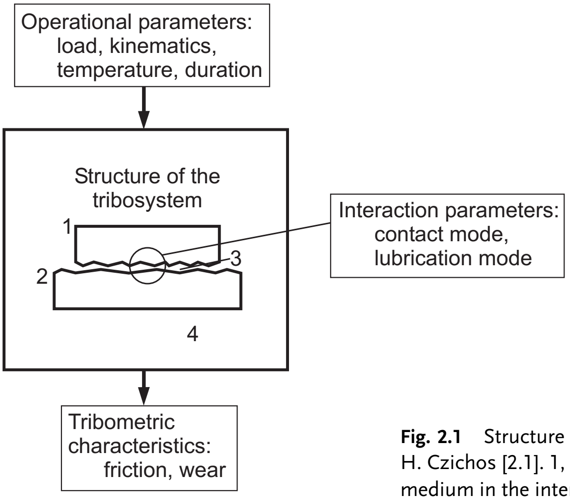

**Fig. 2.1** Structure of the tribosystem according to H. Czichos [2.1]. 1, 2. material pair; 3. interface and medium in the interface (lubricant); 4. environment.

## 2.3.1
## Types of Friction

Friction is the mechanical force which resists movement (dynamic or kinetic friction) or hinders movement (static friction) between sliding or rolling surfaces. These types of friction are also called external friction.

Internal friction results from the friction between lubricant molecules; this is described as viscosity (Chapter 3).

The causes of external friction are, above all, the microscopic contact points between two sliding surfaces; these cause adhesion, material deformation, and grooving. Energy which is lost as friction can be measured as heat and/or mechanical vibration. Lubricants should reduce or avoid the micro-contact which causes external friction.

### 2.3.1.1 Sliding Friction

This is friction in a pure sliding motion with no rolling and no spin (Fig. 2.2).

Figure 2.3 defines the coefficient of friction as the dimensionless ratio of the friction force *F* and the normal force *N*. The proportionality between normal force and frictional force is often given in dry and boundary friction conditions but not in fluid-film lubrication.

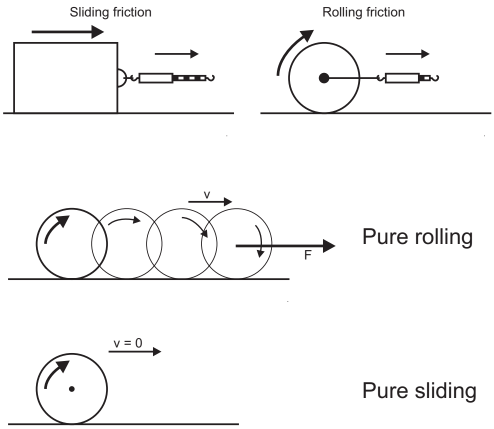

**Fig. 2.2** Sliding and rolling.

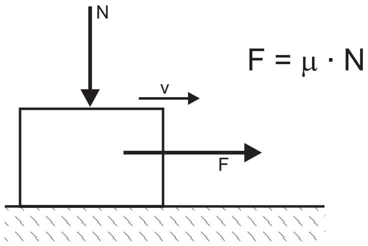

**Fig. 2.3** Coefficient of friction.

#### 2.3.1.2 **Rolling Friction**

This is the friction generated by rolling contact (Fig. 2.3). In roller bearings, rolling friction mainly occurs between the rolling elements and the raceways, whereas sliding friction occurs between the rolling elements and the cage. The main cause of friction in roller bearings is sliding in the contact zones between the rolling elements and the raceways. It is also influenced by the geometry of the contacting surfaces and the deformation of the contacting elements. In addition, sliding also occurs between the cage pockets and the rolling elements.

If rolling motion and sliding motion combine to any significant extent, as for gear tooth meshing, special terminology has been created. The word 'Wälzreibung' which is derived from 'Wälzen' (rolling, e.g. steel rolling) is used in Germany. Situations in which a high sliding/rolling ratio occur require totally different lubrication than does pure sliding. Figure 2.4 shows this 'rolling friction' during rolling and during gear meshing.

#### 2.3.1.3 **Static Friction**

The static coefficient of friction is defined as the coefficient of friction corresponding to the maximum force that must be overcome to initiate macroscopic motion between two bodies (ASTM).

#### 2.3.1.4 **Kinetic Friction**

Different from static friction, kinetic friction occurs under conditions of relative motion. ASTM defines the kinetic coefficient of friction as the coefficient under conditions of macroscopic relative motion of two bodies. The kinetic coefficient of friction sometimes called dynamic coefficient of friction is usually somewhat smaller than the static coefficient of friction.

#### 2.3.1.5 **Stick–Slip**

Stick–slip is a special form of friction which often results from very slow sliding movements when the friction partners are connected to a system which can vibrate. The process is influenced by the dependence of the coefficient of sliding friction on speed. This generally occurs when the static coefficient of friction ($f_{\text{stat}}$) is larger than the dynamic coefficient of friction ($f_{\text{dyn}}$). Stick–slip is normally encountered with machine tools which operate with slow feeds. Stick–slip can cause chatter marks on components (Fig. 2.5).

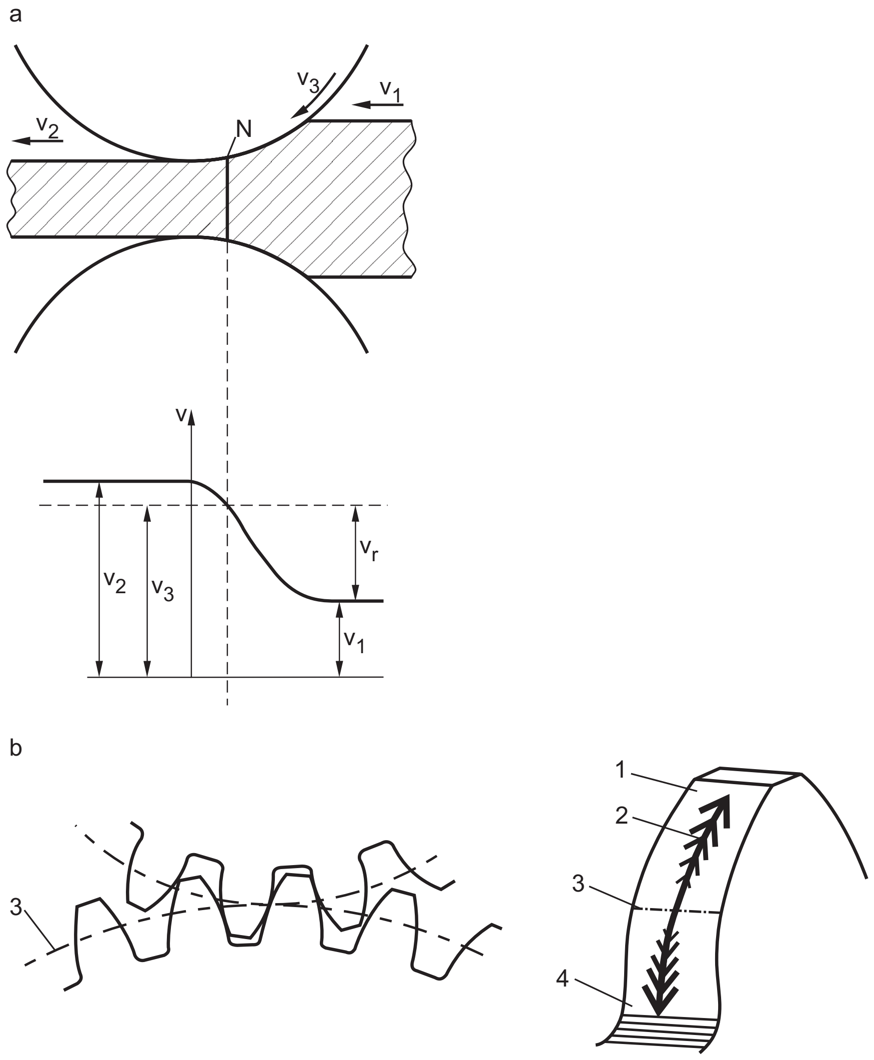

**Fig. 2.4** 'Wälzreibung', mixing of rolling and sliding motions. a. rolling in metal forming; $\upsilon_1$, initial speed of the sheet metal; $\upsilon_2$, final speed of the sheet metal; $\upsilon_3$, speed of the roller; $\upsilon_r$, speed difference in the roll gap (sliding part); N, neutral point (non-slip point, pure rolling); b, engagement of gear teeth, 1, 2, 4. high sliding/rolling ratio; 3. pitch circle (pure rolling, no slip).

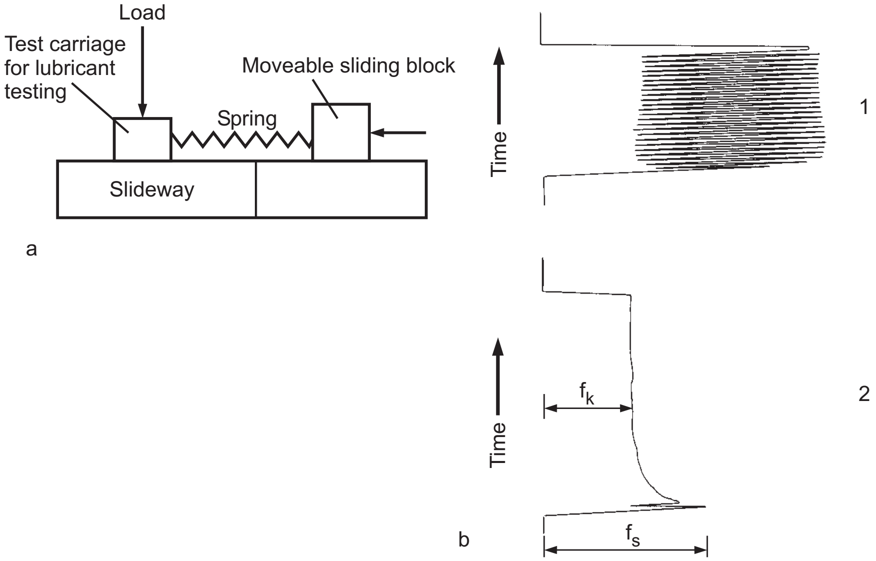

**Fig. 2.5** Stick–slip. a. Test equipment for stick–slip; b. Results of stick–slip behavior of two oils; 1. oil with bad stick–slip behavior; 2. oil with good stick–slip behavior; $f_k$. relative kinematic coefficient of friction; $f_s$. relative static coefficient of friction.

## 2.3.2
## Friction and Lubrication Conditions

In tribological systems, different forms of contact can exist between contacting partners.

### 2.3.2.1 Solid Friction (Dry Friction)

This occurs when two solids have direct contact with each other without a separating layer. If conventional materials are involved, the coefficients of friction and wear rates are high. Lubrication technology attempts to eliminate this condition.

### 2.3.2.2 Boundary Friction

The contacting surfaces are covered with a molecular layer of a substance whose specific properties can significantly influence the friction and wear characteristics. One of the most important objectives of lubricant development is the creation of such boundary friction layers in a variety of dynamic, geometric and thermal conditions. Such layers are of great importance in practical applications when thick, long-lasting lubricant films to separate two surfaces are technically impossible. Boundary lubricating films are created from surface-active substances and their chemical reaction products. Adsorption, chemisorption, and tribochemical reactions also play significant roles.

Although boundary friction is often allocated to solid friction, the difference is of great significance to lubricant development and the understanding of lubrication and wear processes especially when the boundary friction layers are formed by the lubricants.

#### 2.3.2.3 **Fluid Friction**

In this form of friction, both surfaces are fully separated by a fluid lubricant film (full-film lubrication). This film is either formed hydrostatically or more commonly, hydrodynamically. From a lubricants point of view, this is known as hydrodynamic or hydrostatic lubrication (Fig. 2.6). Liquid or fluid friction is caused by the frictional resistance, because of the rheological properties of fluids.

If both surfaces are separated by a gas film, this is known as gas lubrication.

#### 2.3.2.4 **Mixed Friction**

This occurs when boundary friction combines with fluid friction. From a lubricants technology standpoint, this form of friction requires sufficient load-bearing boundary layers to form. Machine elements which are normally hydrodynamically lubricated experience mixed friction when starting and stopping.

For roller bearings, one of the most important machine elements, it has been shown that the reference viscosity either of lubricating oils or of the base oils of greases is not sufficient to ensure the formation of protecting lubricant layers and the required minimum lifetime. Under mixed friction conditions it is important to choose the appropriate lubricant, i.e. that which enables the formation of tribolayers by anti-wear and extreme pressure additives [2.7].

In 2004, Schwarze and Wiersch described a means of calculating mixed lubrication contacts over a wide range of operating conditions and applications. The performance of the mixed friction model was demonstrated using the example of a cam tappet contact [2.8, 2.9].

#### 2.3.2.5 **Solid Lubricant Friction**

This special form of friction occurs when solid lubricants are used (Chapter 17). It cannot be allocated to the previously mentioned forms of friction because particle shape, size, mobility and, in particular, crystallographic characteristics of the particles justify a separate classification.

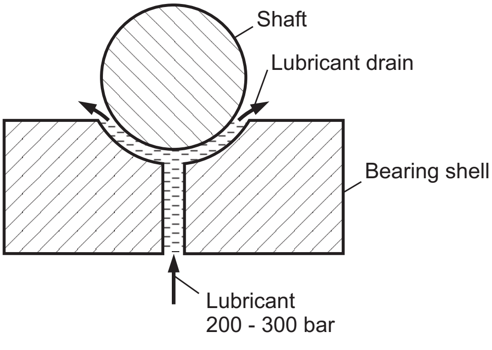

**Fig. 2.6** Hydrostatic lubrication as a form of fluid friction.

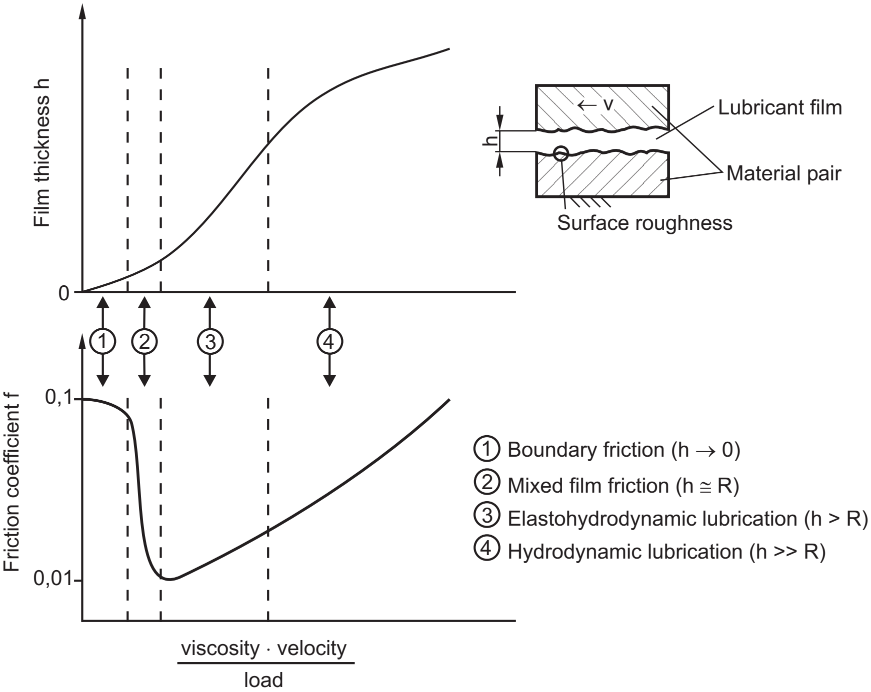

**Fig. 2.7** Stribeck graph according to H. Czichos and K.-H. Habig [2.2].

### 2.3.2.6 Stribeck Diagram

The friction or lubrication conditions between boundary and fluid friction are graphically illustrated by use of Stribeck diagram (Fig. 2.7) [2.2].These are based on the starting-up of a plain bearing whose shaft and bearing shells are, when stationary, separated only by a molecular lubricant layer. As the speed of revolution of the shaft increases (peripheral speed) a thicker hydrodynamic lubricant film is created what initially causes sporadic mixed friction but which, nevertheless, significantly reduces the coefficient of friction. As the speed continues to increase, a full, uninterrupted film is formed over the entire bearing faces; this sharply reduces the coefficient of friction. As speed increases, internal friction in the lubricating film adds to external friction. The curve passes a minimum coefficient of friction value and then increases, solely as a result of internal friction.

The lubricant film thickness shown in Fig. 2.7 depends on the friction and lubrication conditions including the surface roughness, *R*.

### 2.3.2.7 Hydrodynamic Lubrication

Figure 2.8 demonstrates the formation of a hydrodynamic liquid film. The lubricant is pulled into the conical converging clearance by the rotation of the shaft. The created dynamic pressure carries the shaft.

On the basis of the Navier–Stokes theory of fluid mechanics, Reynolds created the basic formula for hydrodynamic lubrication in 1886. Several criteria remained

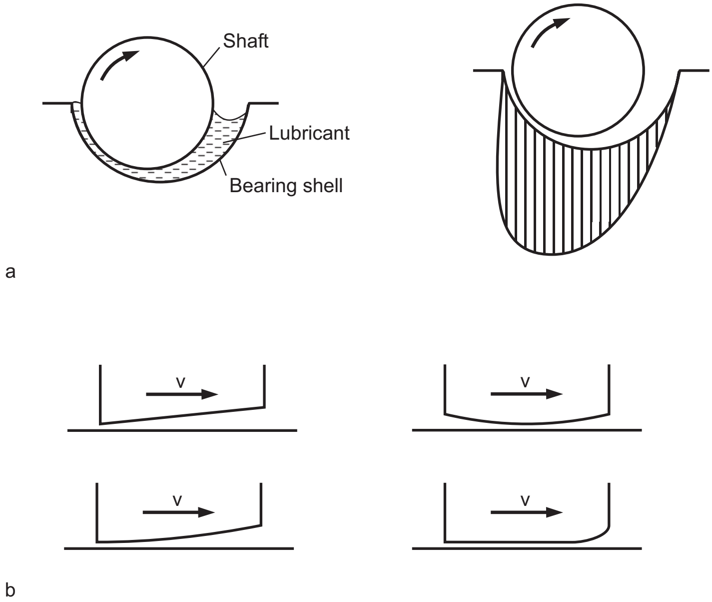

**Fig. 2.8** Formation of a hydrodynamic liquid lubricant film.
a. Rolling, development of pressure in the hydrodynamic film;
b. Sliding, preferred geometry.

excluded, however, especially the influence of pressure and temperature on viscosity. The application of the Reynolds' formula led to theoretical calculations on plain bearings. The only lubricant value was viscosity.

### 2.3.2.8 **Elasto–Hydrodynamic Lubrication (EHD Regime)**

Hydrodynamic calculation on lubricant films was extended to include the elastic deformation of contact faces (Hertzian contacts, Hertz's equations of elastic deformation) and the influence of pressure on viscosity (Chapter 3). This enables application of these elasto–hydrodynamic calculations to contact geometries other than that of plain bearings – e.g. those of roller bearings and gear teeth.

Figure 2.9 shows the elastic deformation of the ball and raceway of a ball bearing and Fig. 2.10 shows an example of Hertzian contacts for various pairs with non-converging lubricant clearance.

### 2.3.2.9 **Thermo-elasto-hydrodynamic Lubrication (TEHD)**

TEHD lubrication theory solves the Reynolds equation, including the energy equation of the lubricant film. Calculation of the energy takes into consideration heat convection in all directions, heat conduction in the radial direction, compression, and heating caused by viscous and asperity friction.

TEHD lubrication theory has been applied, e.g., in important areas of automotive engines, using a model including shear rate-dependent viscosity (Chapter 3) and

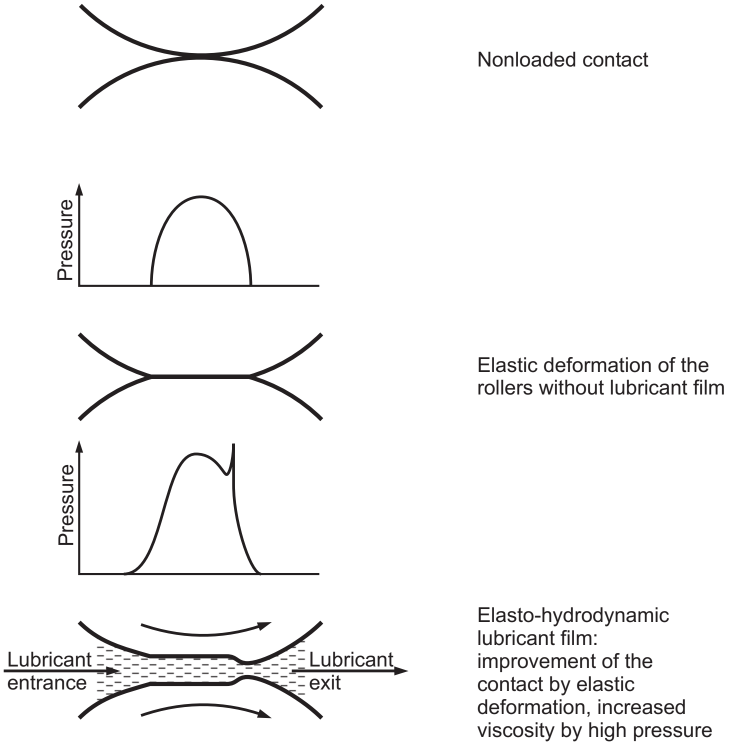

**Fig. 2.9** Improvement of hydrodynamic lubrication clearance between two rollers by Hertzian deformation (elasto–hydrodynamic contact, EHD contact), pressure distribution in the Hertzian contact.

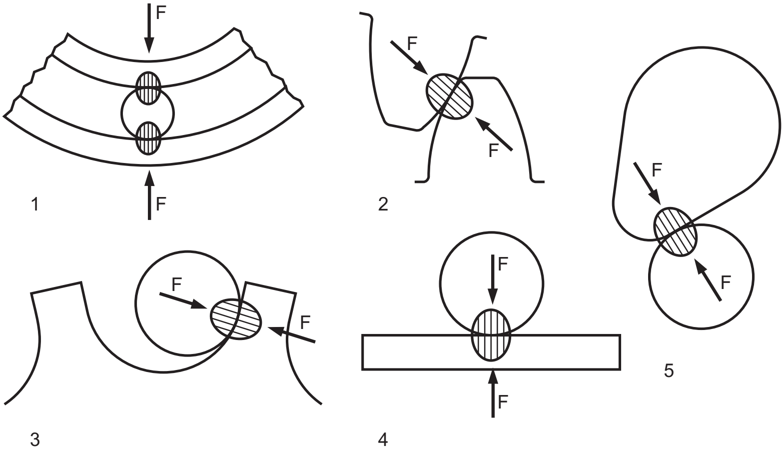

**Fig. 2.10** Hertzian contacts for different pairs with non-converging lubricant clearance [2.3]. 1. Roller bearing; 2. Gear wheels; 3. Chain wheels; 4. Roller on flat path; 5. Cam lifter.

simulation of the lubrication conditions for the main crankshaft bearing of commercial automotive engines [2.10].

## 2.4
## Wear

According to the German DIN standard 50 320, wear is defined as the progressive loss of material from the surface of a contacting body as a result of mechanical causes, i.e. the contact and relative movement of a contacting solid, liquid, or gas to the body.

### 2.4.1
### Wear Mechanisms

Wear is created by the processes of abrasion, adhesion, erosion, tribochemical reactions, and metal fatigue which are important to lubrication technology.

### 2.4.1.1 **Abrasion**

Abrasive wear occurs principally when one body is in contact with another, harder body. The contamination of lubricants with abrasive media (such as sharp quartz particles) can also cause abrasive wear. Abrasion causes the surface peaks to break-off or it gouges further abrasive particles from the surface. This form of wear can combine with other wear mechanisms.

### 2.4.1.2 **Adhesion**

This is the most complex of wear mechanisms. Molecular and atomic interactions between the two friction partners can tear material particles out of the contact faces. This is preceded by formation of a spot joint between the two materials, a process which is often called cold welding. Adhesion takes place when the boundary lubricant layer in the lubricant or other protective layer (e.g. metal oxide) is broken down. The shearing of these micro spots is a significant part of friction.

### 2.4.1.3 **Tribochemical Reactions**

The chemical reactions which occur in tribological conditions is referred to as tribochemistry. It is sometimes wrongly assumed that these reactions are governed by some special laws. Nevertheless, for better understanding of the specific reaction conditions the laws applying to tribological contact should be observed. This applies, in particular to thermal effects (flash temperatures) which are not readily macroscopically recognizable, to frictional effects which lead to chemically reactive surfaces where the valences of the metal's structural matrices play a role in wear and deformation. The removal of reaction layers thus formed constitutes tribochemical wear.

### 2.4.1.4 **Surface Fatigue**

Surface fatigue is the result of periodic loads in the contact zones. Frequent spot loading leads to surface fatigue which is a result of material fatigue. A thick, separating lubricant film either minimizes or eliminates this problem. Machine elements which are subjected to periodically severe loads in the contact zone are particularly prone to this type of damage.

#### 2.4.1.5 Erosion

Erosive wear which is a loss of material from a solid surface occurs during the impingement on a surface by a fluid containing solid particles.

#### 2.4.1.6 Fretting

The contacting partners are part of a system which oscillates at small amplitude. Most of the previously mentioned types of wear are included in fretting wear and this explains why fretting wear is seen as a separate form. Besides mechanical components tribochemical reactions play an important role. Oxidation creates layers and fretting wear is sometimes known as fretting corrosion. Fretting wear can be avoided if suitable surface-active additives are added to lubricants (especially as pastes for threaded fasteners).

#### 2.4.1.7 Cavitation

Materials can be damaged by imploding gas and vapor bubbles entrained in lubricating oils or hydraulic fluids. In systems which carry lubricants, the elimination of dragged-in air, low boiling point substances, or the use of surface active components scale-down the gas bubbles and thus reduce cavitation.

The condition of surfaces effected by cavitation can be further damaged by corrosion. This process can be controlled by the use of specific inhibitors.

### 2.4.2
### Types of Wear

There are several criteria which could be used to classify the different types of wear. For example, according to the types of (kinematic) friction which lead to wear (sliding wear, rolling wear, fretting wear), according to wear mechanisms (adhesive wear, abrasive wear, tribochemical wear) or according to the shape of the wear particle.

There is also highly specialized wear terminology for different lubricant applications; this is oriented to the geometry of the various bearing faces (e.g. clearance and crater wear in the field of chip-forming tools).

### 2.4.3
### The Wear Process

Wear can be measured gravimetrically, volumetrically or in terms of area over a period of time or against increasing load. Uniformly decreasing wear which stabilizes at a very low level can be described as running-in wear. This can be controlled by the tribochemical reactions of the additives in the lubricants. Wear can occur at relatively constant speed and ultimately lead to the functional failure of the bearing. Wear at an increasing wear rate can lead to progressive wear. In time, the material damage caused by wear will also lead to the failure of a component. To facilitate projection of possible functionality, the concept of failure analysis was introduced.

Analysis of machine element failure as a result of wear and lubrication can be performed by use of practically oriented tests. Determination of the effect of different greases on the projected failure of roller bearings has become increasingly important.

### 2.4.4
### Tribomutation

Gervé [2.6] introduced this term to describe the processes of friction and wear from the tribosystem. His aim, in particular, was to separate the interpretation of wear from the confusing association with material characteristics and to highlight much more complex processes in the tribosystem in which lubricants are also included. Accordingly, friction and wear are purely system-related values. Gervé created the basis for his friction and wear concepts by conducting numerous sensitive wear measurements in the nanometer range using the established radionuclide wear measurement technique [2.4, 2.5] and by successful ion implantation such as that used in metal-cutting tools and wear parts in combustion engines. He described the two most important tribological properties $Tp_i$ (once for wear and once for friction) in the equation:

$$Tp_i = f_i(r, P\ (M, G, L)\ e, \tau$$

where r are running conditions, e.g. speed, load, temperature and others, P are parameters such as M materials, G micro and macrogeometry, and L the lubricant and the lubrication conditions, e are environmental conditions such as humidity, dust, temperature and $\tau$ is the life of the tribosystem.

Figure 2.11 demonstrates the system dependence of wear rate for changing running conditions for two parameters. For the running conditions $r_1$, the wear rate for parameter $p_1$ is significantly lower than for parameter $p_2$; this is reversed for $r_1$.

Figure 2.12 shows the alteration of the material caused by tribomutation. The materials $M_1$ and $M_2$ at differing running times ($t_1$ and $t_2$) show wear occurring in the reverse order. Tribomutated materials have changed their tribological characteristics.

Changes to the material caused by tribomutation, which often occur under the surface of the material down to a depth of 150 nm, and which are reinforced by surface wear, can be quantified by sensitive methods of analysis of material composition. Gervé explained the material change caused by tribomutation by the part of the friction energy which is not consumed by heat generation and the binding energy of the wear particles.

Tribological system analysis and the associated tribomutation questions the practical application of a series of friction and wear tests. Apart from what happens during running-in, other friction and wear phenomenon can be explained while the metalworking process along with the cutting fluid must be seen as an important influence on the tribological properties of a component.

Figure 2.13 shows the possible changes under tribologically stressed surfaces.

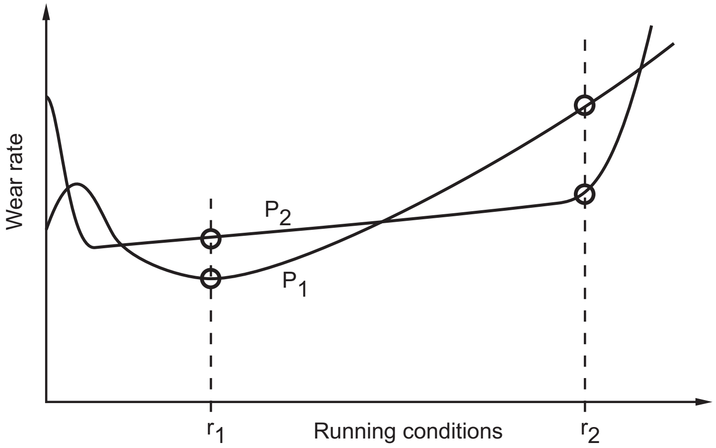

**Fig. 2.11** Tribomutation. System dependence of wear rate for changing running conditions for two sets of parameters (according to A. Gervé [2.6]).

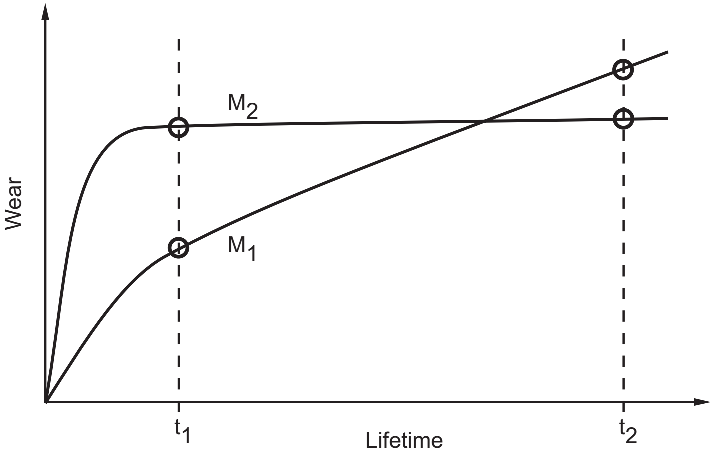

**Fig. 2.12** Tribomutation. Wear of tribosystem under constant running conditions with different materials (according to H. Gervé [2.6]).

Friction and wear development is highly influenced by the changed conditions created by tribomutation.

### 2.4.5
### Nanotribology

The preceding chapter on tribomutation and Fig. 2.13 reveal that deeper knowledge of frictional and wear processes on the micro and nano scale can enable explanation of different tribological phenomena. This implies the need for novel measurement methods or adaptation of current measurement procedures to the new tasks, the

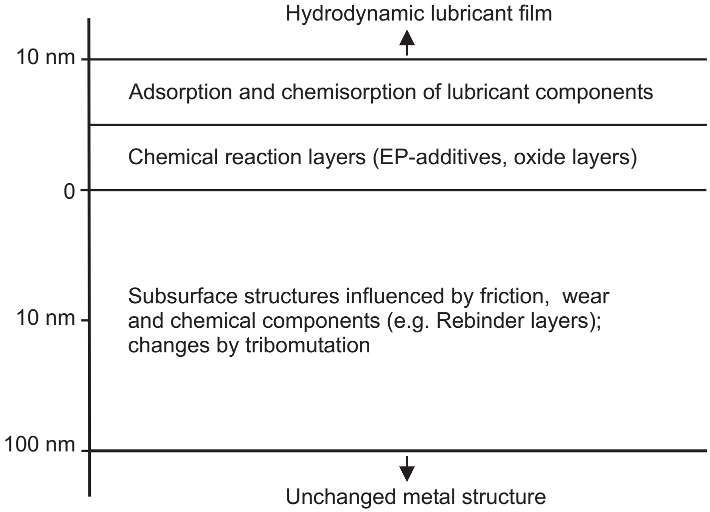

**Fig. 2.13** Lubrication conditions and possible changes by tribomutation.

development and application of which constitute an essential part of nanotechnology in general and of nanotribology in particular. This enables visualization and measurement of different criteria up to the molecular scale. In the last ten years public interest in nanotechnology and nanotribology has grown substantially, because they are expected not only to enable theoretical explanation of different phenomena but also to improve economical realization [2.11]. Gervé [2.12] assigns three functions to micro and nanotribology:

1. Analysis of the movement of single atomic groups in relation to the basic material and measurement of unequally distributed friction forces with nanometer resolution. In addition, the new definition of topographical roughness is relevant to understanding of the interrelationship between roughness and friction. This also includes experimental and theoretical analysis of frictional interactions at the atomic level, excitation of lattice oscillations, and related reduction of the bond strength of the lattice elements, finally leading to wear.
2. Nano and microtribology implies measurement and calculation of classical and tribological characteristics.
3. Examination of the effects of frictional phenomena on macroscopic tribosystems in the millimeter and micrometer range. This includes tribomutation. A recently started interesting project detected the effects of tribomutation on the piston–liner system of automotive vehicles. It was proved that production-related material changes (different cutting, grinding, and honing processes, special metalworking fluids, dry cutting) had an effect on the friction and wear properties of engine components [2.13].

One of the most important aspects of nanotribology is the rapid development of microscale and nanoscale test equipment [2.13, 2.14, 2.15, 2.16], for example the microtribometer for detection of forces in the micronewton range on springs, interferometer-based tribometers or fiber-optics-based microtribometers, microanalysis for hardness measurement and scratch testing, and nanoscale probe techniques, in which the atomic force microscope (AFM, RKM) has become extremely important. This enables nanoscale scanning of surfaces with a tip passing over the surface with an accuracy of $10^{-10}$ m, corresponding to the diameter of an atom. In combination with the aforementioned spring and optical or capacitive instruments, this creates an image of the surface. The instrument also enables friction force measurement (FFM) on the nanoscale [2.17].

Also of interest are biological, micro, and nanotribology [2.18], the objective of which is to gather information about friction, adhesion, and wear of biological systems and to apply this new knowledge to the design of micro electromechanical systems and the development of new types of monolayer lubrication and other technology [2.18]. Nanotribology is the study of nanomaterials used as friction and wear-reducing lubricant additives. Carbon nanotubes and fullerene-like materials have entered lubricant research laboratories [2.19]. Submicron graphite particles, on the other hand, have been used as lubricant additives for many years.

### 2.4.6
### Tribosystems of Tomorrow

To develop tribosystems and, especially, lubricants for tomorrow, collaborative research centers must be formed to integrate all the necessary competence. This could be done by organizing mutual research of the lubricant industry with universities and with the companies which use lubricants. Creation of a collaborative research center at the university level must involve all those institutes which can contribute to the complex area of lubrication, wear, friction materials, and environmental and toxicological problems. The most fascinating project in this field, "Environmentally Friendly Tribosystems for the Machine Tool by Use of Suitable Coatings and Fluids", was started in the middle of the nineties in Germany at the Technical University of Aachen (RWTH Aachen). The institutes involved are:

- a technical chemistry institute for development of appropriate lubricants;
- an institute for characterizing the environmental and ecotoxicological behavior of the new lubricants;
- a surface engineering institute for developing the appropriate coatings (low temperature PVD coatings for gears, bearings, and pistons of hydrostatic units, high-temperature PVD coatings for tools used in metal cutting and forming);
- a manufacturing technology institute;
- an institute for machine tools and production engineering;
- an institute for fluid power; and
- an institute for machine elements and machine design [2.20].

# 3

# Rheology of Lubricants

*Theo Mang*

Consistency, flow properties, or viscosity in the case of oils, are key parameters to create lubrication efficiency and the application of lubricants. These are terms which appear in nearly all lubricant specifications. Viscosity is also the only lubricant value which is adopted into the design process for hydrodynamic and elasto–hydrodynamic lubrication.

## 3.1

## Viscosity

Friction generated by a fluid surrounding contacting partners, i.e. without contact of the partners, is the internal friction of the fluid. In the right-hand branch of the Stribeck graph (Fig. 2.7), internal friction increases with bearing speed. The measure of internal friction in a fluid is viscosity. Viscosity and its dimensions are best explained with a model of parallel layers of fluid which could be viewed molecularly (Fig. 3.1). If this packet of fluid layers is sheared ($\tau$), the individual fluid layers are displaced in the direction of the shearing force. The upper layers move more rapidly than the lower layers because molecular forces act to resist movement between the layers. These forces create resistance to shearing and this resistance is given the term dynamic viscosity. The difference in velocity between two given fluid layers, related to their linear displacement, is referred to as shear rate S. This velocity gradient is proportional to the shear stress ($\tau$). The proportionality constant $\eta$ is called dynamic viscosity and has the units Pa · s. Analysis of the dimensions uses the following equations:

$$S = \frac{dv}{dy}\left[\frac{\mathrm{m}}{\mathrm{s}\cdot\mathrm{m}}\right] \text{ or } \left[\mathrm{s}^{-1}\right] \tag{3.1}$$

$$\eta = \frac{\tau}{S}\left[\frac{\mathrm{N}}{\mathrm{m}^2\cdot\mathrm{s}^{-1}}\right] \text{ or } \left[\frac{\mathrm{Pa}}{\mathrm{s}}\right] \text{ or } \left[\mathrm{mPa}\cdot\mathrm{s}\right] \text{ or (Centipoise)} \tag{3.2}$$

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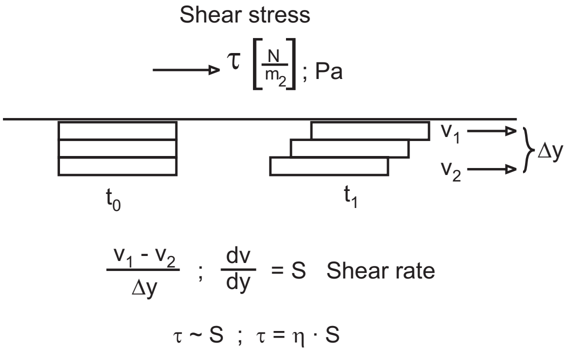

**Fig. 3.1** Explanation of viscosity.

The laboratory determination of viscosity in run-out or capillary tubes is influenced by the weight of the fluid. The relationship between dynamic viscosity and specific gravity is referred to as kinematic viscosity ν. The following unit analysis applies:

$$\nu = \frac{\eta}{\varrho}\left[\frac{\mathrm{Pa}\cdot\mathrm{s}}{\mathrm{kg}\cdot\mathrm{m}^{-3}}\right] \text{ or } \left[\frac{\mathrm{N}\cdot\mathrm{s}\cdot\mathrm{m}^3}{\mathrm{m}^2\cdot\mathrm{kg}}\right] \text{ or } \left[\frac{\mathrm{kg}\cdot\mathrm{m}\cdot\mathrm{s}^{-2}\cdot\mathrm{s}\cdot\mathrm{m}^3}{\mathrm{m}^2\cdot\mathrm{kg}}\right] \text{ or } \left[\frac{\mathrm{m}^2}{\mathrm{s}}\right]$$
$$\left[\frac{\mathrm{mm}^2}{s}\right] \text{ or (Centistoke)} \tag{3.3}$$

Fluids which display the above proportionality constant between shear stress and shear rate are referred to as Newtonian fluids, i.e. the viscosity of Newtonian fluids is independent of shear rate (Fig. 3.2). Deviations from this Newtonian behavior are sometimes referred to as structural viscosity. Those viscosities are named as apparent viscosities.

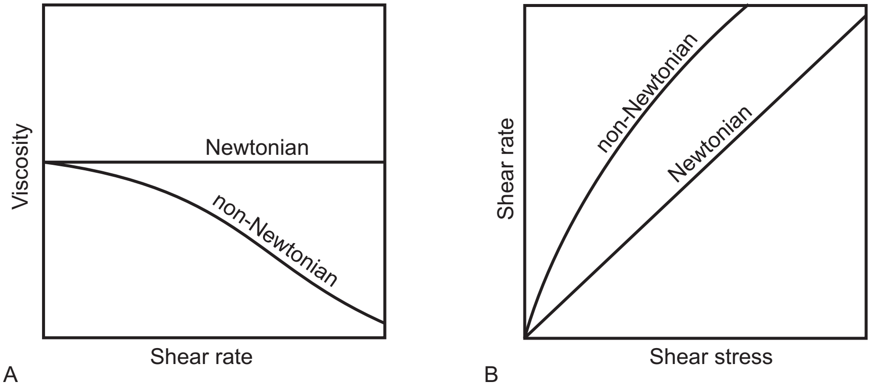

**Fig. 3.2** Flow characteristics of Newtonian and non-Newtonian lubricants. A. viscosity as a function of shear stress; B. shear rate as a function of shear stress.

As described above, kinematic and dynamic viscosity differ by way of density. Taking the example of a medium viscosity paraffinic mineral oil cut (ISO VG 32, Section 3.6), the difference is between 12 and 25 % at temperatures between 0 and 100 °C (the higher values apply to kinematic viscosity).

## 3.2
## Influence of Temperature on Viscosity (*V–T* Behavior)

The viscosity of all oils used for lubrication purposes drops significantly when their temperature increases. In linear systems, this *V–T* behavior is hyperbolic and the practical differentiation necessary in practice is difficult to replicate and the interpolation between two measured viscosities is also problematic. For these reasons, *V–T* behavior has been allocated to a function which results in a straight-line graph if suitable co-ordinates are selected.

The Ubbelohde–Walter equation has become generally accepted and also forms the basis of ASTM, ISO and DIN calculation guide lines.

$$\mathrm{lglg}\,(\nu + C) = K - m \cdot \lg T \tag{3.4}$$

In this double-logarithmic formula, $C$ and $K$ are constants, $T$ is temperature in Kelvin and $m$ is the *V–T* line slope. Figure 3.3 show the linear and Ubbelohde–Walter double-logarithmic *V–T* curves of three oils with significantly differing *V–T* lines (naphthenic oil, paraffinic HC-II / Group III-oil / Chapter 4) and a natural vegetable (rapeseed) oil.

The constant $C$ for mineral oils in the *V–T* equation is between 0.6 and 0.9.

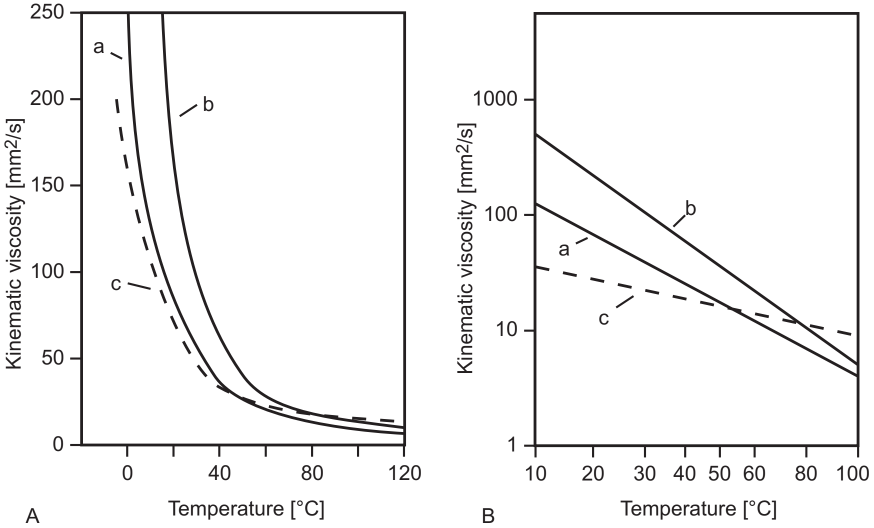

**Fig. 3.3** *V–T* behavior of various oils. A. linear; B, double-logarithmic; a, paraffinic base oil; b, naphthenic base oil; c, rapeseed oil.

The constant $C$ only plays a minor role in the viscosity calculation, larger differences are only apparent at very low temperatures.

The use of kinematic viscosity dominates in the lubricants industry even though dynamic viscosity is a much more important parameter in lubrication technology.

The Vogel–Cameron equation is used for the rapid, computer-based calculation of dynamic viscosity. The Vogel–Cameron viscosity–temperature equation is:

$$\eta = A \cdot \exp\left(\frac{B}{T + C}\right) \tag{3.5}$$

where $A$, $B$, and $C$ are constants and $T$ is again temperature in Kelvin.

The $m$ value in the Ubbelohde–Walter equation which represents the double-logarithmic $V$–$T$ graph slope is sometimes used to characterize the $V$–$T$ behavior of oils. The $m$ value for lubricant base oils is between 4.5 and 1.1 [3.1]. The smaller values apply to oils which are less affected by temperature.

$$m = \frac{\lg\lg(\nu_1 + 0.8) - \lg\lg(\nu_2 + 0.8)}{\lg T_2 - \lg T_1} \tag{3.6}$$

The viscosity–temperature constant (VTC) was introduced to better differentiate $V$–$T$ behavior when the influence of temperature is low.

$$\mathrm{VTC} = \frac{\nu_{40} - \nu_{100}}{\nu_{40}} \tag{3.7}$$

### 3.2.1
### Viscosity Index

Neither the $m$ value nor the viscosity–temperature constant have found particular acceptance in lubrication technology. This is not the case for the viscosity index *VI*, which today defines the international description of viscosity–temperature behavior. This value was first introduced in the USA in 1928. It was based on the then greatest (*VI* = 100) and the smallest (*VI* = 0) temperature dependence of US base oils.

*VI* is graphically illustrated in Fig. 3.4. Values over 100 are calculated as VIE.

Viscosity index is also defined in the most international standards.

The evaluation of $V$–$T$ behavior at low temperatures according to Ubbelohde–Walter lines or other straight-line $V$–$T$ graphs often leads to inaccuracies. The previously described dependency effects do not apply to base oils which can suffer thickening caused by crystallization of some components (e.g. paraffins) at low temperatures or those whose polymer molecules simulate viscosity effects at low temperatures.

Table 3.1 shows various $V$–$T$ characteristics for a number of oils.

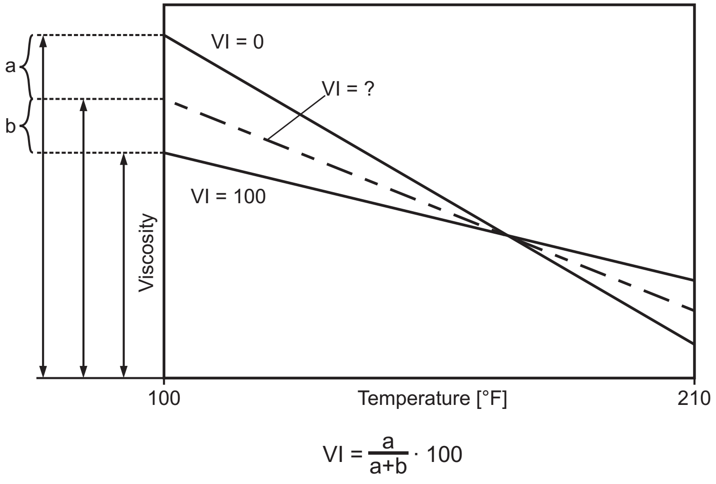

**Fig. 3.4** Graphical illustration of viscosity index (*VI*).

**Table 3.1** Various *V–T* characteristics for several oils [3.1]

| | *Kinematic viscosity (mm$^2$ s$^{-1}$)* | | *Viscosity index (VI)* | *m Constant* | *Viscosity temperature constant (VTC)* |
|---|---|---|---|---|---|
| | *40 °C* | *100 °C* | | | |
| ***Naphthenic spindle oil*** | 30 | 4.24 | 40 | 4.05 | 0.847 |
| ***Paraffinic spindle oil*** | 30 | 5.23 | 105 | 3.68 | 0.819 |
| ***Medium solvent extract*** | 120 | 8.0 | –50 | 4.51 | 0.939 |
| ***Medium polyglycol*** | 120 | 20.9 | 200 | 2.53 | 0.826 |
| ***Medium silicone oil*** | 120 | 50.0 | 424 | 1.14 | 0.583 |
| ***Multigrade motor oil (SAE 10W-30)*** | 70 | 11.1 | 165 | 2.82 | 0.841 |
| ***Ester oil*** | 30 | 5.81 | 140 | 3.40 | 0.806 |

## 3.3
## Viscosity–Pressure Dependency

The significance of viscosity–pressure dependency (*V–p* behavior) was, and still is, underestimated for numerous lubrication applications. *V–p* behavior has become a part of the calculation of elasto–hydrodynamic lubricant films. The exponential dependence of viscosity on pressure means that viscosity increases very rapidly with pressure. Metal-forming lubricants can be subject to such pressures that the viscosity of such oils can increase by a number powers of 10.

$V$–$p$ behavior can be described by the formula:

$$\eta_p = \eta_1 \cdot e^{\alpha(p - p_1)} \tag{3.8}$$

where $\eta_p$ is the dynamic viscosity at a pressure $p$, $\eta_0$ is the dynamic viscosity at 1 bar and $\alpha$ is the viscosity–pressure coefficient. The following function generates $\alpha$:

$$\alpha = \frac{1}{\eta_p} \cdot \left(\frac{d\eta_p}{dp}\right)_T \tag{3.9}$$

$V$–$p$ dependence is defined by the chemical structure of the substances with the steric geometry of the molecules being of particular significance.

Figure 3.5 shows the development of viscosity against pressure for a number of oils with different chemical structures.

$\alpha$ can increase significantly with falling temperature which has an exponential effect on viscosity. Practical lubrication technology therefore necessitates consideration of pressure and temperature to make a reasonable evaluation of viscosity.

It has recently been shown [3.3] that VI Improvers have an important effect on pressure–viscosity behavior. This affects the lubricant film thickness between lubricated highly stressed contacts under elasto-hydrodynamic (EHD) conditions. Depending on the molecular weight and concentration of the VI improvers (Chapters 9 and 6), lubricants behave differently when such films are formed. At low temperatures a thin lubricant film is formed. At higher temperatures, VI-improved lubricants form a thicker film as a consequence of a higher viscosity pressure coefficient $\alpha$.

## 3.4
## The Effect of Shear Rate on Viscosity

The definition of the viscosity of Newtonian fluids (Section 3.1), is a constant (proportionality factor) between the shear force $\tau$ and the shear rate S. This means that viscosity does not change (with the exception of temperature and pressure dependence) even when subject to greater shear forces in a friction contact zone or in other words, in isothermal and isobaric conditions. Lubricants which display dependence on shear rate are known as non-Newtonian or fluids with structural viscosity.

Oils containing polymers with specific additives or thickeners and mineral oils at low temperatures (long-chain paraffin effects) display such structure-viscous behavior. At normal application temperatures, most major lubricant base oils such as hydrocarbon oils (mineral oil raffinates or synthetic hydrocarbons), synthetic esters and natural fatty oils can withstand very high shearing forces (e.g. $10^9$ $s^{-1}$) as found in highly loaded machine elements (e.g. gearboxes) and are independent of shear rate.

Engine oils containing polymer *VI* improvers (Chapter 9) or polymer ash-free dispersants display structural–viscosity effects at low and high temperatures. As a rule, the dependence of viscosity on shear rate is undesirable. However, one can use this effect in fuel-efficiency oils. At high sliding speeds when hydrodynamic lubrications

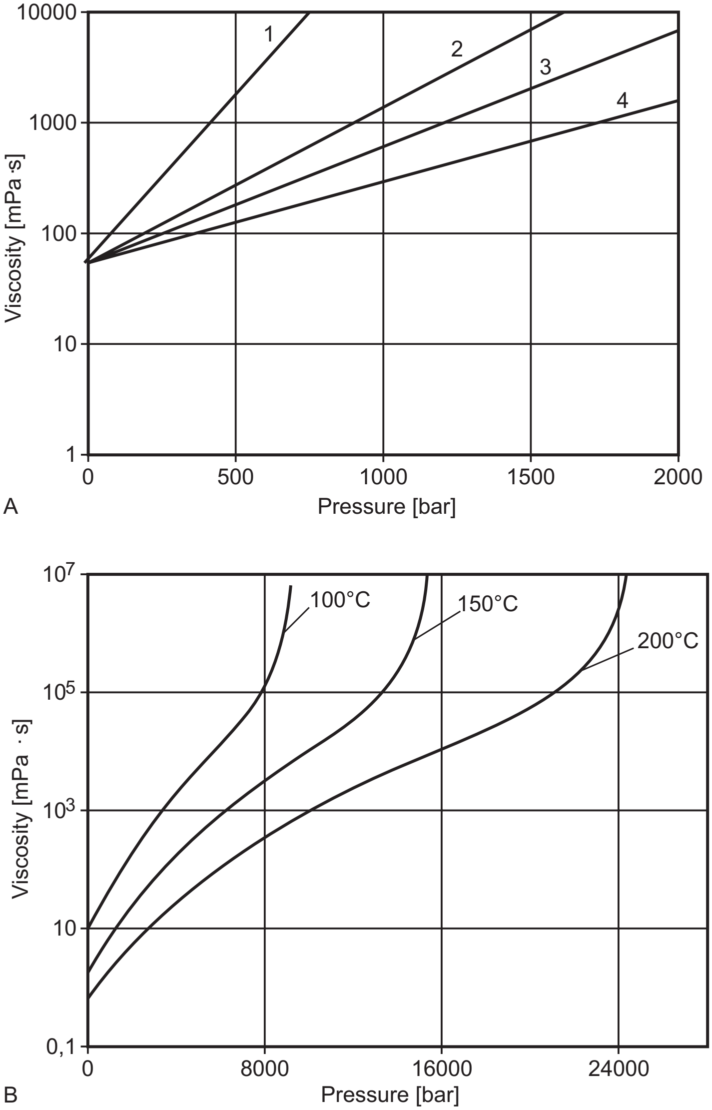

**Fig. 3.5** Development of viscosity against pressure. A. various oils: 1. aromatic oil; 2, naphthenic oil; 3, paraffinic oil; 4, biodegradable polyolester. B. increase of the viscosity–pressure coefficient with falling temperature according to H. Holland [3.2].

is given, lower viscosity generates lower friction and lower energy consumption. To keep this process under control, HTHS viscosity (high temperature high shear) was introduced. This measures viscosity at higher temperatures (corresponding to the oil temperature at friction points) and at high shear rates. This figure appears in engine oil specifications as a threshold value (Chapter 9).

While the reduction in viscosity caused by the shearing of structure-viscous fluids is reversible, i.e. after the shearing stops, the original viscosity returns, polymer-based oils can suffer a permanent reduction in viscosity. In these cases, the shearing forces lead to a mechanical change or reduction in the size of the polymer molecules so that their desired effects are minimized. These effects have been observed, in particular, with multigrade engine oils and high-*VI* hydraulic oils. The shear stability of these polymers is therefore an important quality parameter.

## 3.5
## Special Rheological Effects

Apart from the above-described structure-viscosity and the permanent reduction in viscosity caused by shearing, lubricants are subject to further rheological effects and in particular, colloidal systems consisting of solid or fluid dispersions (solid dispersions or emulsions).

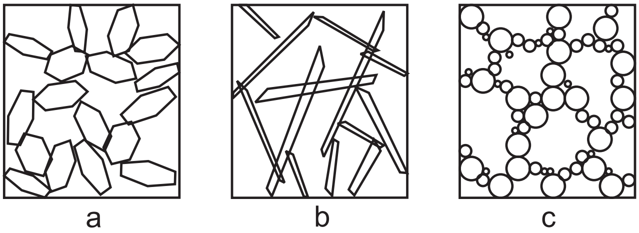

**Fig. 3.6** Various lubricant structures (a, b, c) with high sensitivity against shear stress.

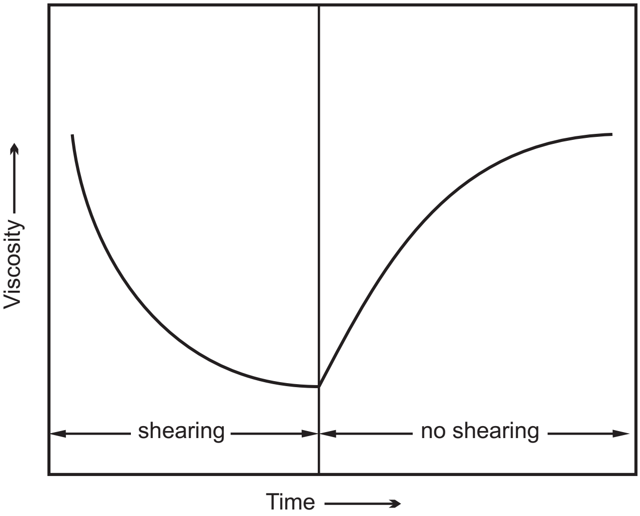

**Fig. 3.7** Flow characteristics of thixotropic lubricants.

Even small mechanical loading such as vigorous stirring can cause a system to change completely, e.g. pasty systems can breakdown into low-viscosity systems. Figure 3.6 shows such systems for different structures. If this process is time-based, i.e. continuous mechanical load causes apparent viscosity to fall over time and the original viscosity is restored after a certain rest period, such fluids are called thixotropic, Fig. 3.7 This effect is used for sheet forming lubricants (Chapter 15). In this case, a low-viscosity fluid is preferred for the application of the product (e.g. spraying) but a high-viscosity lubricant film which resists run-off is required on the panels. The rheopexy effect, in which continuous shearing causes viscosity to increase, is not applicable to lubrication technology.

Time-based viscosity changes can also be caused by the separation of colloidal particles such as paraffins from mineral base oils or additives during the storage of lubricants. This undesirable effect can occur during the cooling phase following production or by long-term storage at low temperatures. This separation process can also be independent of temperature and be caused by the solubility status of additives in the base oils.

### 3.5.1
### Greases

The rheological description of greases is also complex because of their complex system structure (fluids, soap thickeners, solid thickeners, additives, Chapter 16). The mathematical description of apparent viscosity has not gained general acceptance in practice. The Sisko equation has developed some significance for shear rates between $10^{-2}$ and $10^{-4}$ $s^{-1}$ [3.1].

$$\eta_S = a + b\gamma^{n-1} \tag{3.10}$$

where $a$ is dynamic base oil viscosity, $\gamma$ the shear rate and the constants $b$ and $n$ describe pseudoplastic behavior.

At lower shear loads, greases behave similarly to structure-viscous substances but at high shear loads, like Newtonian base oils.

## 3.6
## Viscosity Grades

To simplify the classification of lubricants according to their application, viscosity grades were introduced which are now internationally accepted. ISO viscosity grades apply to industrial lubricants while SAE classifications apply to automotive engine and gear oils.

### 3.6.1
### ISO Viscosity Grades

18 viscosity grades are laid down in the ISO standard 3448. Over the range from 2 to 2,500 $mm^2\ s^{-1}$, these are the international standard number series E6 rounded to whole numbers when the 6 numerals correspond to one power of ten (the first and fourth power of 10 are reduced). The viscosity grades were also adopted into or added to national standards such as ASTM or DIN.

Viscosity grades are not used for all industrial lubricants. Particularly oils for chip-forming and chip-less metalworking processes are not classified in this way.

Apart from the viscosity grades, ISO 3448 defines tolerances as well as median viscosities.

### 3.6.2
### Other Viscosity Grades

#### 3.6.2.1 Engine Oils

To define the viscosity of engine oils, two or three viscosity thresholds were selected to define flow properties at low temperatures and to define a minimum viscosity at high temperatures. Maximum viscosity at low temperature should ensure the rapid oil circulation to all lubrication points and permit a sufficiently higher cranking speed for starting and the minimum viscosity at 100 °C should ensure that adequate lubrication of the bearings occurs at high temperatures. Although the classification system was introduced by the Society of Automotive Engineers (SAE) together with the American Society for Testing and Materials (ASTM), it is used throughout the world and has been adopted into all national standards. Low temperature viscosity is measured as dynamic viscosity with a specially constructed rotational viscosimeter (cold cranking simulator) at low shear rates (Chapters 9 and 13).

Oils which only fit into one viscosity grade are known as monograde oils. The $V$–$T$ behavior of such oils corresponds to that of conventional mineral oils without $VI$ improvers. Oils which cover two or more viscosity grades are multigrade oils and are based on oils containing $VI$ improvers or base oils with high natural $VI$.

#### 3.6.2.2 Automotive Gear Oils

Specific SAE viscosity grades have been created for automotive gearbox, axle and differential oils. Compared with engine oils, the low temperature behavior of these oils is more heavily weighted in that a single maximum dynamic viscosity figure and the corresponding maximum temperatures are determined for a number of viscosity grades.

#### 3.6.2.3 Industrial Gear Oils

AGMA (American Gear Manufacturers Association) defines 9 viscosity ranges for industrial gear oils.

### 3.6.2.4 Viscosity Grades for Base Oils

Mineral base oils are traditionally classified according to Saybolt Universal Seconds (SUS). A 150 N base oil shows a viscosity of 150 SUS at 100 °F.

### 3.6.2.5 Comparison of Viscosity Grades

Figure 3.8 compares viscosity ranges for various applications.

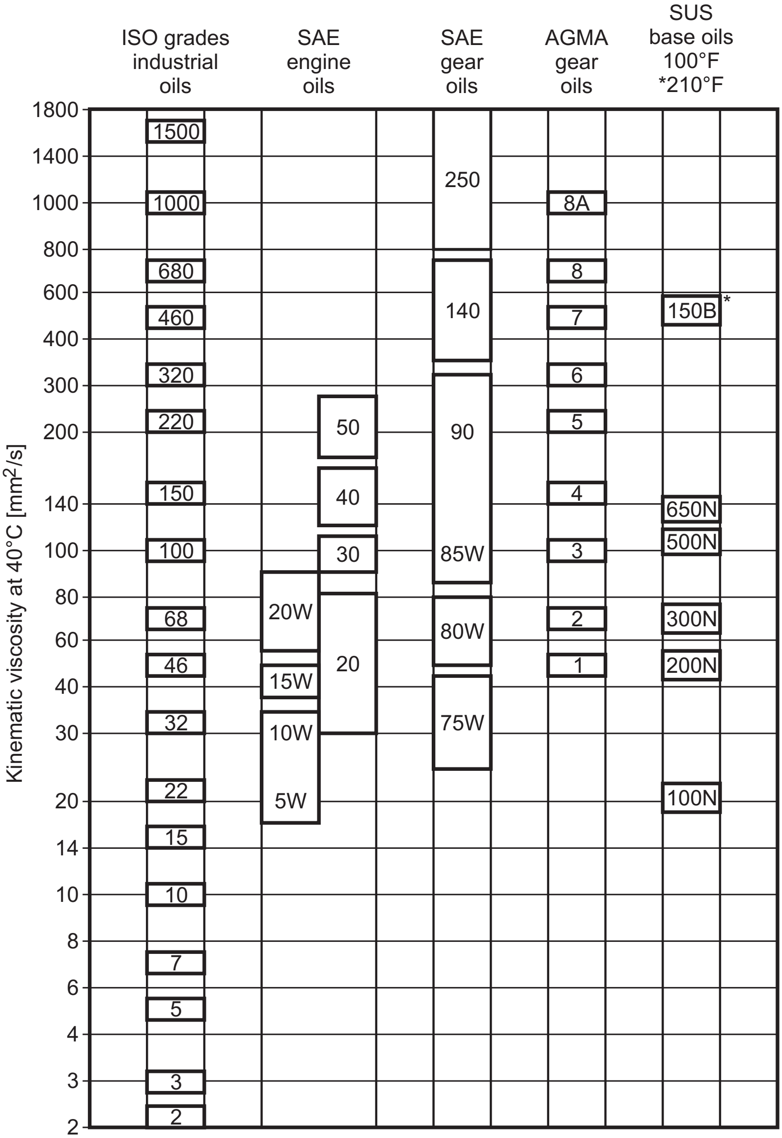

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**Fig. 3.8** Comparison of viscosity grades for various applications.

# 4
# Base Oils

*Theo Mang and Georg Lingg*

In terms of volume, base oils are the most important components of lubricants. As a weighted average of all lubricants, they account for more than 95 % of lubricant formulations. There are lubricant families (e.g. some hydraulic and compressor oils) in which chemical additives only account for 1 % while the remaining 99 % are base oils. On the other hand, other lubricants (e.g. some metalworking fluids, greases, or gear lubricants) can contain up to 30 % additives.

The origin of the overwhelming quantity of mineral lubricant base oils has led to lubricants being viewed as a part of the petroleum industry and this is underlined by their inclusion in petroleum statistics. Over the last few years, lubricants have increasingly become a separate discipline with clear differences from petroleum mass products. This was caused by the high added value which is generated in this product segment along with the fact that many high-performance lubricants no longer contain petroleum base oils.

## 4.1
## Base Oils – A Historical Review and Outlook

Although the most important requirement of base oils in the fifties was the correct viscosity and the absence of acidic components, base oils in the sixties were downgraded to solvents or carriers for additives in the euphoria surrounding chemical additives. In the seventies, there was a realization that some synthetic fluids with uniform basic chemical structures offered performance superior to that of mineral base oils. At that time, the considerably higher price of these products hindered their market acceptance. In the eighties however, lower-price, quasi-synthetic hydrocracked oils were introduced in Western Europe which closely matched the properties of synthetic hydrocarbons (Shell, BP, Fuchs). In the nineties, base oil developments were influenced by the ever-increasing demands on lubricant performance, and by environmental and health and safety criteria. This led to chemically more pure oils such as hydrocracked products, polyalphaolefins and esters gaining acceptance. Natural fatty oils, particularly their oleochemical derivatives, have experienced a renaissance because of their technical characteristics but, above all, because of their rapid biodegradability.

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The trend towards ever-greater performance and even better environmental compatibility continues in the first decade of the new millennium. The significantly higher price of the new lubricants, which will be increasingly characterized by their base oils and less so by their chemical additives, will probably be accepted by users who will benefit from long product life and lower overall system costs. In 2004, throughout the world, approximately 7 % (*w*/*w*) of base oils were synthetic products (including Group III hydrocracked oils; Section 4.6). This segment is forecast to grow to 10 % in 2015 [4.1]. In Germany, furthermore, approximately 5 % of lubricant base oils were rapidly biodegradable (natural and synthetic) esters in 2005. In addition, gasification of carbon-containing raw materials, for example natural gas, biomass, and carbon, will enable the production of synthetic lubricant base oils of high quality. Significant industrial-scale production of such base oils will start at the beginning of the second decade of the new millennium (Section 4.4.8).

## 4.2
## Chemical Characterization of Mineral Base Oils

The characterization of mineral oil fractions, whether crude oil or lubricant base oil fractions, by use of normal chemical practices to determine their exact structure is not possible without great expense. Crude oil generally consists of many thousands of single components and these are reflected in the processing of each fraction. It was, therefore, always an objective to describe mineral oil fractions by the comparatively simple expedient of defining their technical properties or to identify and quantitatively determine groups of components with similar chemical character. Advanced physicochemical methods are, however, increasingly being used in routine testing.

### 4.2.1
### Rough Chemical Characterization

#### 4.2.1.1 Viscosity–Gravity Constant (VGC)

This value enables only rough chemical characterization of oils. Values near 0.800 indicate paraffinic character whereas values near to 1.000 point to a majority of aromatic structure (ASTM D 2501-91)

#### 4.2.1.2 Aniline Point

The aniline point is also a help when characterizing the hydrocarbon structure of mineral oils. When aniline and oil are mixed and then cooled to a certain temperature (aniline point), two phases form. Because of their good solubility, aromatic structures give the lowest values.

### 4.2.2
### Carbon Distribution

The most important means of analysis for characterization of mineral oil hydrocarbons was, and remains, the determination of carbon in terms of its three categories of chemical bond – aromatic ($C_A$), naphthenic ($C_N$), and paraffinic ($C_P$).

N–D–M analysis uses physicochemical data which are easy to obtain. These include refractive index, density, and molecular weight. Molecular weight can be determined by measuring the viscosity at different temperatures (e.g. ASTM D 2502-92). Carbon distribution is given in % $C_{A,}$ % $C_N$ and % $C_P$ (100 % in total). N–D–M analysis also determines the average total number of rings per molecule ($R_T$) and the breakdown into aromatic and naphthenic rings ($R_N$) per molecule ($R_N = R_T - R_A$).

Brandes [4.2] created a method of determining carbon distribution according to specific bands in the infrared spectrum. The method has proven itself for lube base oils and can be performed at acceptable expense. Exact determination of aromatic carbon content can be performed by high-resolution nuclear magnetic resonance (NMR) (ASTM D 5292-91).

### 4.2.3
### Hydrocarbon Composition

A further refinement in the characterization of lubricant base oils is the determination of molecular families. Chromatography is used first to separate components and the fractions are then subjected to advanced analytical procedures.

To differentiate mineral oils including lube base oils in the boiling range from 200 to 550 °C, high ionizing voltage mass spectrometry (ASTM D 2786-91) is used for saturated fractions and ASTM D 3239-91 for aromatic fractions. Saturated fractions are separated into alkanes (0-ring), 1-ring, 2-ring, 3-ring, 4-ring and 5-ring naphthenes. The aromatic fractions are subdivided into seven classes: monaromatics, diaromatics, triaromatics, tetraaromatics, pentaaromatics, thiopheno aromatics, and unidentified aromatics.

### 4.2.4
### Polycyclic Aromatics in Base Oils

Polycyclic aromatics (PAH, polycyclic aromatic hydrocarbons, or, in general, PCA, polycyclic aromatics) are carcinogenic, environmentally harmful substances which are found in crude oils. In general they are not created when a lubricant is used. PAH formed by the combustion of gasoline can gather in engine oils. They can also gather in quenching oils after long periods of heavy-duty use.

In traditional solvent refining processes, PAH largely remain in the extract. Non-solvent extracted distillates contain PAH in line with their boiling point.

The carcinogenic characteristics of non-severely treated distillates in the petroleum industry was established by the IARC (International Agency for Research on Cancer) in 1983 [4.3]. This led to considerable limitations in the manufacture and application of naphthenic base oils and the ending of the use of aromatic extracts as lube base oils. Previously, Grimmer and Jakobs [4.4–4.6], in particular, confirmed the presence of PAH in lubricant base oils analytically and highlighted their carcinogenic characteristics.

The standardization of analytical methods of determining PAH and the setting of threshold values was the subject of lively discussions between 1985 and 1995. Whereas one group was primarily interested in establishing exact analyses and others wanted benzo(*a*)pyrene as a reference, another group was pressing ahead with the IP 346 method. This was later adopted into national legislation in several countries. PAH can be determined by HPLC with anthracenes or other aromatics as markers or by GC–MS after appropriate sample preparation.

The IP 346 method does not analyze PAH directly but an extract is obtained in DMSO (dimethyl sulfoxide), in which PAH accumulate. As a rule, the extract, which is often wrongly referred to as a PAH concentrate, largely contains naphthenes or mono-aromatics. According to IP 346, DMSO extracts contain only 0.1 % PAH. After numerous skin-painting tests on mice, the carcinogenicity of petroleum products corresponds to the percentage of DMSO extract.

Criticism of the IP 346 threshold value continues. The weakness of the value is clear when used gasoline engine oils are evaluated. The PCA enrichment caused by gasoline combustion is not considered by the IP method. For example, a 1000 ppm increase in PCA only increases the DMSO extract of the 'used' base oil from 1.0 (fresh oil) to 1.1 %.

Figure 4.1 illustrates the increasing carcinogenicity of petroleum products corresponding to the percentage of DMSO extract and various analytical methods for the determination of PAH content [4.7–4.11].

#### 4.2.4.1 **Aromatics in White Mineral Oils**

White oils which are used for pharmaceutical, medical or food processing applications should be free from aromatics, or at least low in aromatics, and preferably contain no traces of polycyclic aromatics. Ultraviolet absorption in the range 260 to

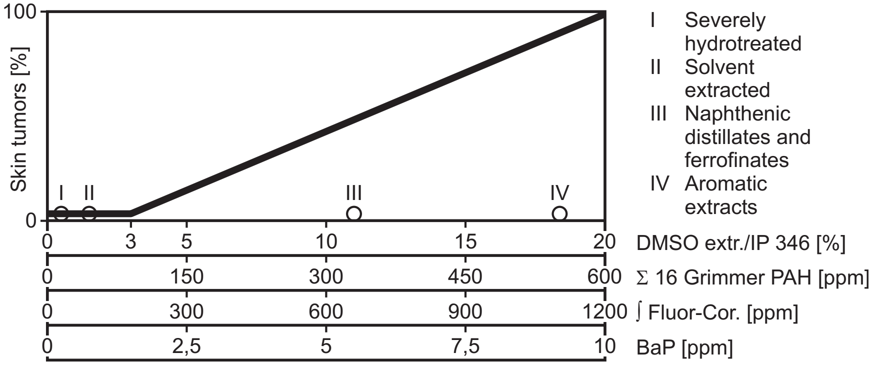


**Fig. 4.1** Dependence of the carcinogenicity of petroleum products on the PAH content (Mang 1989). Skin painting tests and rough statistical classification of different PAH analyses.

350 nm is used. Enrichment by extraction is worthwhile. ASTM D 2296-99 describes such a process including pre-enrichment with dimethyl sulfoxide extraction.

## 4.3
## Refining

Since the beginning of the petroleum industry, mineral oils have been used for lubricant base oils. The process of converting crude oil into a finished base oil is referred to as refining. As far as base oil manufacturing is concerned, the actual refining process begins only after the distillation stages. Refining is thus the term often used to describe all the manufacturing stages after vacuum distillation.

Lubricant refineries are divided into integrated and non-integrated plants (Fig. 4.2). Integrated refineries are linked to primary crude oil refineries and are fed with vacuum distillate by pipeline. Non-integrated refineries purchase vacuum distillate on the open market or buy atmospheric residues and perform their own vacuum distillation. Occasionally they perform vacuum distillation on crude oil.

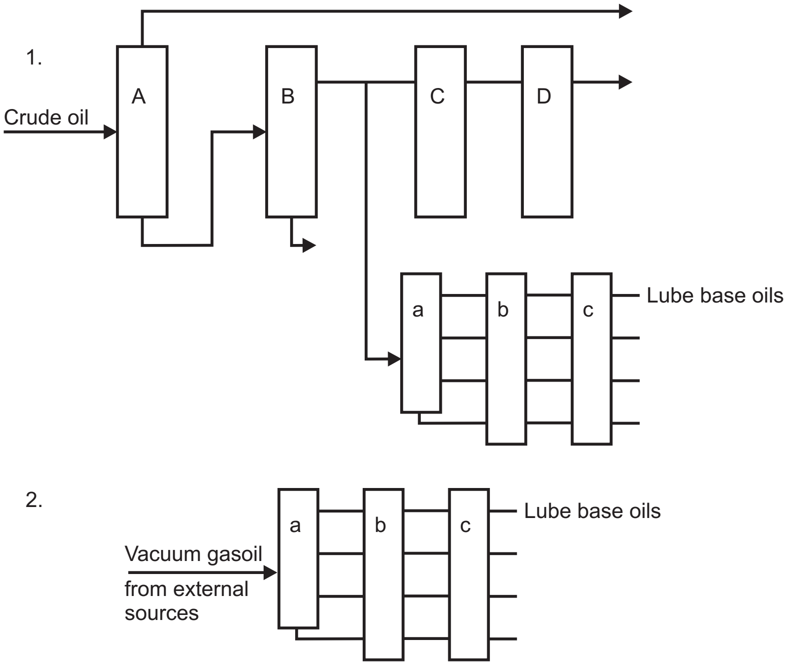


**Fig. 4.2** Integrated (1) and not integrated (2) lube refinery. A. Atmospheric distillation; B. Vacuum distillation; C, D. Processing of vacuum distillates for non-lube production. a. Fractionating vacuum distillation; b, c. Lube refining processes.

### 4.3.1
### Distillation

By way of fractional distillation, products are removed from crude oil which approximately meet the viscosity grades ultimately required. Often only four or five cuts suffice to fulfil lubricant requirements. As described later, the viscosity of the primary vacuum distillate is independent of the finished base oils in hydrocracking processes because the hydrocracking process creates new molecule dimensions.

After the corresponding separation of the lighter components from the crude oil by atmospheric distillation, the lubricant components are in the atmospheric residue. Figure 4.3 shows the yields of the various cuts in conventional lube oil refining with the corresponding boiling ranges of a typical lube crude. The atmospheric residue is now subjected to vacuum distillation to remove the components required for lubricants. In a vacuum, the boiling points of the heavier cuts fall so that distillation without thermal destruction (cracking) is possible.

### 4.3.2
### De-asphalting

Although the vacuum residue still contains highly viscous hydrocarbons which can supply valuable components for lube base oils, distillation cannot separate these from the asphalt which is also present and extraction processes must be used to separate these

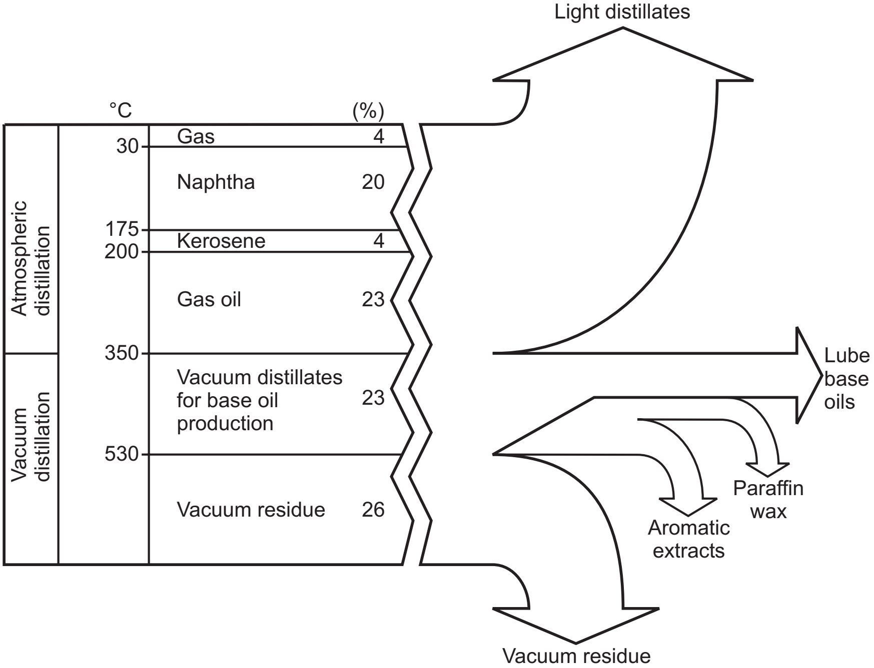


**Fig. 4.3** Yield of the various cuts in conventional lube oil refining of a typical lube crude.

highly viscous base oils, commonly known as brightstocks. Brightstocks are produced in lube oil refineries when the use of the asphaltene by-product (hard asphalt) is worthwhile. The quality of the hard asphalt for the manufacture of high quality bitumen depends on the crude oil. Extractive separation uses light hydrocarbons (propane to heptane), of which propane is the leading product for de-asphalting. Brightstocks can be manufactured with viscosities of more than 45 $mm^2\ s^{-1}$ at 100 °C.

Figure 4.4 shows a flow chart for the manufacture of suitable feeds for lube oil refining by distillation and propane de-asphalting. Figure 4.5 shows the de-asphalting process.

### 4.3.3
### Traditional Refining Processes

Vacuum distillation cuts principally determine the viscosity and flashpoint of later base oils. The precision of the fraction at the upper and lower boiling limits of a cut are of great importance.

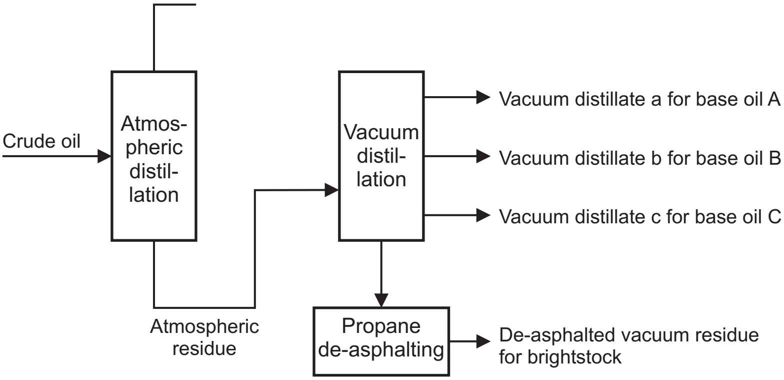


**Fig. 4.4** Flow chart for the manufacture of suitable feeds for lube oil refining by distillation and propane de-asphalting.

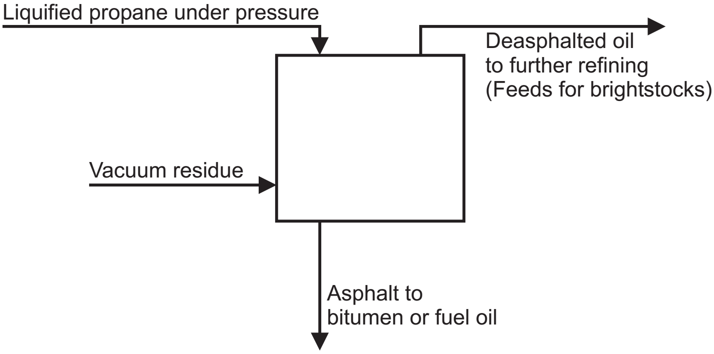


**Fig. 4.5** Flow chart of the de-asphalting process.

The distillates still contain components which can detrimentally affect aging, viscosity–temperature behavior and flowing characteristics, and components which are hazardous to health. To eliminate these disadvantages several refining methods were developed, of which solvent refining has become the most accepted method over the past few decades. Today, however, new plants increasingly use hydrotreatment.

#### 4.3.3.1 **Acid Refining**

Acid refining has become less popular because the acid sludge waste produced is difficult to dispose of and this method has been replaced by solvent extraction. Acid refining is still used to some extent for the re-refining of used lubricating oils (Chapter 8) and for the production of very light-colored technical or pharmaceutical white oils and petroleum sulfonates as by-products.

When the distillates are treated with concentrated sulfuric acid or fuming sulfuric acid (oleum), substances which accelerate oil aging are removed. Oleum treatment (wet refining) has a greater chemical effect on the structure of aromatics and not only readily removes reactive oil components such as olefins but also reduces the aromatic content, which in turn increases the viscosity index of the product. Reactions with saturated paraffinic structures lead to refining losses. Acid-refined oils require complex neutralization and absorption follow-up treatment to remove all traces of acid and undesirable by-products. Some lubricant specifications still require base oils to be free from acid even though modern base oils no longer come into contact with acids.

#### 4.3.3.2 **Solvent Extraction**

Whereas acid refining uses chemical reaction to reduce aromatic content and to eliminate reactive, oil-aging accelerators, solvent extraction is based on physico-chemical separation (Fig. 4.6). Solvent extraction creates base oils which are known as solvent raffinates or solvent neutrals (SN). Extraction processes using solvents create both a base oil and, after evaporation of the solvent, an aromatic-rich extract.

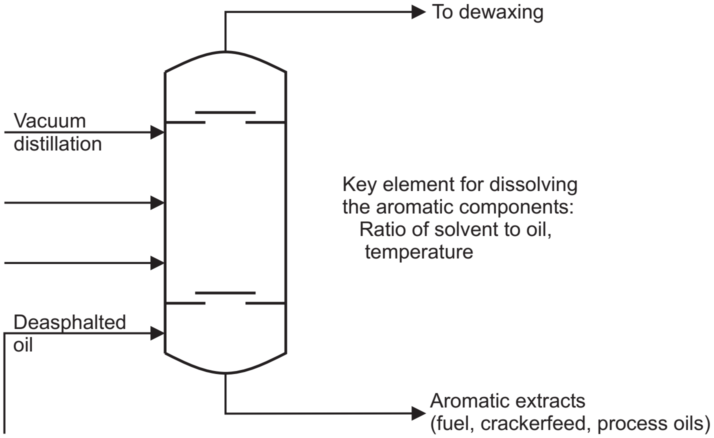


**Fig. 4.6** Solvent extraction.

The selectivity of the extraction media for aromatics is an important selection parameter.

In particular the selectivity towards polycyclic aromatics with three or more aromatic rings has attracted attention because of the carcinogenicity of these compounds. Numerous extraction media have been developed, of which furfural, NMP (*N*-methyl-2-pyrrolidone), and phenol have become economically significant. Sulfur dioxide ($SO_2$) should also be mentioned because of its historical importance as B. Edeleanu introduced extraction to petroleum technology in 1912. In 1999, sulfur dioxide was only used in very few refineries (one in Germany until 1999) for the refining of naphthenic distillates.

In recent years several furfural extraction plants have been converted into NMP plants, and even phenol plants have taken a back seat. NMP is a non-toxic solvent and can be used in a low solvent-to-oil ratio with high selectivity. This generates significant energy savings. NMP in new plants results in physically smaller units and thus lower capital expenditure.

As a rule, extraction plants are the first refining step for vacuum distillates because subsequent solvent dewaxing is the more complex refining process in terms of capital expenditure and operating costs. This route means that the extract part does not undergo unnecessary dewaxing.

Depending on the crude, the extract part of paraffinic oils can be 30 to 50 %. As the standard requirement of solvent neutral oils is a viscosity index (VI) of at least 95, the extraction severity is matched to this demand.

A higher proportion of aromatic and naphthenic hydrocarbons in the distillates requires greater extraction severity and thus a larger quantity of extract. The percentage share of the extract is a major economic factor in conventional lube refining. In general, extracts can only be used as products which are of lower value than the finished base oils. The large quantities are used as cracker feed and only a comparatively small proportion can be used as process oil. Their use as plasticizers has been severely limited in recent years because their carcinogenicity. The same applies to their use as lubricant base oils. Aromatic extracts have a very high viscosity–pressure coefficient (Chapter 3) and in the past were often used in metal-forming operations, such as cold extrusion.

A 1-point increase in viscosity index as a result of greater extraction severity creates, on average, 1 % more extract. Some refineries these days produce so-called semi-raffinates with VIs of between 70 and 80. It is cheaper to adjust the VI later with VI improvers than to produce 15 or 20 % more extract. These half-raffinates, however, have disadvantages resulting from the lower extraction severity – e.g. lower oxidation stability, possibly higher amounts of sulfur or higher amounts of polycyclic aromatics.

Solvent extraction is generally only economic to a minimum VI of 50 of the vacuum gas oil [4.12]. Figure 4.7 shows the average yield of waxy base oils by solvent extraction to achieve 100 VI. It must, however, be remembered that subsequent solvent dewaxing further cuts the yield by approx. 20 %.

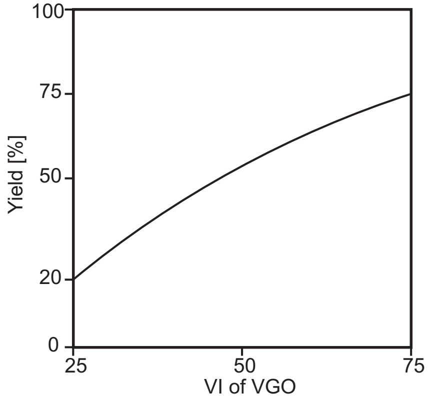


**Fig. 4.7** Average yield of waxy base oils in solvent extraction to get 100 VI.

### 4.3.4
### Solvent Dewaxing

In traditional refining processes, solvent extraction is followed by solvent dewaxing. Long-chain, high melting point paraffins negatively affect the cold flow properties of lube oil distillates and lead to a high pour point. This is caused by the crystallization of waxy substances at low temperatures which results in turbidity and an increase in viscosity. Their removal has, therefore, been an important consideration since the beginning of crude oil refining.

Dewaxing by crystallization of paraffins at low temperatures and separation by filtration are the principal processes in traditional refining. Compared with catalytic dewaxing with hydrogen, urea-dewaxing to separate *n*-paraffins is of relatively minor importance in lube oil refining. Crystallization methods involve mixing the solvent with the oil; this improves filtration, as a result of dilution, and promotes the growth of large crystal formations.

The important solvents are ketones and chlorinated hydrocarbons. Dewaxing with ketones (dimethyl ketone, methyl ethyl ketone, MEK) is normally used for pour points down to –12 °C. For lower pour points, the Di–Me (dichloroethane–dichloromethane mixtures) method is used. This also enables the manufacture of hard and soft waxes. As for the extracts created by solvent extraction, the by-product situation is again problematic. The paraffins are only worthwhile if their quality and processing is good and they can then be sold as candle wax, for coatings, and for other purposes. They are often used as cracker feed in fuel catalytic crackers (FCC) and the cracker yield is then assessed by use of special formulas.

Figure 4.8 illustrates the conventional solvent dewaxing process.

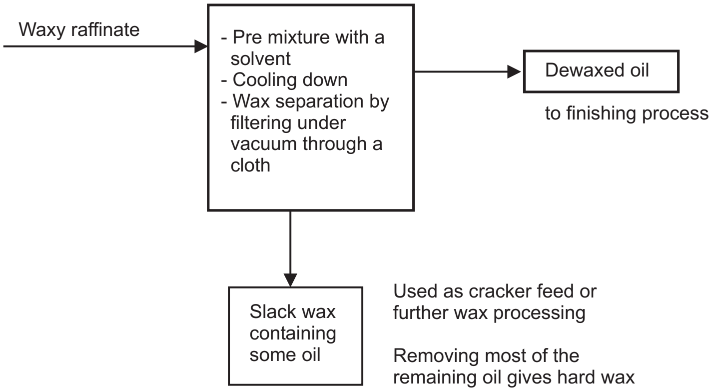


**Fig. 4.8** Illustration of the conventional solvent dewaxing process.

### 4.3.5
### Finishing

A finishing stage often follows the two refining stages of extraction and dewaxing. In the past, methods which employed absorbents were often used but these days the processes almost all use hydrogen (hydrofinishing). Finishing should improve the color of the product and remove surface-active substances which can negatively affect the air-release or demulsifying properties of a lubricating oil.

Depending on the temperature, pressure, catalyst, and space velocity of the hydrofinishing, a more or less severe hydrogenating process can be initiated. In general, the hydrofinishing process is referred to as mild hydrotreating and focuses on improving color, odor, and ultra-violet stability. Ferrofining (BP) has achieved major economic importance as a finishing process. The process parameters do not generally lead to de-sulfurization. More severe processes with pressures in excess of 100 bar can bring about significant de-sulfurization and some de-aromatization.

Additional finishing with absorbents (bleaching clays, bauxite) is sometimes used for the manufacture of refrigerator, transformer, or turbine oils. The subsequent filtration process in filter presses or other filtering equipment represents additional complexity and the disposal of the filter residue is an increasing problem.

#### 4.3.5.1 Lube Crudes

Crude oils which yield high-grade base oils are preferred for the production of lube base oils. Of importance to the vacuum gas oils (VGO) which are derived from the crude oil and which are the direct feeds for lube refining are, particularly, VI, wax content and sulfur content. A high VGO VI leads to low extraction losses in solvent extraction processes and low hydrogen consumption during hydrogenating processes. High wax contents increase production costs, as a result of the high operating costs of the dewaxing plant, as well as reducing base oil yield. In a base oil production unit using a wax isomerization process, high wax content can also contribute to a higher VI. The finished base oils have a high sulfur content, especially after conventional solvent refining. Hydrogenating processes almost fully eliminate sulfur but desulfurization consumes hydrogen.

Table 4.1 shows the VI and wax content of VGO from various lube crudes.

**Tab. 4.1** VI and wax content of vacuum gas oil (VGO) from various crudes.

| *Crude* | *VI after solvent dewaxing* | *Wax in VGO (%, w/w)* |
|---|---|---|
| Pennsylvania | 100 | 0 |
| Ordovician | 85 | 13 |
| Brent | 65 | 19 |
| Arab light | 60 | 9 |
| Arab heavy | 55 | 9 |
| Iranian light | 55 | 16 |
| Lagomedio | 45 | 10 |
| Urals | 40 | 11 |
| Iranian heavy | 40 | 16 |
| ANS | 15 | 8 |

## 4.4
## Base Oil Manufacturing by Hydrogenation and Hydrocracking

Traditional solvent refining is the separation of unwanted components from vacuum distillates. Hydrogenation and hydrocracking in the manufacture of lubricant base oils significantly influence the chemical structures of mineral oil molecules. On one hand, unstable molecules are chemically stabilized by the removal of the heteroatoms (sulfur, oxygen, nitrogen) and on the other, severe hydrogenation can convert aromatics into saturated naphthenic or paraffinic structures. In addition to the hydrogenation process, hydrocracking breaks-down or cracks larger molecules into smaller ones. Larger molecular structures can re-form from small fragments. The principal process criteria are temperature, pressure, the catalyst and space velocity. If special conditions are met, a focal point of the process is the isomerization of paraffinic structures. Besides the saturation of aromatics, opening of the naphthenes rings can occur.

It is clear that lubricant base oils can be much more easily tailored using these processes than is possible with simple solvent refining separation. The future of lube base oil production thus lies with hydrogenation and hydrocracking. An additional advantage of advanced hydrocracking is the lower dependence on the quality of the crude oil. Although the economic boundaries of solvent refining are set by yield (extract and paraffin quantities), altering hydrocracking process parameters can compensate for varying crude oil qualities.

The roots of manufacturing lubricant base oils by hydrogenation lie in previous attempts to liquefy coal by high-pressure hydrogenation. On the basis of the results of Bergius (1913, Hanover, Germany), the first technical plant began operations in 1921 in Mannheim–Rheinau, Germany. Lubricating oil cuts were, however, inferior to those obtained from petroleum. The combination of results with the above and experience from ammonia synthesis of the high pressure behavior of hydrogen (1910) and methanol synthesis of carbon dioxide and hydrogen (1923, 200 bar, BASF Ludwigshafen, Germany) formed the basis of the catalytic high-pressure hydrogenation of coal, tars, and petroleum products. Key contracts between the German IG and the American Standard Oil Co. of New Jersey for the hydrogenation of petroleum in 1927 and 1929 led to the construction of two large-scale hydrogenation plants in Baton Rouge, Louisiana and Baybay, New York in 1930 and 1931. Apart from light products, 'Essolube' lubricant base oils were thus created [4.13].

Disregarding finishing processes and white oil production, high-pressure hydrogenation processes to create lubricant base oils from petroleum became fashionable again only in the early seventies. In the German Democratic Republic, lubricant base oils were manufactured by the high-pressure hydrogenation of lignite tars up to the early nineties.

In addition to hydrocracking and hydroisomerization, the following typical chemical processes occur during hydrogenation (Table 4.2).

While the processes mentioned are generally replacing solvent extraction for the manufacture of base oils, there are hydrogenation processes which either crack long-chain paraffins with specific catalysts into light products and remove them from the base oil or convert them into isoparaffins with good low-temperature characteristics without a significant loss in yield. If one combines the previously mentioned hydrogenation processes with this type of catalytic hydrogenating dewaxing, this is called the 'all-hydrogen route'.

### 4.4.1
### Manufacturing Naphthenic Base Oils by Hydrogenation

Only about 10 % of the petroleum base oils used in lubricants are naphthenic. Before 1980, naphthenic oils were significantly more important (USA 25 %). In 1983, a publication by the IARC (International Agency for Research on Cancer) defined a number of mineral oil products as carcinogenic. None of these was a severely treated distillate. Naphthenic oil, which normally has good natural low-temperature properties, did not need to be dewaxed and it was possible to convert the vacuum distillates into low viscosity index (LVI) and moderately good aging behavior lubricant base oils by simple refining (acid refining or hydrofinishing). In the past these base oils were much cheaper than paraffinic solvates and this also explained their popularity. Although the IARC publication and subsequent legislation and classification requirements led to the disappearance of this type of oil from important markets, naphthenic base oils were still required for some lubricant applications (greases, metalworking oils, and refrigerator oils) because of their outstanding solubility and these are now manufactured with severe hydrogenation which produces non-carcinogenic base oils. Solvent extraction (e.g. the Edeleanu process) with its high proportion of extracts and low yields is now less advantageous. Figure 4.9 shows a flowchart for the manufacture of naphthenic base oils. Severely hydrotreated Group C distillates are the most accepted naphthenic base oils in western markets [4.14].

**Tab. 4.2** Typical chemical processes during hydrogenation.

Desulfurization

Mercaptans

$R-SH \longrightarrow RH + H_2S$

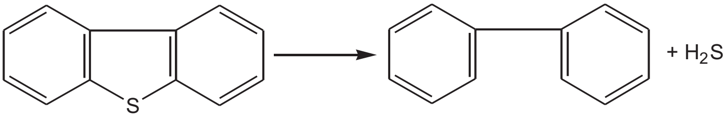
Disulfides

$R-S-S-R \longrightarrow RH + H_2S$

Sulfides

$R-S-R \longrightarrow RH + H_2S$

Thiophenes



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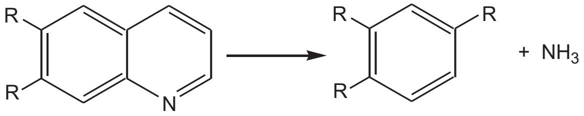
Denitrogenation



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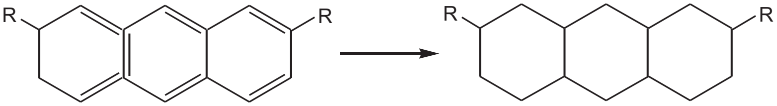
Polyaromatic saturation



| | | |
|---|---|---|
| Viscosity Index: | –60 | 20 |
| Pour point: | +50 °C | +20 °C |

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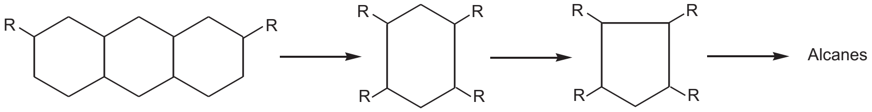
Naphthenic ring opening



| | | |
|---|---|---|
| Viscosity Index: | 20 | ~130 |
| Pour point: | +20 °C | –10 °C |

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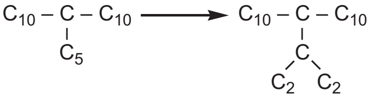
Isomerization



$$C_{10}-\underset{\displaystyle C_5}{\underset{|}{C}}-C_{10} \longrightarrow C_{10}-\underset{\underset{\displaystyle C_2\quad C_2}{\underset{/\ \backslash}{}}}{\underset{\displaystyle C}{\underset{|}{C}}}-C_{10}$$

| | | |
|---|---|---|
| Viscosity Index: | 125 | 125 |
| Pour point: | +20 °C | –40 °C |

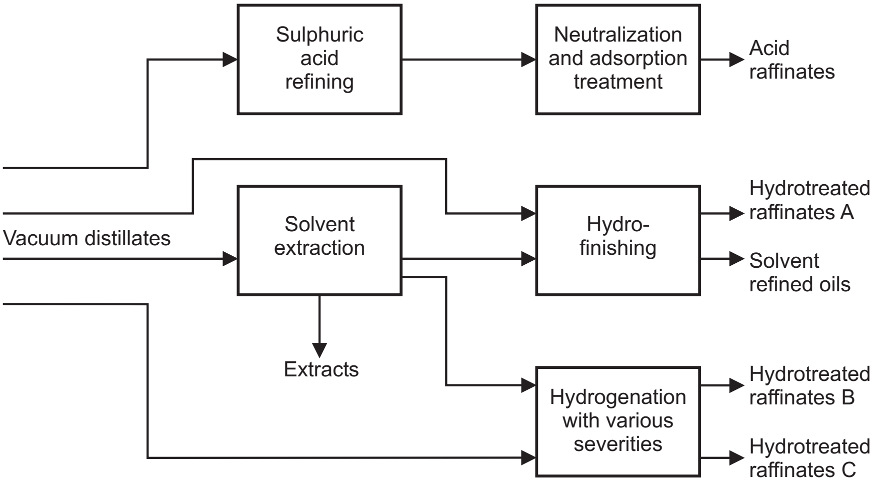


**Fig. 4.9** Manufacture of naphthenic base oils.

### 4.4.2
### Production of White Oils

The large-scale production of white oils by high-pressure hydrogenation are part of the first steps in the manufacture of lubricant base oils by hydrogenation. White oils – light-colored, odorless, low-aromatic or aromatic-free mineral oil raffinates – are used in a number of applications as medical and pharmaceutical white oils or technical white oils in, above all, the food and beverage industries as food-grade lubricants. BASF Germany was the first to develop white oil manufacturing from the high-pressure hydrogenation technology used to liquefy coal. Up to that point, white oils were manufactured exclusively by acid treatment.

The feeds for hydrogenated white oil manufacturing are generally naphthenic or paraffinic solvent-treated vacuum distillates or untreated vacuum distillates. In the BASF process hydrogenation takes place in two stages: the first at 300–380 °C and 80–200 bar with a NiMo catalyst and the second at 200–300 °C and 100–200 bar with an Ni catalyst. The first stage produces the technical qualities (according to the US

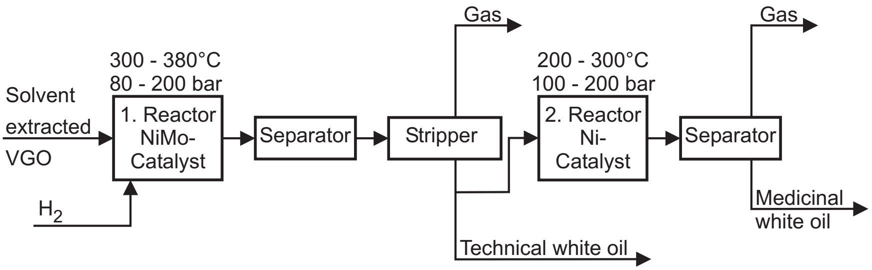


**Fig. 4.10** Flow chart for white oil manufacture [BASF].

Food and Drug Administration) while the second stage produces the medical grades (e.g. German Pharmacopoeia). Figure 4.10 shows a flow chart for white oil manufacture [4.12].

### 4.4.3
### Lube Hydrocracking

The principal elements of the lube hydrocracking process are the cracking of low-VI components and the saturation of aromatics.

Hydrocracking as a major method of future mineral base oil manufacture can be performed in two types of refinery. The first is a lube oil refinery which operates a hydrocracker with the principal objective of producing lubricating oils and the second is a refinery which operates a fuel hydrocracker to convert vacuum gas oil into high-grade fuels. Fuel hydrocracker residues are excellent feeds for the manufacture of lubricant base oils and hydrocrackers used to make olefin feeds for steam crackers can supply a premium quality feed for lube base oils. The severity has the most important effect on the quality of the base oil produced. High severity (e.g. 80 % light products) generates a high-VI and low-evaporation oil. While hydrocracking refineries for base oils with normal VIs and normal Noack evaporation (HVI oils, Group II oils, Section 4.6) at moderate severity are being built in the USA and the Far East, based mainly on Chevron Technology, Western European hydrocracking plants were designed for low-evaporation base oils (Shell, Petit Couronne, France, 1972; Union FUCHS, Wesseling, Germany, 1986 and BP, Lavera, France). These produce Group III oils, on one hand from waxes and on the other from vacuum gas oils with hydrocracker residues.

Hydrocracked base oils differ from solvent-extracted oils by their extremely low aromatic content and their chemical purity, i.e. only traces of heteroatoms such as sulfur, nitrogen, or oxygen. Furthermore, Group III oils which were manufactured at high severity or from waxes have Noack evaporation characteristics which are about 50 % down on equiviscous, solvent-extracted vacuum distillates. If catalytic dewaxing is performed, even lower pour points can be achieved than with SN oils.

Since 1995, more than half of all new base oil manufacturing plants have been built or are planned using hydrocracking technology

Apart from high-value base oils, sufficiently large hydrocracking plants offer favorable operating costs and greater crude oil flexibility, despite high initial investment costs. A lube oil hydrocracking plant which operates at approx. 50 % vacuum distillate severity generates just as many high-quality light products as lube base oils while, at present, just as many low-value extracts and paraffin waxes are generated by solvent extraction.

To increase VI from 100 to 125, the hydrocracking severity has to be increased so much that up to one half of the base oil yield is lost [4.12].

The increasing number of fuel hydrocrackers, which are a response to increasing demands for low-sulfur oils (diesel fuels with < 50 ppm sulfur), represents a growing source of hydrocracked feeds. Fuel hydrocracker residue can be processed into high-quality, hydrocracked base oils by distillation and subsequent dewaxing (wax isomerization) at acceptable cost.

During the hydrocracking process, especially if vacuum gas oils are severely treated to create very high viscosity (VHVI) oils, polycyclic aromatics can be formed along with saturated structures (aromatics to naphthenes or paraffins and isoparaffins) under some process conditions. These have to be removed by subsequent high-pressure hydrogenation or by extraction if hybrid methods are used. This subsequent hydrogenation can also be manipulated so that it also produces a significant increase in VI.

### 4.4.4
### Catalytic Dewaxing

The most complex stage of traditional base oil refining is solvent dewaxing. In addition to the high capital expenditure and, above all, operating cost, the solvent-related limits to the achievable pour point are a disadvantage.

In recent years various methods have been developed which can remove unbranched, long-chain or short-chain paraffins, less branched paraffins, and some other petroleum components by catalytic and hydrogenation reactions or convert them into such components which improve the low-temperature characteristics of the base oil.

The first technologies were based on the catalytic cracking of these above-mentioned substances. In 1979, Mobil introduced the ‘Mobil Lube Dewaxing Process’ (MLDW). The development of catalysts since then has led to strong hydroisomerization activity processes (Mobil selective dewaxing, MSDW).

While the MLDW process mainly involves the cracking of long-chain paraffins and the production of larger quantities of light by-products and poor base oil yields, MSDW processes create high-yield, non-waxy isoparaffins from the waxy parts of the feed.

At the beginning of the nineties, Chevron successfully introduced its isodewaxing process. In 1999 it is the most important technology for catalytic dewaxing [4.12]. The process combines relatively high yields with low pour points and high VIs. The method uses a hydrogenation component as an intermediate pore-silicoaluminum-phosphate molecular sieve (e.g. Pt on SAPO-11). In terms of process economy, high base oil yields but also the creation of mainly high-value $C_5$+ liquids as by-products are of significance. In particular, the formation of low-value by-products such as propane is avoided.

In the development of new dewaxing catalysts, it is important to achieve the right balance between hydrogenation activity and acid activity. Increasing hydrogenating properties usually leads to a reduction in isomerization and thus a worsening of the pour point. Higher acid activity increases cracking and thus yield losses. Sulfur and nitrogen in the feed play important roles. Nitrogen is detrimental to acid activity and sulfur is poison for catalysts for metal hydrogenation components.

Although isodewaxing enables manufacture of lubricant base oils with pour points below –45 °C, with catalytic dewaxing processes one must never forget that dewaxing to very low pour points (depending on process conditions and catalysts) leads to VI losses.

A major success of this new dewaxing process are the molecular sieve catalysts – zeolites with exactly defined mesh sizes. Figure 4.11 illustrates the procedure of catalytic dewaxing with zeolite catalysts.

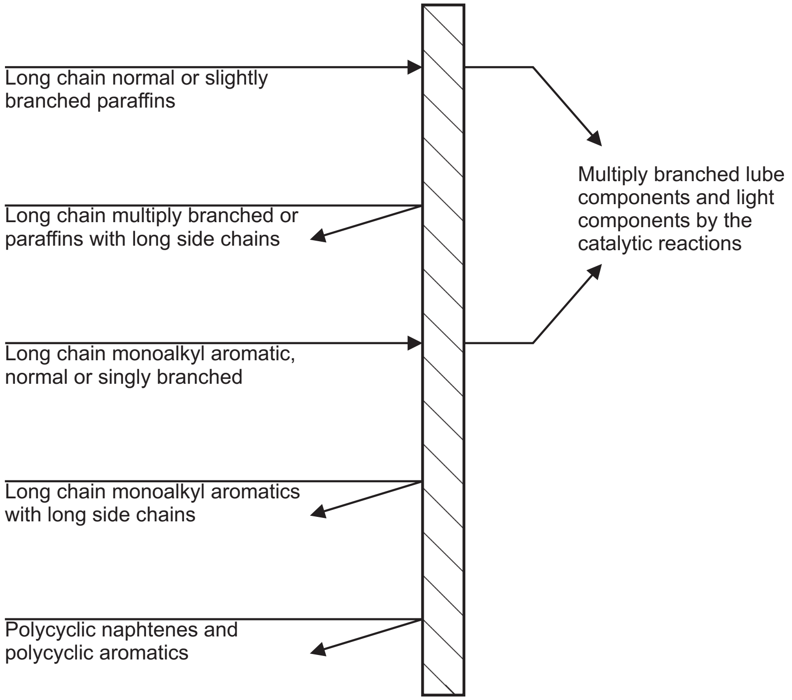


**Fig. 4.11** Catalytic dewaxing with zeolite catalysts.

Although highly branched paraffins or polycyclic naphthenes or aromatics are not affected by the zeolites, the long-chain less or unbranched molecules are acted upon by the catalysts [4.15–4.17].

Figure 4.12 shows the flow chart for catalytic dewaxing (hydrodewaxing) including a hydrofinishing reactor.

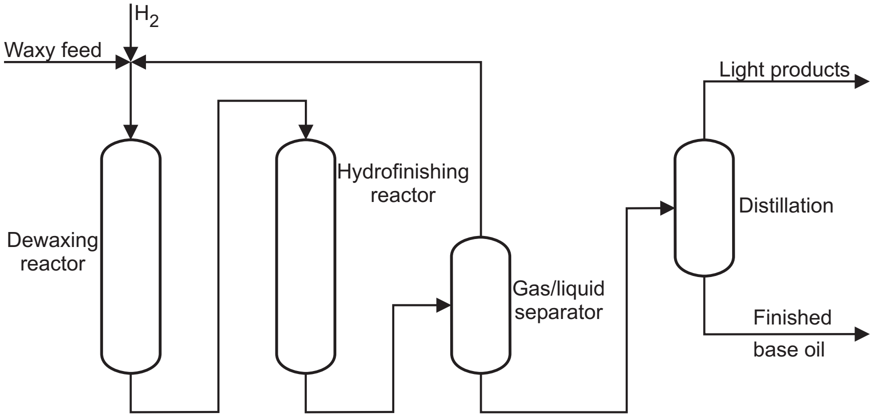


**Fig. 4.12** Flow chart for catalytic dewaxing (hydrodewaxing) including hydrofinishing.

### 4.4.5
### Wax Isomerization

VHVI oils have been manufactured from waxes since the early seventies. Feeds were wax cuts from solvent extraction processes. The conversion of long-chain normal paraffins or less branched paraffinic components into isomerized products with good low-temperature characteristics and high VIs succeeded, with suitable catalysts, in isomerizing hydrocracking processes. Because the input feed is already aromatic- and naphthene-free, the catalysts can be fully optimized to the conversion of the paraffinic material. At relatively high yield losses, the process creates high-quality but comparatively expensive hydrocracked oils (Shell XHVI, Exxon Exxyn, Mobil MWI-2 catalyst). Figure 4.13 shows the manufacture of XHVI oils by wax isomerization and hydrocracking, including solvent dewaxing.

### 4.4.6
### Hybrid Lube Oil Processing

The combination of traditional solvent refining with severe hydrotreating and hydrocracking processes is known as hybrid processing. On one hand, such processes should represent an extension of existing refineries operations, and on the other, exploit the favorable properties of hydrogen processes. To create higher VIs (> 105) and to reduce sulfur content, hydrotreating can follow solvent extraction (e.g. in the manufacture of turbine oils).

The combination of a mild furfural extraction with hydrocracking can produce hydrocracked oils by low-hydrocracking conversion in small hydrocracking reactors. The low hydrogen consumption adds to the economy of such processes given that the extraction plant exists and the hydrocracker can be integrated into the infrastructure of a lube oil refinery. The introduction of a hydrocracking stage into a conventional solvent refinery offers the attractive possibilities of de-bottlenecking if the corresponding dewaxing capacity is available.

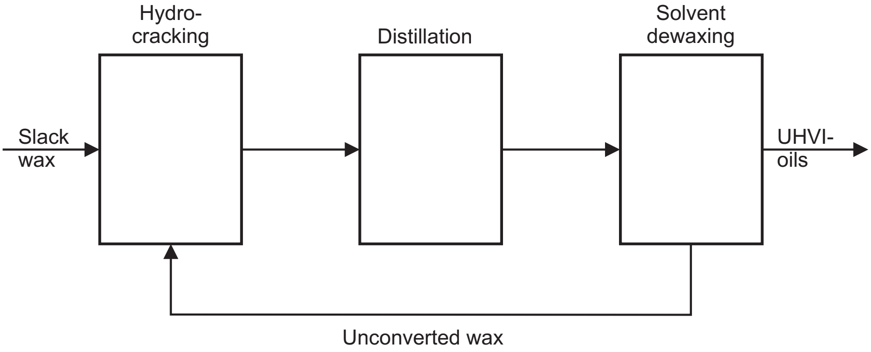


**Fig. 4.13** Process for the production of ultra-high-viscosity index oils by wax hydrocracking and isomerization.

Catalytic dewaxing can, on the other hand, follow solvent refining. This presupposes that the catalytic dewaxing catalysts can withstand, and are not poisoned by, the sulfur and nitrogen components in the solvent raffinate. In the Mobil processes, this is more likely with MLDW catalysts than with MSDW catalysts.

In connection with the isomerization of slack waxes from petroleum distillates, several other high-paraffin components should be considered as future base oil feeds. These could include high-wax (> 70 %) natural distillates and Fischer–Tropsch waxes or synthetic fluids from natural gas. Significant future importance is given to this latter group of products.

#### Hystart

The quality of low-quality lube feeds (VGO) can be improved by hydrogenation before solvent extraction. This process is called Hy-starting or Hystart.

### 4.4.7
### All-Hydrogen Route

The production of base oils by hydrocracking and catalytic, solvent-free dewaxing is called the all-hydrogen route. Figure 4.14 shows a flowchart for such a refinery used to manufacture VHVI oils. Depending on the severity of the hydrocracker, Group II HVI or Group III VHVI oils can be produced (HC-I and HC-2 oils, Table 4.3). Leading examples for HC-I oils are the Chevron refinery in Richmond, California and the Conoco–Pennzoil refinery in Lake Charles (Exel Paralubes) [4.18]. Some HC-I refineries have been prepared to produce HC-2 oils by the all-hydrogen route by increasing the severity of the hydrocracker. Another example of a hydrocracker refinery is the Petro-Canadas plant, Fig. 4.15 [4.19].

**Tab. 4.3** API/ATIEL classification of base oils.

| *Group* | *Sulfur (% w/w)* | | *Saturates (% w/w)* | *Viscosity index* |
|---|---|---|---|---|
| I | > 0.03 | and/or | < 90 | 80–120 |
| II | ≤ 0.03 | and | ≥ 90 | 80–120 |
| III | ≤ 0.03 | and | ≥ 90 | > 120 |
| IV | All polyalphaolefins (PAO) | | | |
| V | All base oils not included in Groups I–IV or VI | | | |
| VI | All polyinternalolefins (PiO) | | | |

The use of a fuel hydrocracker for the production of base oils using the all-hydrogen route was first realized by the SK Corporation (Ulsan, Korea) in 1995. By recycling the hydrocracker bottom and the special integration of the fuel hydrocracker in the lube oil process, SK also developed a specific method (UCO process, Fig. 4.16) [4.20].

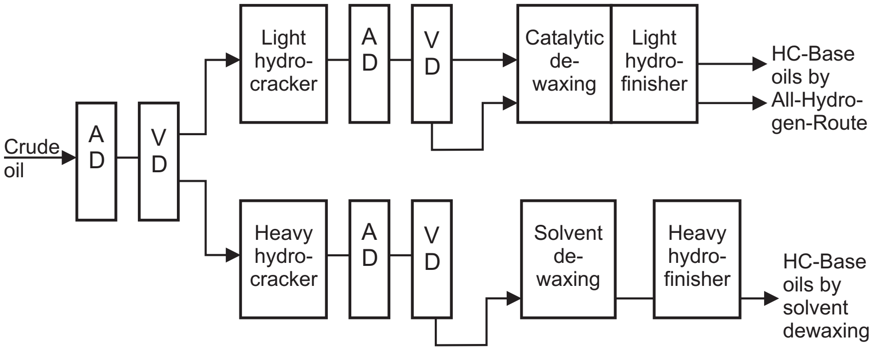


**Fig. 4.14** All-hydrogen route in Chevron's Richmond (California, USA) refinery.

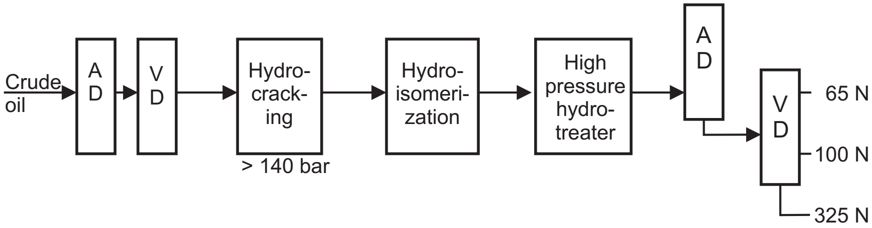


**Fig. 4.15** Lube base oil production with severe hydrocracking and hydroisomerization (All-hydrogen route, Petro-Canada)

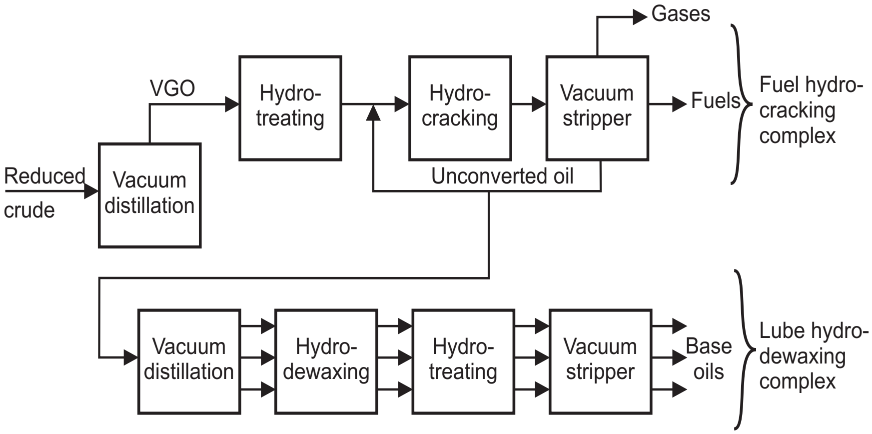


**Fig. 4.16** VHVI base oil production based on fuel hydrocracker residue (SK Corporation, Korea).

### 4.4.8
### Gas-to-Liquids Conversion Technology

As a result of efforts to increase the value of natural gas in logistically favorable locations, the chemical liquefaction of natural gas (also the chemical reaction route) was developed (on the basis of the Fischer–Tropsch process). This process creates high-quality liquid products and paraffin wax. High-quality UHVI oils can then be obtained from natural gas by part oxidation, polymerization, and isomerization, Fig. 4.17 [4.21,4.22].

The base oil market could undergo dramatic changes if Fischer-Tropsch waxes, which are part of gas-to-liquids technology, become more generally available. The 80-year-old Fischer-Tropsch technology has attracted considerable attention in the last few years. The focus of this attention is the better utilization of natural gas. Syngas (CO and $H_2$) is made from methane, oxygen (air) and water vapor and this, in turn, is made into fluid and solid hydrocarbons in the Fischer-Tropsch reactor. The solid hydrocarbon waxes (> 99 % paraffins) are hydrocracked, hydro-isomerizated and iso-dewaxed into super-clean base oils. Shell has used such waxes from its Bintulu plant in Malaysia for its XHVI's. New technologies for smaller, efficient plants have been developed by Rentech and Syntroleum in the last years.

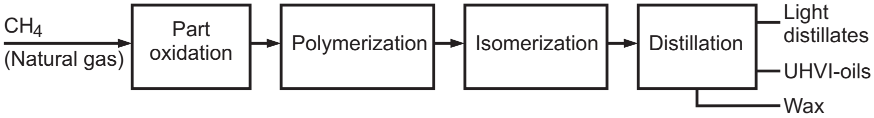


**Fig. 4.17** UHVI lube base oil production with gas-to-liquids conversion technology.

It is, nevertheless, most likely that major oil companies will be the first to operate large-scale gas-to-liquid (GTL) plants. ExxonMobil, Shell, and SasolChevron have each announced GTL projects including base-oil-production units in Qatar [4.25]. These plants start production between 2009 and 2012.

GTL base oils will have premium characteristics, including very high viscosity indices, essentially no sulfur and nitrogen, very low evaporative losses, and almost no aromatic content [4.26]. They will most probably be classified as Group III+ base oils, because their characteristics will vary between UHVI base oils (Group III) and polyalphaolefins (Group IV).

Besides natural gas, all carbon containing materials can, in principle, be used for production of liquid products and paraffin wax by Fischer–Tropsch technology. Because of the limited availability of crude oil, gasification and liquefaction of carbon, biomass, and even oil sands [4.27] are of increasing interest.

### 4.5
### Boiling and Evaporation Behavior of Base Oils

In traditional solvent refining, the boiling point distribution of base oils is largely determined during vacuum distillation. Three, four or five (including bright stock) cuts are taken whose boiling point distribution is reflected in the finished base oil.

Lighter products can only be created during hydrofinishing as the final refining stage. Components which negatively affect the evaporation behavior of the base oil can remain if these light components are not fully stripped.

In hydrocracking processes, the decisive distillation stages take place after cracking or after catalytic dewaxing. The function of the stripper or fractional distillation columns play an important role whenever light cracked products are created.

The target of base oil distillation is the viscosity desired at 40 °C and 100 °C. The same distillation cut (same boiling point distribution) with different chemical structures leads to different viscosities. A highly naphthenic cut produces a higher viscosity than a paraffinic cut. In other words, equiviscous cuts of different chemical structures have different boiling-point distributions.

In practice, ISO viscosity grades or other required viscosities are created by blending different cuts. If the boiling points of both cuts are too far apart, the flashpoint (Chapter 18) drops significantly and evaporation increases. Flashpoint and evaporation are generally determined by the base oil components with the lowest boiling points. Figure 4.18 shows the distillation curves of three paraffinic solvent refined cuts. One line is a blend of cut A with cut C which creates an equiviscous oil with cut B. The share with a boiling range up to 380 °C increases from 10 % (equiviscous cut) to 20 % (equiviscous blend).

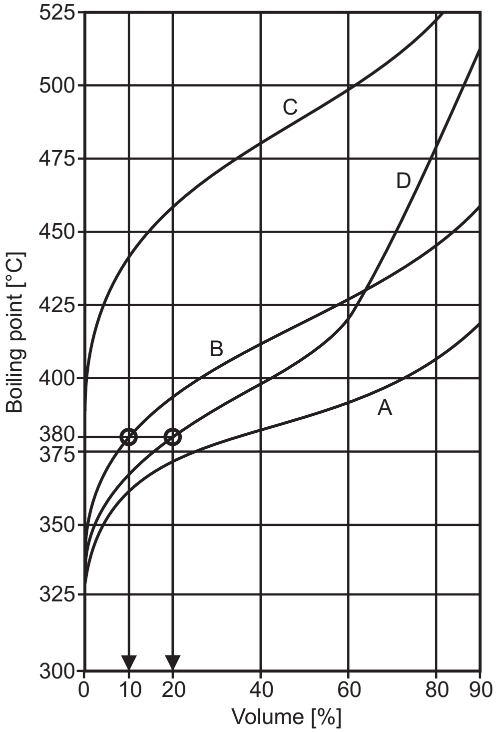


**Fig. 4.18** Distillation curves of three paraffinic solvent refined cuts. A. Light cut (low boiling); B. Medium light cut; C. Heavy cut (high boiling); D. Blend of A and C, equiviscous with B.

Apart from the use of a variety of laboratory distillation apparatus to determine the boiling point distribution of a base oil, gas chromatographic methods of determining boiling point have gained popularity over the last few years. The use of gas chromatography to determine the boiling range has developed continuously, especially as a result of improvements in columns and software, and has found its way into various standards. Both non-polar packed and capillary gas chromatographic columns can be used. The injection port temperature is between 360 and 390 °C depending on the length of the column. The initial column temperature can be as low as –50 °C, the final column temperature is between 360 and 390 °C and the programming rate is usually below $10^0$ $\text{min}^{-1}$. The maximum temperatures stated are the decomposition thresholds of the fractions being tested or of the column material.

Figure 4.19 shows a typical calibration curve (ASTM 2887-93). A mixture of normal paraffins including *n*-$C_5$ to *n*-$C_{44}$ is used for calibration. The boiling points of *n*-paraffins are: *n*-$C_{16}$ 287 °C, *n*-$C_{30}$ 449 °C, *n*-$C_{44}$ 555 °C. ASTM D 5307-92 describes another method of determining boiling-range distribution by gas chromatography.

Figure 4.20 shows the first application of high-temperature gas chromatography to determine the boiling point distribution of high-boiling-point coal tars [4.23]. Normal paraffins in an non-polar column were used for reference. Figure 4.21 shows the distillation curve of a mixture of two solvent-refined cuts determined by gas chromatography and the latest software.

In recent years, the evaporation characteristics of lubricants have become increasingly important quality criteria. The reasons for this are the emissions created when a lubricant evaporates, and the accompanying change to the lubricant's composition. The topic of evaporation behavior has become increasingly important in line with the trend towards lower viscosity oils for most applications (energy-saving oils).

Evaporation is dependent on the vapor pressure of the base oil components at a given temperature and the ambient conditions (such as atmospheric pressure, turbulence

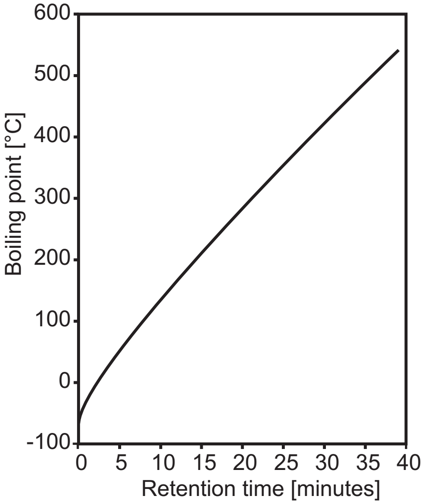


**Fig. 4.19** Typical calibration curve for determination of the boiling-point distribution of lube base oils by gas chromatography.

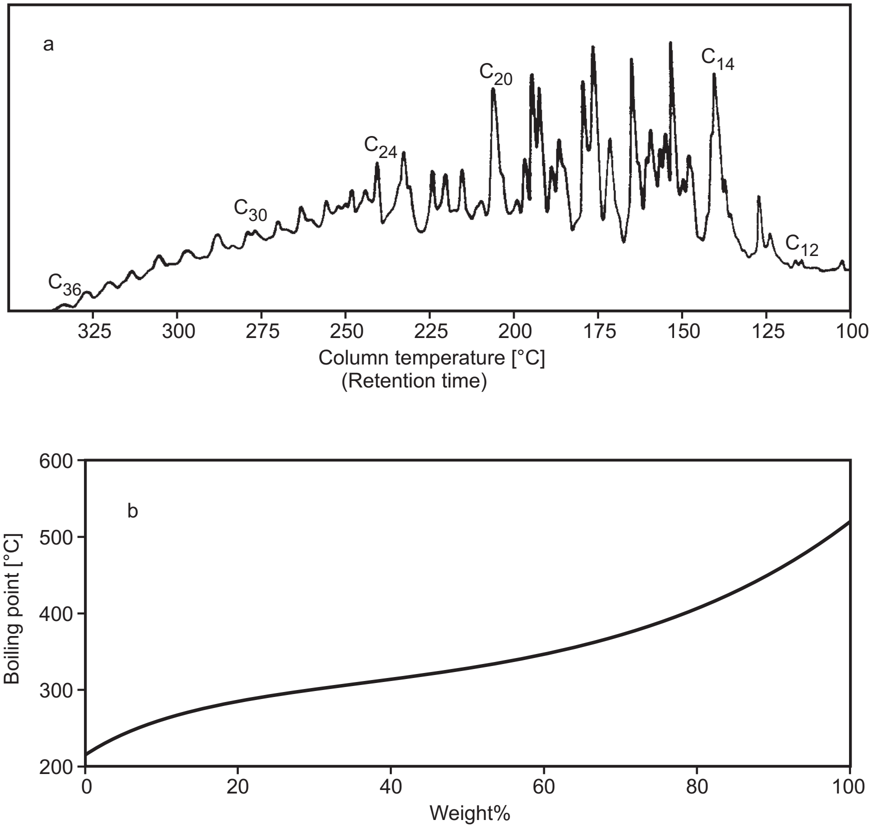


**Fig. 4.20** Use of high-temperature gas chromatography to determine boiling point distribution [4.23]. a. Gas chromatogram; b. Boiling point distribution.

etc.). Vapor pressure is dependent on temperature and follows the Antoine equation for base oil components. Mathematical models have been created for complex hydrocarbon mixtures. As a simple laboratory method, Noack evaporation (1 h at 250 °C) has become the established method of characterizing evaporation behavior in lubricant specifications with the evaporation losses being given in % w/w. Gas chromatographic methods are also used but these can produce somewhat deviating results.

This led to a simulated Noack evaporation method using gas chromatography. In the USA, a gas chromatographic process was developed to determine engine oil volatility (ASTM D5480). In this case, evaporation takes place at 371 °C (700 °F) and the evaporating components are measured to ca. $n$-$C_{22}$. Although this process is easily reproducible, the values obtained are not comparable with those from the Noack method.

Figure 4.22 shows typical evaporation losses for well-cut paraffinic solvates; values for VHVI oils (HC-2 oils, API Group III oils) and typical ester base oils (Chapter 5) are shown for comparison.

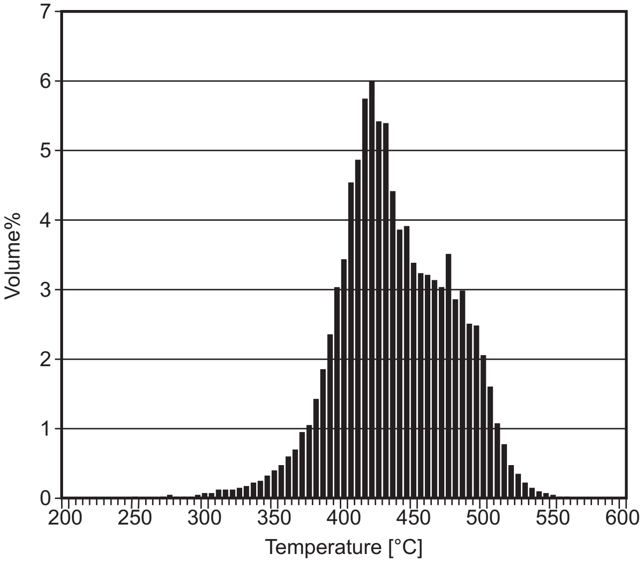


**Fig. 4.21** Distillation curve of a blend of two solvent-refined base oils obtained by gas chromatography and modern software.

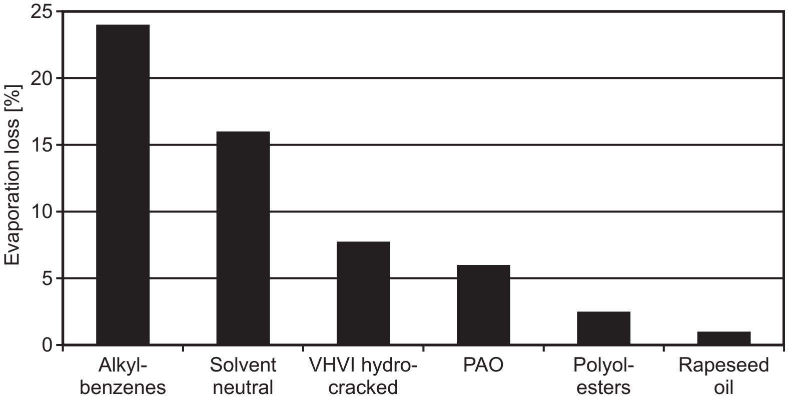


**Fig. 4.22** Typical evaporation losses (Noack evaporation) of various lube base oils (250 °C, 1 h).

## 4.6
## Base Oil Categories and Evaluation of Various Petroleum Base Oils

The American Petroleum Institute (API) and the Association Technique de l'Industrie Européenne des Lubrifiants (ATIEL) have classified base oils according to their chemical composition. Initially there were four groups; after the introduction of VHVI oils in Europe this was increased to five. The most important reason for these groups was the necessity to regulate base oil interchangeability for engine oils. (Chapter 9). The classification of petroleum base oils (Groups I to III) considers three parameters: saturates content, sulfur content, and viscosity index. Table 4.3 shows this classification. Accordingly, Group I oils are solvent extracted HVI oils (SN oils), Group II oils are hydrogenated or hydrocracked oils (as the sulfur content of < 300 ppm shows) and Group III products are VHVI oils manufactured by severe hydrocracking and or wax isomerization (VI > 120, sulfur < 300 ppm).

Table 4.4 shows typical data of various hydrocracked base oils (HC-oils), in comparison with solvent refined oils and polyalphaolefins. Although there are several technical intermediate possibilities, severely hydrocracked Group III oils have become well-established on the market.

**Tab. 4.4** Typical data for 4 $mm^2 s^{-1}$ base oils – HC oils – in comparison with conventional solvent refined oils and polyalphaolefins.

| | ***Solvent refined*** | ***HC-1*** | ***HC-2*** | ***HC-3*** | ***Polyalpha-olefins*** |
|---|---|---|---|---|---|
| ***Viscosity ($mm^2 s^{-1}$) at 100 °C*** | 4 | 4 | 4 | 4 | 4 |
| ***Viscosity index*** | 100 | 105 | 125 | 130 | 125 |
| ***Volatility, Noack evaporation loss (% w/w).*** | 23 | 18 | 14 | 13 | 12 |
| ***Pour point (°C)*** | –15 | –15 | –18 | –20 | –65 |
| ***API group*** | I | II | III | | IV |

Figure 4.23 illustrates the reduction of aromatics (as indicated by infrared spectroscopy) by different refining processes including production of HC-2 oils [4.24].

HC-2 oils are classified as very high viscosity index (VHVI) oils. HC-3 oils are also known as extra or ultra high viscosity index (UHVI) oils. They are generally made by hydrocracking and isomerization slack waxes followed by solvent dewaxing.

Table 4.5 shows typical hydrocarbon compositions of HC-oils in comparison with conventional solvent refined oils and polyalphaolefins [4.19].

**Tab. 4.5** Typical hydrocarbon composition of HC-oils in comparison with conventional (4 $mm^2 s^{-1}$) solvent refined oils and polyalphaolefins.

| | ***Solvent refined SN 100*** | ***HC-1*** | ***HC-2*** | ***HC-3*** | ***Polyalphaolefins*** |
|---|---|---|---|---|---|
| ***n- and iso-paraffins*** | 25 | 30 | 55 | 75 | 96 |
| ***Mono-cycloparaffins*** | 20 | 35 | 24 | 15 | 4 |
| ***Poly-cycloparaffins*** | 30 | 34 | 20 | 10 | – |
| ***Aromatics*** | 24 | 0.5 | 0.3 | 0.1 | – |
| ***Thiophenes*** | 0.5 | – | – | – | – |

The first characteristics of hydrocracked oils which attracts attention is their low sulfur content, or even the absence of sulfur, and the high VI and low evaporation characteristics of HC-2 and HC-3 oils. The expected, poorer additive response of antioxidants (Chapter 6), pour point depressors, or other additive groups were not

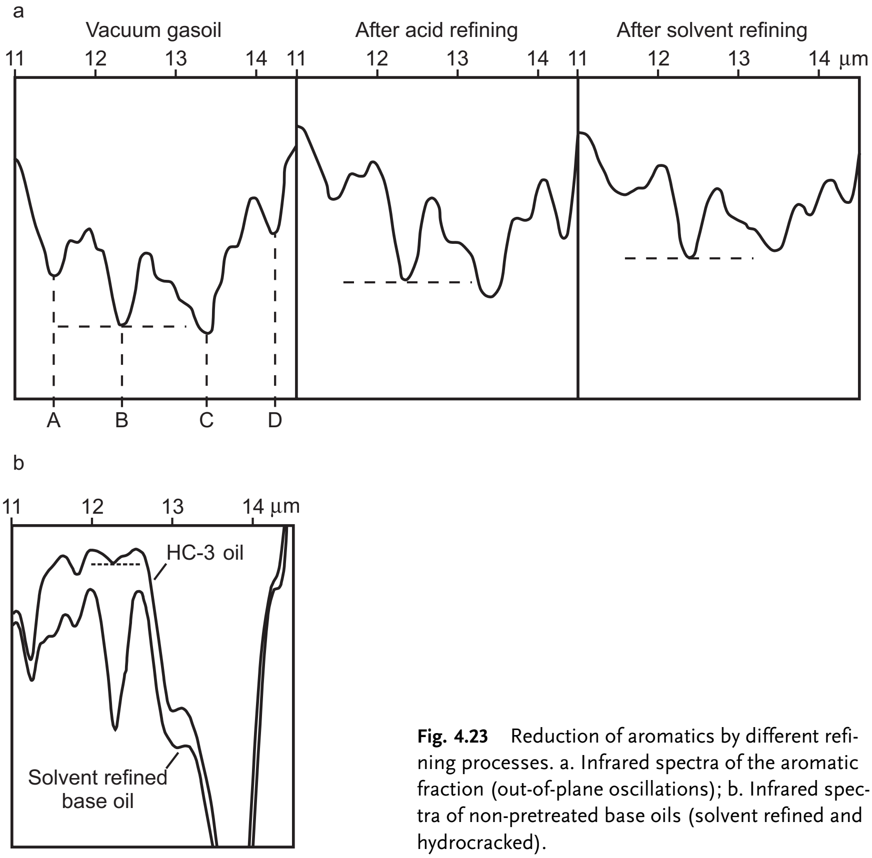


**Fig. 4.23** Reduction of aromatics by different refining processes. a. Infrared spectra of the aromatic fraction (out-of-plane oscillations); b. Infrared spectra of non-pretreated base oils (solvent refined and hydrocracked).

confirmed when hydrocracked oils were introduced. When inhibitor compositions were optimized, all hydrocracked oils were more stable to oxidation than solvent refined oils (if the right additives were used). This is primarily a result of their high chemical purity. The missing 'natural' sulfur inhibitors are more than compensated for by the addition of suitable antioxidants.

HC-2 and HC-3 oils are being increasingly compared with synthetic polyalphaolefins (PAO); the close similarity of the performances of some hydrocracked oils and PAO, and their significantly lower cost, make them increasingly attractive for lubricant formulations. The term 'synthetic', which has been used in Europe and recently also in the USA, has led to heated legal arguments. As considerable synthesis takes place during severe hydrocracking and catalytic dewaxing, the term seems justified although this then eliminates the terminological differentiation into HC-2 and HC-3 oils. In Germany, the term HC-synthesis has gained acceptance for HC-2 oils. The synthesis terminology applies even more to oils which originate from liquefied natural gas and which can be classified as HC-3.

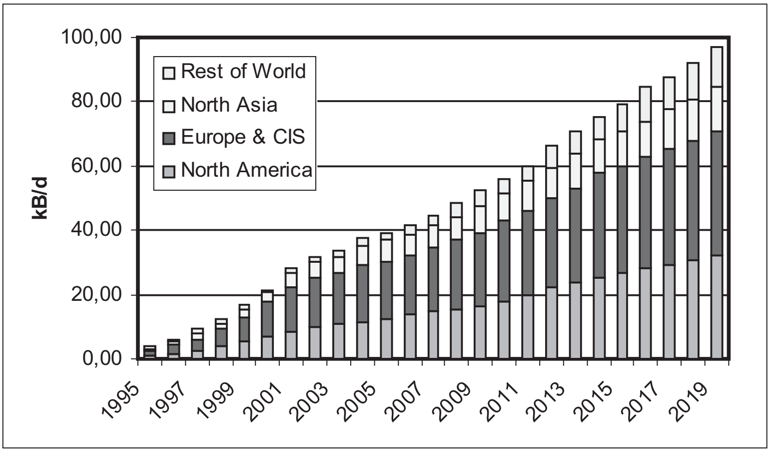


**Fig. 4.24** Development of VHVI and UHVI oils (HC-2 and HC-3) and PAO in the different regions of the world.

Figure 4.24 Illustrates the development of VHVI and UHVI oils (HC-2 and HC-3 oils) and PAO in the different regions of the world. Whereas Shell and BP in France and Union Fuchs in Germany were the only manufacturers of VHVI oil in the eighties, by the mid-nineties similar activities had spread from Western Europe around the world [4.21].

Because of the high quality and performance of UCBOs, these products have a higher market value than Group I or Group II base oils. The market price for future GTL base fluids will be determined by product quality, economies of scale, and the marketing strategies of the GTL players.

# 5
# Synthetic Base Oils

*Wilfried Dresel*

According to Zisman [5.1] the rise of synthetic lubricating oils on an industrial scale began in 1931, when Sullivan [5.2] and his coworkers published the results of their attempts to make tailor-made saturated lubricating oils with low pour points by catalytic polymerization of olefins. Gunderson and Hart [5.3] in 1962 edited an outstanding book on synthetic lubricants. It covered nine classes of lubricating oils and also contained Zisman's contribution to their history. In 1993 Shubkin [5.4] edited a remarkable new book on the subject that omitted some of the older classes and added some new ones instead (Table 5.1).

**Tab. 5.1** Classes of synthetic lubricant.

| Gunderson and Hart (1962) [5.3] | Shubkin et al (1993) [5.4] |
|---|---|
| | Alkylated aromatics |
| Chlorofluorocarbon polymers | Chlorotrifluoroethylene |
| | Cycloaliphatics |
| | Dialkylcarbonates |
| Dibasic acid esters | Esters |
| Fluoro esters | |
| Neopentyl polyol esters | |
| | Perfluoroalkylpolyethers |
| Phosphate esters | Phosphate esters |
| | Phosphazenes |
| | Polyalphaolefins |
| | Polybutenes |
| Polyglycols | Polyalkylene glycols |
| Polyphenyl ethers | |
| | Silahydrocarbons |
| Silicate esters | |
| Siloxanes | Siloxanes |

Although many of the synthetic base oils available today had been developed decades ago, their use on a large technical scale has increased only slowly because of their considerably higher cost [5.5, 5.6, 5.111] (Table 5.2).

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**Tab. 5.2** Relative costs of synthetic base oils

| | |
|---|---|
| Mineral oils | 1 |
| Alkylated aromatic compounds | 2–3 |
| Polybutenes | 3–5 |
| Dibasic acid esters | 4–15 |
| Polyalphaolefins | 4–15 |
| Poly(alkylene glycol)s | 6–15 |
| Neopentyl polyol esters | 10–20 |
| Silicones | 25–200 |
| Perfluoralkyl polyethers | 350–800 |

Although the consumption of synthesized lubricating oils is, on the whole, responding only reluctantly to benefits which have long been recognized [5.7], and to the needs of machinery that has to work under increasingly extreme conditions, in terrestrial applications mainly at higher temperatures and pressures [5.8–5.10], in space applications mainly at lower temperatures and very low pressures [5.11, 5.12], the consumption of polyalphaolefins, the most common synthetic lubricating oils, has increased enormously in the last two decades [5.13–5.15].

In contrast with mineral oil-based oils, that contain many different hydrocarbons, and nitrogen-, oxygen-, and sulfur-containing chemical derivatives of these hydrocarbons, that must be purified and refined and distilled (Chapter 4), synthetic base oils usually are prepared by reaction of a few defined chemical compounds – although in many cases based on petroleum also – and tailored to their application by the right choice of reaction conditions. This comparably simple chemistry not only has advantages, of course, but, mainly with regard to additive response and elastomer compatibility, some disadvantages, also [5.16, 5.17].

Synthetic base oils have been classified according to both the production process and their chemical composition [5.6, 5.18]. From a chemical point of view the latter method is preferable.

## 5.1
## Synthetic Hydrocarbons

Synthetic hydrocarbons were developed simultaneously in Germany and the United States of America. In Germany low-temperature performance and the need to overcome the general shortage of petroleum base stocks was the driving force behind the work of Zorn [5.19]. It is known today, that all synthetic hydrocarbons – and the other economically important synthetic lubricating oils – can be synthesized starting with ethylene (Fig. 5.1). Ethylene itself is one of the most important petrochemicals [5.20] and today mainly produced in steam crackers.

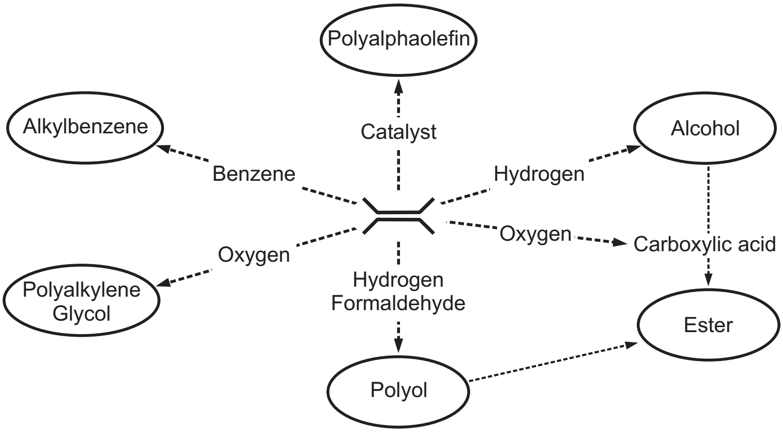


**Fig. 5.1** Ethylene (a source of many synthetic base oils).

### 5.1.1
### Polyalphaolefins

The term polyalphaolefin (PAO) is derived from the source of this class of base oil, usually $\alpha$-decene or a mixture of $\alpha$-olefins containing, in general, a minimum of six and a maximum of twelve carbon atoms, The oligomers are saturated, i.e. hydrogenated, and therefore belong to the aliphatic or branched paraffinic hydrocarbons. Linear $\alpha$-olefins were first used for the synthesis of lubricating oil by Montgomery, Gilbert, and Kline [5.21]. Polyalphaolefins have been described in detail by Shubkin [5.15].

Free radical – or thermal – oligomerization of $\alpha$-olefins is possible but is no longer important because of high activation energy, low yield, and quality, even when peroxides are used as catalysts. Oligomerization with Ziegler–Natta catalysts of the aluminium triethyltitanium tetrachloride type tends to yield a wide range of oligomers that can be controlled more easily when a catalyst of the alkylaluminum halide–alkoxide–zirconium halide type is used. Boron trifluoride-based Friedel–Crafts oligomerization with alcohols as co-catalysts has, nevertheless, proved superior, although even today the mechanism is not fully understood. Possible mechanisms have been described in detail by Mortier and Orszulik et al, [5.22]. The boron trifluoride method has become predominant for the production of grades with lower viscosities, 2–10 $mm^2 s^{-1}$. For higher viscosities, 40 and 100 $mm^2 s^{-1}$, other catalysts must be used, and for the production of grades with viscosities of 10–25 $mm^2 s^{-1}$, intended to replace disadvantageous blends and not yet available in comparable amounts, C-12 and C-14 olefins can be used; these yield mainly dimers, [5.23]. The next step of the manufacturing process comprises the catalytic hydrogenation of the unsaturated olefins. This is achieved with the aid of classical catalysts, e.g. nickel on kieselguhr or palladium on alumina. In a third and final step the saturated oligomers are distilled (Fig. 5.2). The oligomerization of $\alpha$-decene or $\alpha$-olefin mixtures always results in complex mixtures of isomers with more branching than expected.

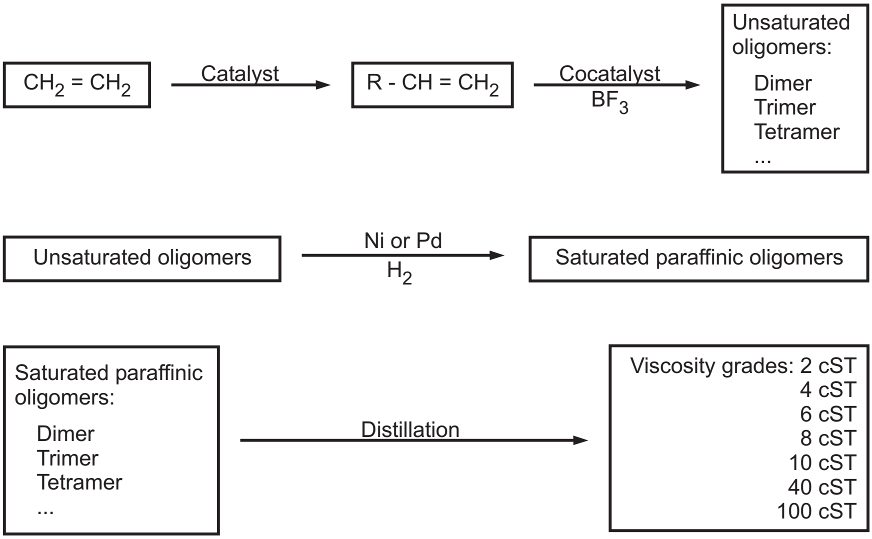


**Fig. 5.2** The three steps of polyalphaolefin production.

Because the rearrangements are intramolecular, the molecular weights of the products can be kept within a narrow range. A typical saturated α-decene trimer for example looks like a three-pointed star [5.24] (Fig. 5.3).

Polyalphaolefins satisfy some of the requirements of ideal hydrocarbon lubricants that can be predicted from chemical structure considerations – ideal hydrocarbon

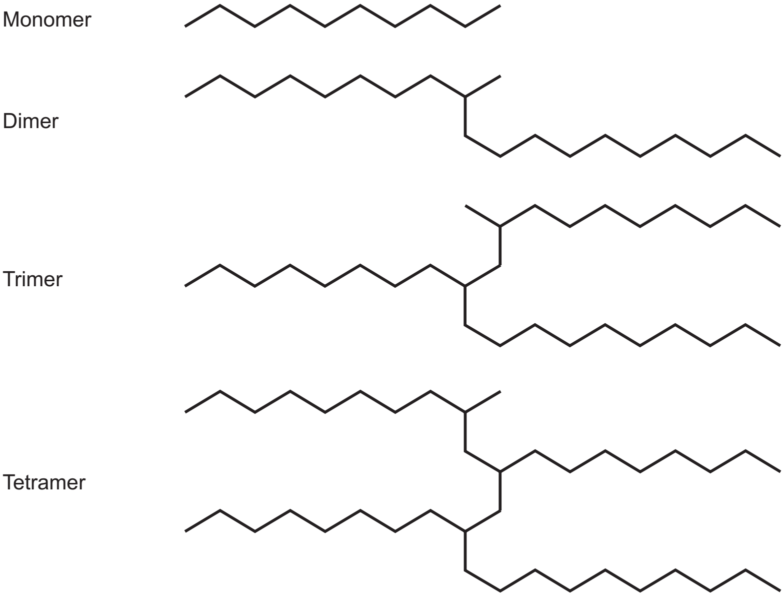


**Fig. 5.3** The shapes of typical polyalphaolefin oligomers.

lubricants should have straight chains, be completely saturated and crystallize at low temperatures. The viscosities of straight-chain alkanes do indeed increase with chain length; the same is true for their pour points and viscosity indexes (VI). At constant molecular mass branching leads to increased viscosity and a decrease in pour point and VI. The length and position of the side chains influence all three properties. When branching occurs in the middle of the main chain, pour points are lower. Long side chains improve viscosity–temperature (*V*–*T*) characteristics.

Polyalphaolefins therefore have several advantages – narrow boiling ranges, very low pour points, viscosity index (VI) > 135 for all grades with kinematic viscosity > 4 $mm^2\ s^{-1}$ at 100 °C. Their volatility is lower than that of all possible and equiviscous mineral oil grades and they contain only small quantities of unsaturated and polycyclic aromatic compounds and only traces of nitrogen, sulfur, or other impurities. Although in some oxidation tests without additives some mineral oils seem superior to polyalphaolefins, this is because of the presence of natural antioxidants in the mineral oils that have survived the refining process – the oxidation of mineral oils has been described by Rasberger [5.25] in detail. The response of the synthetic products to antioxidants and their EP/AW synergists is better [5.26–5.28] than that of mineral oils. Their low polarity, on the other hand, leads to poorer solvency for very polar additives and can cause problems with seals. Therefore they tend to be used in combination with smaller amounts of (dicarboxylic acid) esters or solvent-refined mineral oils.

Polyalphaolefins have been used traditionally in aerospace and lifetime applications, but today they are used in a wider variety of applications and have gained additional importance because of the increasing need for them in automotive lubricants [5.29].

### 5.1.2
### Polyinternalolefins

Polyinternalolefins (PIO) are rather similar to polyalphaolefins. Both kinds of hydrocarbon are prepared by the oligomerization of linear olefins. The difference is that polyinternalolefins are made from cracked paraffinic base stocks. Plants for the manufacture of polyinternalolefins and polyalphaolefins therefore look similar, also (Fig. 5.4). Internal olefins are more difficult to oligomerize and the resulting products have VIs 10 to 20 units lower than the VIs of equiviscous polyalphaolefins [5.30].

Mixtures of polyalphaolefins with polyethylenes with kinematic viscosities from 100 to 2000 $mm^2\ s^{-1}$ at 100 °C are available commercially. It can be expected that polyinternalolefins having these viscosities genuinely will be available in the future.

While the cationic polymerization of ethylene with aluminium chloride yields oils with VIs up to 120 and molecular masses from 400 to 2000, the polymerization of propylene gives oils with lower VIs and poor thermal stability. Both kinds of lubricating oil have lost importance, but copolymers of both base materials are said to have the potential for gaining importance again.

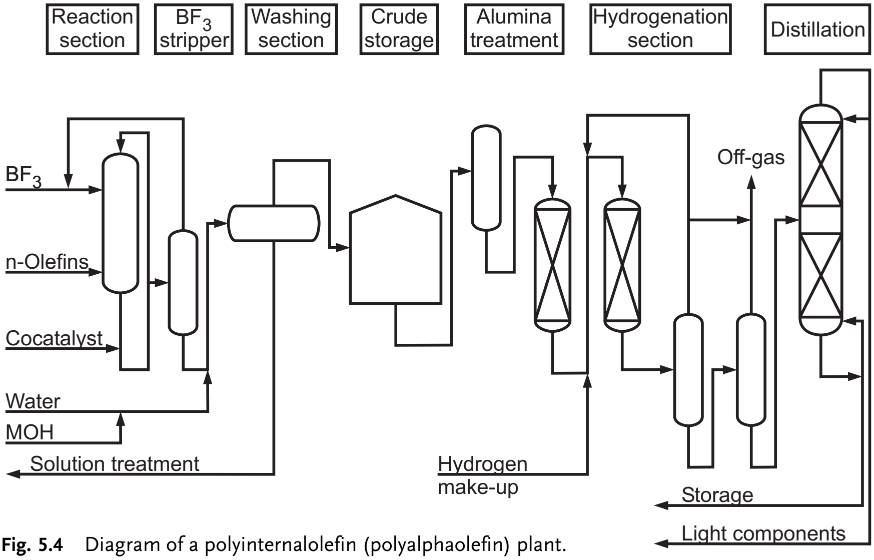
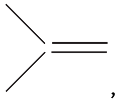


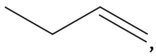
**Fig. 5.4** Diagram of a polyinternalolefin (polyalphaolefin) plant.

### 5.1.3
### Polybutenes

Polyisobutylene rubbers were manufactured as early as the 1930s. It was one or two decades before their liquid analogs became available. Polybutenes (PBs) as synthetic base oils have been described in detail by Fotheringham [5.31]. They consist mainly of isobutene and are, therefore, often also known as polyisobutenes (PIBs).

They are produced by the polymerization of a hydrocarbon stream that besides isobutene **5.1** contains the other two butenes (**5.2**, **5.3**) and the butanes. The main sources of the C-4 feedstock are naphtha steam crackers and refinery catalytic crackers. The Lewis acid-catalyzed process yields a copolymer with a backbone built mainly from isobutene units. The higher the molecular weight, the lower the content of other butenes, that is, the lower the molecular weight the more complex the structure. At the end of the carbon chain there remains one double bond. Therefore polybutenes are less resistant to oxidation than polyalphaolefins, polyinternalolefins, and alkylated aromatics – above 200 °C they begin to depolymerize and form gaseous products. Normally the end-group is *cis-* and *trans-*trisubstituted. It is possible to replace it by a disubstituted vinylidene group, that is more reactive. With maleic anhydride it yields polybutenylsuccinic anhydride derivatives, that are used as corrosion inhibitors and detergents.


isobutene
**5.1**

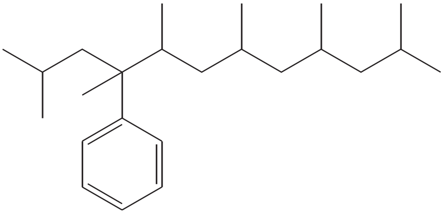

butene-1
**5.2**


butene-2
**5.3**

Polybutenes with molecular masses from about 300 to 6000 are important as VI improvers. They are used as components in two-stroke oils, gear and hydraulic oils, metal working lubricants, greases, compressor lubricants, and wire-rope protectives [5.32]. One of the options for future polybutene applications is the increased use as synergistic components in binary or ternary base oil systems [5.33].

### 5.1.4
### Alkylated Aromatics

Alkylnaphthalenes as lubricants have been available in Germany since ca. 1930 [5.34]. Alkylated aromatics have been described by Dressler in detail [5.35]. Low-cost dialkylbenzenes are produced as by-products in the manufacture of linear and branched monoalkyl benzenes that themselves are feedstocks for the manufacture of detergents. The Friedel–Crafts alkylation of benzene with olefins tends to yield polyalkylated products, because of the reactivity of the primary product. With excess benzene, a suitable olefin, e.g. propene, and the right choice of a catalyst the reaction can be controlled and specialized alkyl benzenes can be synthesized. The structure of a typical alkylbenzene comprises six propene units **5.4**. The properties of synthetic alkyl benzenes vary widely and are difficult to generalize.



**5.4**

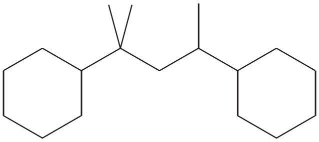
Some of the properties of alkylated benzene can be explained by chemical structure considerations – ring formation leads to a larger increase in viscosity and decrease in VI values than alkyl substitution; side chains on a naphthenic ring have the opposite effect. A shift of cyclohexyl substituents from the end toward the middle of an alkyl chain has only a small effect on viscosity, but a strong negative effect on *V–T*; it also lowers the pour point; cyclopentane rings have a similar effect and alkylated aromatics have effects similar to those of equimolecular alkylnaphthenes but the products have lower viscosities and less favorable *V–T* dependence.

Lower-cost dialkylbenzenes are used in a wide variety of industrial areas, e.g. in transformer oils, where high resistance to gas evolution is important. Tailored grades, especially the linear products, although having poorer properties than polyalphaolefins, because of their excellent solvency, their suitability for low temperatures, and their compatibility with elastomers still are an option [5.36]. The most widespread use of these products is, however as refrigerator oils.

### 5.1.5
### Other Hydrocarbons

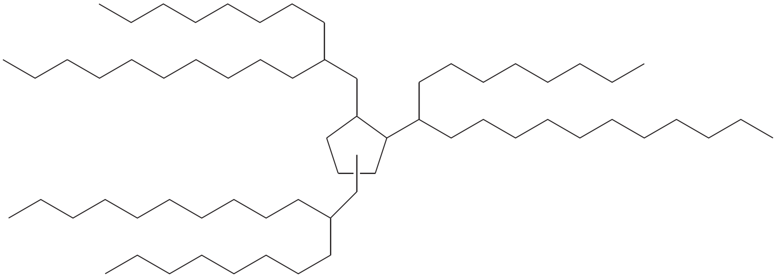
Cyclic saturated or alicyclic hydrocarbons occur naturally as components of mineral oils and here are known under the name naphthenes. In contrast with paraffins, that have their analogs in polyalphaolefins, and aromatics, that have them in alkylbenzenes, naphthenes do not yet have comparable synthetic analogs available on a technical scale. Former attempts with naphthalenes have, however, been renewed and polyalkylated oligomers are expected to be suitable as high-temperature lubricants [5.37].

Some synthetic cycloaliphatic hydrocarbons have coefficients of friction up to 1.2. One reason is that when bulky cyclopentyl or cyclohexyl groups are introduced into branched short-chain molecules, intramolecular rotation is hindered. The high molecular packing state and the stiffness of some polycyclic hydrocarbon molecules also makes them suitable as components of traction fluids for friction gears [5.38], where these properties of the lubricant are important design elements [5.39, 5.40]. The basic material for some of the best described traction fluids is 2,4-dicyclohexyl-2-methylpentane **5.5**, the hydrogenated dimer of α-methylstyrene.



**5.5**

Other cyclic aliphatics that have potential use as lubricating oils have been described in detail by Venier and Casserly [5.41]. Of these compounds tris(2-octyldodecyl)cyclopentane **5.6** has found limited use in high-vacuum applications, for example in greases, because of its extremely low volatility [5.42].



**5.6**

Tri(*n*-alkyl)methylmethanes and tetra(*n*-alkyl)methanes have been synthesized and have been compared with polyalphaolefins of similar viscosity. The products seem to be useful in applications that demand extreme thermo-oxidative stability [5.43]. Alkylated biphenyls and diphenylmethanes have also been proposed as high-temperature lubricants, but are not yet available. Adamantane-based synthetic lubricants have been synthesized and seem to have some potential as electrically insulating fluids [5.44].

## 5.2
## Halogenated Hydrocarbons

Chlorinated hydrocarbons are very stable and not combustible. They were once widely used as insulating oils, heat-transfer fluids, and hydraulic fluids, but this use has been discontinued because of the environmental problems they cause.

Commercial liquid chlorofluorocarbons, all oligomers of chlorotrifluoroethylene contain between two and twelve units. The first products were developed in the 1940s and have been described in detail by Ashton and Strack [5.45] and by Ruesch [5.46]. They are suitable for use as lubricants mainly because of their extraordinary chemical stability, including against oxygen.

Their low corrosivity, the good low-temperature characteristics of the low-viscosity grades, and their lubricating properties are also advantageous. Disadvantages are their high volatility and their not ideal V-T dependence. The properties of the chlorofluorocarbons are a consequence of the larger volume of the chlorine and fluorine atoms, which hinders the flexibility of the molecule, of the reduced intermolecular cohesive forces, and of the different bond lengths. Their density naturally is relatively high, (between 1.7 and 2 g $mL^{-1}$ at 40 °C). They are used as lubricants in oxygen compressors, in pumps for mineral acids, halogens, and oxygen, and in mills or mixers for strong oxidizing agents. They are also suitable lubricants for turbine pumps in rocket engines and as base oils for non-flammable hydraulic fluids [5.47]. Although the halogenated hydrocarbons are comparable with perfluorinated polyethers (see Section 5.5) with regard to their performance, but not as expensive, their future is unclear.

## 5.3
## Synthetic Esters

### 5.3.1
### Esters of carboxylic acids

Synthetic ester lubricants for the jet age were developed in Germany during World War II [5.48].

Hydrocarbon oils could not satisfy the demands on aircraft engine oils and alkyl esters of aliphatic carboxylic acids had favorable properties. Carboxylic groups, because of their strong dipole moments, reduce the volatility and increase the flash point of lubricating oils, and at the same time positively affect thermal stability, (the bonds of the COO group are thermally more stable than the C–C bond), solvency, lubricity, and biodegradability; on the other hand, however, they negatively affect the hydrolytic stability of a lubricant and the reactivity with metals or alloys that contain copper or lead [5.49]. The general properties of synthetic esters of carboxylic acids have been described in detail in Ullmann's Encyclopedia of Industrial Chemistry [5.50, 5.51] and by Randles [5.52].

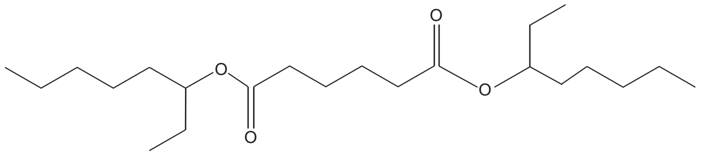
In principle all esters of carboxylic acids can be prepared in the same manner:

1 the carboxylic acid is reacted with excess alcohol in the presence of a catalyst, (mineral acids, ideally adsorbed on a solid, ion exchangers, Lewis acids, e.g. boron trifluoride, ideally as the etherate, and amphoteric hydroxides such as aluminium hydroxide can be used as catalysts);
2 to shift the equilibrium to the product side, the reaction water is removed during the process; and
3 the unreacted acid is neutralized with sodium carbonate or calcium hydroxide and then removed by filtration and the ester is distilled.

#### 5.3.1.1 **Dicarboxylic Acid Esters**

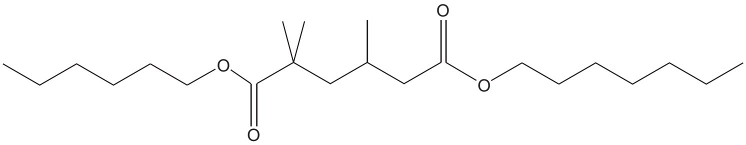
Dicarboxylic acid esters have been described in detail by Dukek and Popkin [5.53]. Two types of ester molecule or their combinations have been found to be most suited as lubricant components:

1 esters of branched primary alcohols with straight dicarboxylic acids **5.8**; and
2 esters of straight primary alcohols with branched dicarboxylic acids **5.9**.



**5.8**

di(2-ethylhexyl)adipate

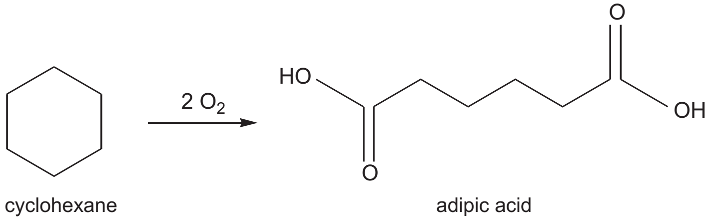


**5.9**

diundecyl(2,2,4-trimethyl)adipate

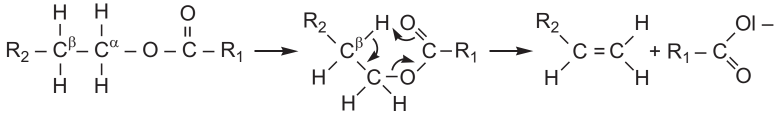
The alcohols that are needed for the production of dicarboxylic acid ester oils can be obtained synthetically by the hydroformylation of olefins with carbon monoxide and hydrogen (Eq. 5.1). The dicarboxylic acids can be obtained either by oxidation of vegetable oils, e.g. castor oil, as for azelaic and sebacic acid [5.54], or by cleavage of corresponding alicyclic hydrocarbons with oxygen, as for adipic acid (Eq. 5.2) [5.55].

$$R-CH=CH_2 \xrightarrow{CO+H_2} R-CH_2-CH_2-CHO \xrightarrow{H_2} R-CH_2-CH_2-CH_2OH \quad (5.1)$$



(5.2)

Esters of straight-chain dicarboxylic acids have better *V–T* characteristics than mineral oils, and higher VI values. The values decrease with increasing branching, but branching improves the low-temperature properties. Esters with little branching have the best properties, especially esters with methyl groups adjacent to the carboxyl group. When the esters contain tertiary hydrogen atoms, however, their stability toward oxidation is not improved (Eq. 5.3). The sterically hindered esters have favorable thermal and hydrolytic stability. The hydrolytic stability has been further improved with the aid of carbodiimides [5.55]. At low temperatures the viscosity of esters follows a time function and additives can have a negative effect on the viscosity and the pour point. With methacrylates as VI improvers values between 170 and 180 are reached. With complex esters as VI improvers degradation as a result of shear stress is less pronounced. Additives are mainly added to improve the EP performance, although the load-carrying capacity of esters in general is twice that of mineral oils.

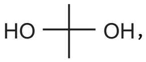


(5.3)

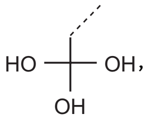
Dicarboxylic acid ester oils can improve the *V–T* characteristics of automotive engine oils without having a negative effect on the low-temperature viscosity or the Newtonian flow behavior. Such oils are suitable for diesel engines also. Esters have also gained importance as lubricity additives in greases and in polyalphaolefins as additives for the improvement of the compatibility with elastomers.

### 5.3.1.2 **Polyol Esters**

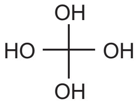
Since the 1940s neopentyl glycol ((2,2-dimethyl)-1,3-propandiol) **5.10**, trimethylolethane and -propane (1,1,1-tris(hydroxymethyl)ethane and –propane) **5.11**, and pentaerythritol (2,2-bis(hydroxymethyl)-1,3-propanediole) **5.12** have been known as the alcoholic components of esters. They have been described in detail by Smith [5.56]. Beside excellent thermal and oxidation stability, the esters have good *V–T* and lubricating properties and excellent viscosity characteristics at low temperatures. The reason is that the thermal stability of a primary OH group is greater than that of a secondary group, and the hydroxy derivatives of neopentane contain only primary hydroxy groups, and short side-chains in a hydrocarbon not only reduce its pour point, but tertiary C or H atoms at the same time facilitate the attack of oxygen; the polyols, however, have no tertiary H atoms and no H atoms in the β-position that could facilitate thermal cleavage.



**5.10**



**5.11**

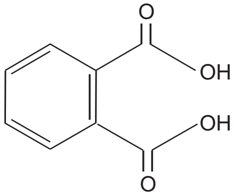


**5.12**

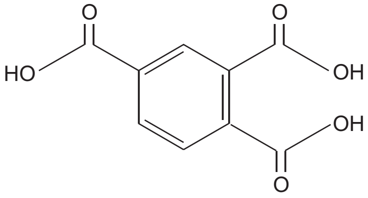
The mechanism of the polymerization of polyol esters during aging has been studied [5.57] and benzotriazole derivatives have been proposed as multifunctional additives [5.58]. Since the 1960s the polyol esters have gained importance as lubricants for high-temperature applications, for example in aircraft engines that are meant for speeds beyond Mach 2, and they have become even more important since the rise of the biodegradable esters.

#### 5.3.1.3 Other Carboxylic Esters

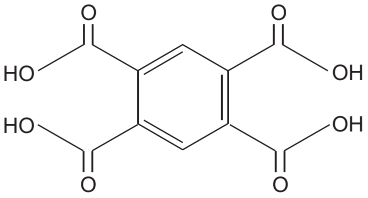
The structural counterparts of the polyol esters are the esters of some aromatic polycarboxylic acids, mainly phthalic acid **5.13**, trimellitic acid **5.14**, and pyromellitic acid **5.15**, with monofunctional alcohols. The latter, also, are suited as high temperature lubricants. The time dependence of the low-temperature viscosity of some grades is rather pronounced. Therefore their use in low temperature applications is problematic.



**5.13**



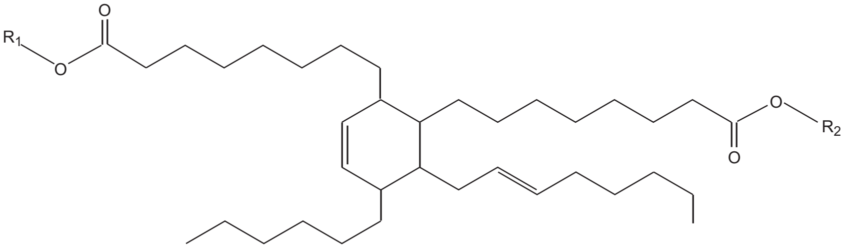
**5.14**



**5.15**

Monoesters, the reaction products of fatty acids and monofunctional alcohols, are used in metal working. The sulfurized esters are excellent EP additives for all kinds of lubricant [5.59].

Dimeric acid esters **5.16**, the acids are made from oleic or tallow fat acid, are used as engine oil and thickener components.



**5.16**

#### 5.3.1.4 **Complex Esters**

Esters that contain straight or branched diols or polyalkylene glycols and straight or branched dicarboxylic acids as well as (mono)carboxylic acids and monofunctional alcohols have attracted interest. Usually first the diol is esterified with the dicarboxylic acid, then, depending on the desired product, the intermediate product is reacted with either a carboxylic acid or a monoalcohol. Two types of complex ester have become important:

ROH···HOOCrCOOH···HORrROH···HOOCrCOOH···HOR **5.17**

RCOOH···HORrROH···HOOCrCOOH···HORrROH···HOOCR **5.18**

The same scheme is valid when neopentane derived polyols [5.60] or glycerol [5.61] are involved.

That complex esters have higher molecular weights and higher viscosities than the common esters was one of the main reasons for their introduction. Esters of type **5.18** are superior to type **5.17** with regard to flash point, pour point, and low-temperature viscosity. In type **5.17** polyalkylene glycols lead to lower pour points than aliphatic glycols. Medium molecular-weight oligomeric esters starting from (mainly vegetable) fatty acid triglycerides [5.112] are of interest for environmentally friendly and high-performance applications, as are esters starting from adipic acid or sebacic acid and neopentyl glycol (type 5.17), because their biodegradability and because their pour points are approximately the same as those of their monomers, although their viscosities are higher [5.113].

High molecular weight complex esters of the type:

R··· (HOrOH···HOOCrCOOH)$_n$···R **5.19**

have also been named polymer esters and find use mainly in applications where mineral oil and solvent resistance is required.

When alphaolefins and alkylmethacrylates are co-oligomerized another kind of polymer ester results [5.62] and a third kind is based on polymers, that have been functionalized [5.63]. With such products attempts have been made to combine the advantages of synthetic hydrocarbons and synthetic esters at a high viscosity level.

#### 5.3.1.5 **Fluorinated Carboxylic Acid Esters**

Esters of fluorinated acids are readily hydrolyzed and the free acids have a strong oxidizing effect, therefore only esters with fluorinated alcohols can be used as lubricants. The thermal stability of fluorinated esters is better than that of the non-fluorinated analogs. The oxidation stability is also higher. Some types of rubber shrink in their presence and some antioxidants have a negative effect. Fluorinated esters were in the first place intended for high-temperature applications. They have been described in detail by Murphy [5.64]. It can be assumed that their use will be limited because of competition from the fluorinated polyethers. Esters containing sulfur as a part of the acidic or alcoholic component are known but have not gained any importance.

In the future, mixtures of all kinds of synthetic hydrocarbon and synthetic ester promise much improvement in all fields of application [5.65–5.67].

### 5.3.2
### Phosphate Esters

Tertiary esters of phosphoric acid with alcohols or phenols were prepared as long as 150 years ago but were not introduced as antiwear additives before 1930. They have also gained importance as plasticizers, fire-resistant hydraulic fluids, compressor oils, and synthetic lubricants. Phosphate esters have been described in detail by Hatton [5.68] and Marino [5.69]. They are usually divided into triaryl, trialkyl, and alkylaryl phosphates and are made by reaction of phosphoryl chloride with phenols or alcohols (Eq. 5.4). Their properties range from low-viscosity fluids to high-melting solids. With increasing molecular weight trialkyl phosphates change from water-soluble to insoluble liquids. The triaryl phosphates have higher viscosities and are insoluble in water. An aryl side-chain reduces the melting point. The properties of alkylaryl phosphates are between those of alkyl and aryl phosphates.

$$3\ \mathrm{ROH} + \mathrm{POCl_3} \rightarrow \mathrm{OP(OR)_3} + 3\ \mathrm{HCl} \tag{5.4}$$

The hydrolytic stability varies between good and poor. Length and branching of the alkyl chain increase hydrolytic stability, tolyl substitution is superior to a phenyl. Alkylaryl phosphates are more susceptible to hydrolysis than trialkyl or triaryl phosphates. Phosphoric acid esters are less stable than carboxylic acid esters, but more stable than silicic or boric acid esters. Their hydrolytic stability can be increased with, for example, epoxides [5.70]. With ion-exchange treatment and vacuum dehydration, hydrolysis of the phosphoric acid esters used as hydraulic fluids can be virtually eliminated [5.71].

The thermal stability of triaryl phosphates is better than that of the alkyl compounds. With aminic antioxidants and rust inhibitors, e.g. some amine salts of primary or secondary phosphates, triaryl phosphates can be used up to 175 °C, but alkyldiaryl phosphates only up to 120 °C. Branching of the alkyl radicals reduces the thermal stability, the effect becomes stronger with decreasing length. Trialkyl phosphates and alkyldiaryl compounds behave similarly. In general phosphoric acid esters are not corrosive. But thermal decomposition leads to the formation of phosphoric acid and this is a corrosion hazard. The high spontaneous ignition temperatures of up to 600 °C underline the good fire resistance of some products. Trialkyl and alkylaryl products have pour points down to –65 °C, when they contain VI improvers.

The lubricating properties of the phosphoric acid esters are excellent, particularly on steel. They can be blended with almost all lubricants and additives. Their dissolving power is, on the other hand, responsible for their incompatibility with rubbers, varnishes, and plastics. Nylon, epoxy and phenol–formaldehyde resins are stable.

## 5.4
## Polyalkylene Glycols

The first polyalkylene glycols suitable as lubricants were developed during World War II. The first patent was probably published in 1947 by Roberts and Fife [5.72]. Polyalkylene glycols have been described in detail by Gunderson and Millet [5.73], by Kussi [5.74] and by Matlock and Clinton [5.75].

Polyalkylene glycols are prepared by the reaction of epoxides, usually ethylene and propylene oxide, with compounds that contain active hydrogen, usually alcohols or water, in the presence of a basic catalyst, e.g. sodium or potassium hydroxide (Fig. 5.5). Variation of the ratio of the epoxides and the end groups leads to different products. Polymers with statistically distributed alkylene groups are made by use of a mixture of alkylene oxides. Separate addition leads to block copolymers. Because ethylene oxide is more reactive than propylene oxide, random copolymers tend to have the propylene oxide units at the chain ends.

Terpolymers with, for example, tetrahydrofuran have also been prepared. Pure tetrahydrofuran polymers can be obtained by polymerization of tetrahydrofuran in the

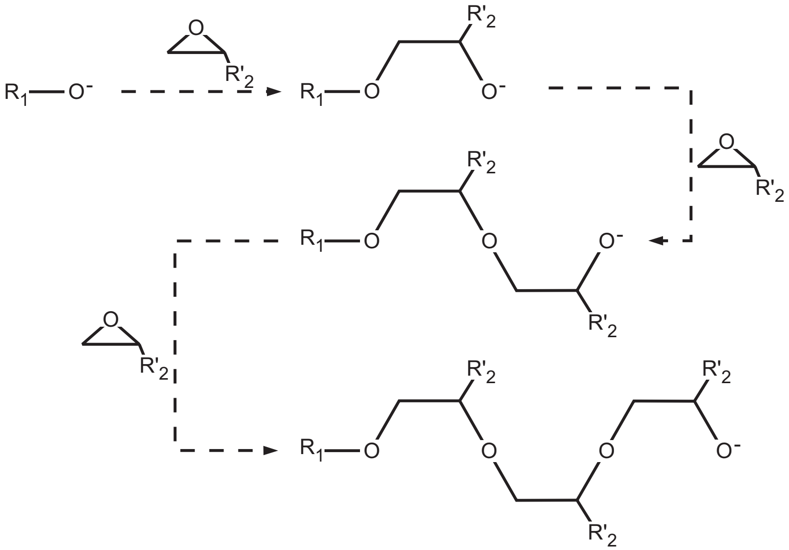


Fig. 5.5 Preparation of polyalkylene glycols.

presence of Friedel–Crafts catalysts. They are colorless oily or waxy substances of very low toxicity.

Polyalkylene glycols have at least one hydroxyl group at one end of the molecule, and can therefore be regarded as alcohols. The number of hydroxyl groups is increased by use of water or multifunctional starters. The reaction of the alcohols with acids leads to esters; reaction with, for example, strong acids and olefins leads to ethers.

Because the carbon–oxygen bond is stronger than the carbon–carbon bond, polyalkylene glycols have solvent properties somewhat different from those of hydrocarbons. Miscibility with water increases with the number of ethylene oxide units. The solubility is a result of hydrogen-bonding of water with the free electron pairs of the oxygen. In solution the water-soluble grades are practically non-flammable. The hygroscopic character of the polyalkylene glycols is dependent on their hydroxyl group content – it decreases with increasing molecular weight and the number of ether bonds. The solubility in water decreases in the same way. That it also decreases with increasing temperature can be explained by a loss of hydrogen bonding. Solubility in hydrocarbons increases with the molecular weight. Polyalkylene glycols in general are soluble in aromatic hydrocarbons.

The molecular weight and viscosity of polyalkylene glycols can be significantly influenced during production and be adjusted within narrow limits. The possibility of engineering products in this way distinguishes them from many other lubricants.

Low molecular weight polyalkylene glycols containing more than 50 % propylene oxide have pour points that go down to –65 °C. The lateral methyl groups are responsible for the disruption of crystallization. On the other hand pure high-molecular-weight polyethylene glycols are wax-like solids with pour points near +4 °C.

Kinematic viscosities range from 8 to 100 000 $mm^2\ s^{-1}$ at 40 °C. With the change from diols to monoethers, ester ethers and diethers, at the same molecular weight, viscosities are reduced, particularly at low temperatures. In comparison with mineral oils, which give straight lines, the *V–T* diagrams of polyglycols show their viscosities are too high at both low and high temperatures. The VI values of polyalkylene glycols usually lie around 200. High-molecular-weight polyethylene glycols have VI values of up to 400.

Prolonged heating of polyalkylene glycols to above 150 °C leads to depolymerization. The resulting aldehydes react further to give acids. It is an advantage that only soluble or volatile products are formed. Traces of alkali or alkaline earth metals promote the degradation. The decomposition can be prevented by addition of aminic antioxidants, to the extent that the oils can be used as heat-transfer fluids up to 250 °C. Antirust and EP additives for use in polyalkylene glycols must have pronounced water tolerance.

The polar nature of polyalkylene glycols gives the products strong affinity for metals, and so the lubricating film remains intact even at high surface pressure, a property useful in lubricants and metal cutting fluids. Because swelling of elastomers decreases with increasing viscosity, polyalkylene glycols can be used with natural and with synthetic rubbers in hydraulic oils and brake fluids. Easy removal by washing with water makes these suitable for applications that are difficult for other products. The toxicity of polyalkylene glycols is similar to that of glycerol for low-viscosity products and similar to that of isopropanol in more viscous products. This is of advantage in the food, pharmaceutical, tobacco, and cosmetics industries. Products with high ethylene oxide content are up to 80 % biodegradable. Polyalkylene glycols lower the freezing point of water. High viscosity water-soluble products are shear-stable liquid thickeners. Hydrophilic and hydrophobic fractions in block copolymers give them surfactant properties. The lubricating properties in mixed-friction areas and in rolling friction contacts can be improved by use of polyalkylene glycols with a broad molecular-weight range, for example; these can bridge the differences between water and the polyethers in terms of adhesion, viscosity, and volatility.

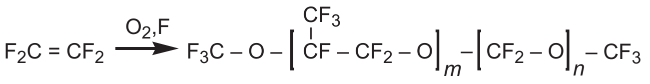
## 5.5
## Other Polyethers

Alkylated aryl ethers, like alkyl ethers, have lower viscosities, lower VI values, lower pour points, and lower boiling points than the corresponding alkanes. Asymmetric substitution also reduces the pour point, but has an adverse effect on the *V–T*. Although such products have low viscosities, some have found use as components of lubricants used in the life sciences.

### 5.5.1
### Perfluorinated Polyethers

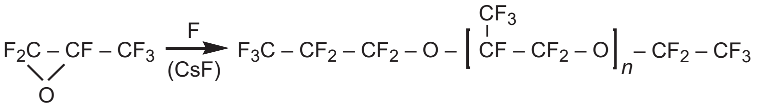
Perfluoroalkylpolyethers (PFPE) as lubricants were probably first mentioned by Gumprecht in 1965 [5.76]. Schwickerath and Del Pesco have described them in detail [5.77, 5.78].

Photochemical polymerization in the presence of oxygen, followed by fluorination with elemental fluorine leads to products of type A (Eq. 5.5), with tetrafluoroethylene. With hexafluoropropylene products of type B (Eq. 5.6), result. Anionic polymerization of hexafluoropropylene epoxide leads to products of type C (Eq. 5.7), and Lewis acid-catalyzed polymerization of 2,2,3,3-tetrafluorooxetane leads to products of type D (Eq. 5.8).

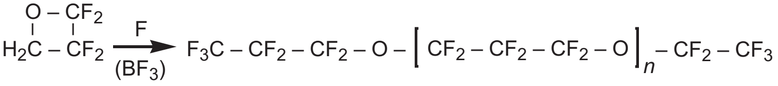


$$F_2C=CF_2 \xrightarrow{O_2,F} F_3C-O-\left[\overset{CF_3}{\overset{|}{C}}F-CF_2-O\right]_m-\left[CF_2-O\right]_n-CF_3 \quad (5.5)$$

$$F_2C=CF-CF_3 \xrightarrow{O_2,F} F_3C-O-\left[CF_3-CF_2-O\right]_m-\left[CF_2-O\right]_n-CF_3 \quad (5.6)$$



(5.7)



(5.8)

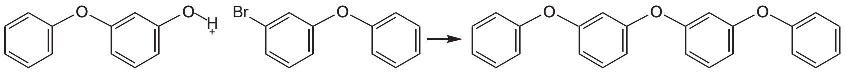
The density of the PFPEs is nearly twice that of hydrocarbons. They are immiscible with most of the other base oils and non-flammable under nearly all practical conditions. The more common types A and B have a good to very good V-T and V-p dependence, and low pour points [5.79]. The viscosity of linear PFPEs changes less with temperature and pressure than that of the non-linear variety, but has a non-negligible deviation from linearity, as has recently been reported [5.114]. In air they are stable up to 400 °C. Trifluoromethyl groups that are adjacent to the ether bonds shield them from acid-catalyzed cleavage, but difluoroformyl groups contribute to a decrease in stability at higher temperatures. PFPEs are remarkably inert chemically, this is including elastomers. Their hydrolytic stability is excellent. Good radiation stability, that was stated earlier, has been doubted [5.80].

The shear stability of PFPEs is better than that of other polymeric lubricants, but in the presence of steel and under boundary lubrication conditions PFPEs do not perform very well [5.81]. It has been reported that impurities adversely affect wetting properties [5.82]. The effect of humidity [5.83], and their thermo-oxidative behaviour have been investigated [5.84]. It has been found that the performance can be improved with α,β-diketones [5.85].

Perfluoropolyalkyl ethers have all the properties required by modern spacecraft: as lubricants and hydraulic fluids they resist to thermal and oxidative attack above 260 °C, possess good low-temperature flow characteristics, and are fire-resistant. They can also be used as power transmission and inert fluids and in transformers and generators as dielectrics with outstanding properties.

### 5.5.2
### Polyphenyl Ethers

Polyphenyl ethers **5.20**, are the reaction products of phenols and halogenated aromatic compounds (Eq. 5.9). They have been described in detail by Mahoney and Barnum [5.86]. Abbreviated formulas are in use. They contain the substitution position and the number of phenyl rings (ϕ) and ether bonds:



ppp5P4E stands for ϕ–O–ϕ–O–ϕ–O–ϕ–O–ϕ (p–p–p) 5.20 (5.9)

The aromatic groups increase the stability, but negatively affect the *V–T* dependence of these polyethers. Alkyl groups lower the high melting points. *para* Derivatives have lower volatilities; for *ortho* products the volatilities are higher. Spontaneous ignition occurs between 550 and 600 °C and alkyl substitution reduces this by ca. 50 °C. With the usual elastomers swelling occurs.

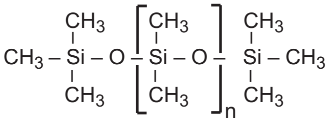
The oxidation stability of polyphenyl ethers is only slightly lower than that of polyphenyls or tetraarylsilanes. Alkyl substituents reduce it. The thermal decomposition temperatures are up to 465 °C. Short-chain substituents reduce it to below 380 °C and with higher alkyl groups it drops to below 350 °C, that is, to temperatures typical for aliphatic hydrocarbons. Trifluoromethyl groups have a worse effect, the thermal decomposition temperature drops below 270 °C. Coke formation is low. It increases with alkyl substitution, particularly in the presence of methyl groups.

Polyphenyl ethers are the most radiation-resistant lubricants. At low temperatures radiation affects the viscosity more pronouncedly than at high temperatures. It increases viscosity, acidity, evaporation loss, corrosivity, and coke formation, but reduces flash and ignition point.

Between 200 and 300 °C the lubricating properties of polyphenyl ethers are reported to be comparable with those of mineral and ester oils and better than those of polysiloxanes and aromatic hydrocarbons. Alkylated polyphenyl ethers have better properties than the unsubstituted ethers. Polyphenyl ethers are suited as high-temperature or radiation-resistant hydraulic fluids and lubricants [5.115], and as lubricants for optical switches [5.116].

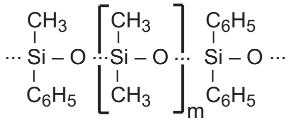
### 5.5.3
### Polysiloxanes (Silicone Oils)

Polysiloxanes can be liquids or solids. A well known book about their chemistry and technology has been written by Noll [5.87]. Silicone oils suitable as lubricants are generally straight-chain polymers of the dimethylsiloxane **5.21** and phenylmethylsiloxane series **5.22**. They have been described in detail by Awe and Schiefer and by Demby, Stoklosa, and Gross [5.88, 5.89].



$$CH_3-Si(CH_3)_2-O-\left[Si(CH_3)_2-O\right]_n-Si(CH_3)_2-CH_3$$

**5.21**



$$\cdots Si(CH_3)(C_6H_5)-O\cdots\left[Si(CH_3)_2-O\right]_m\cdots Si(C_6H_5)_2-O\cdots$$

**5.22**

Methyl silicone oils are simply made from quartz and methanol in the end (Fig. 5.6), but commercial technology employs sophisticated steps, e.g. the so-called equilibration–treatment of a mixture of siloxanes of different molecular weight with strong acid or basic catalysts with the aim of achieving a narrow Gaussian molecular weight distribution [5.90].

Among the unique properties of silicone lubricating oils is their immiscibility with many organic fluids, the low temperature-dependence of their physical properties, and their physiological inertness. It is possible to make them with both very low pour points and high viscosity by increasing the asymmetry of the molecules, usually by partial replacement of the dimethylsilyl groups by phenylmethylsilyl groups. The density of silicone oils is in the region of that of water, those of dimethylsilicone oils are slightly below, those of phenylmethyl silicone oils slightly above.

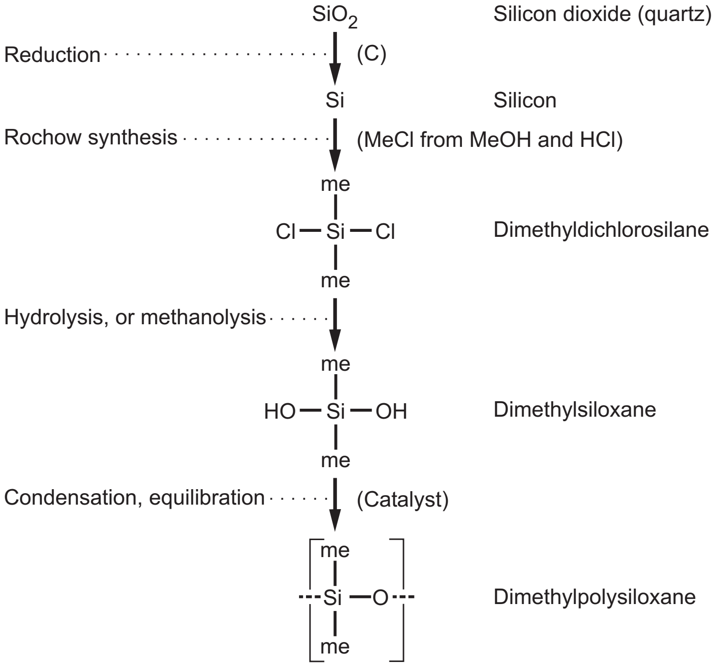


**Fig. 5.6** Preparation of dimethylsilicone oils.

The extremely low viscosity–temperature coefficients (VTC) [5.91], lower than 0.6 for low-viscosity dimethylsilicone oils are mainly due to the extraordinary flexibility of their Si–O chains. Low to medium viscosity silicone oils have Newtonian behaviour up to high shear rates, but with increasing viscosity the apparent viscosity decreases with increasing shear rate, that is, pseudoplastic behaviour is observed. Compressibility and viscosity changes at high pressure depend strongly on the methyl-to-phenyl ratio of the silicone oils and are relatively large, again mainly because of the flexibility of their Si–O chains.

The thermal decomposition of silicone oils begins at approx. 300 °C. In general the decomposition products are not corrosive, but they lower the viscosity of the oils. Up to 200 °C the oxidation stability of silicone oils is superior to that of hydrocarbons, esters, and polyalkylene glycols. The stability of phenylmethylsilicone grades, because of the resonance stabilization of the phenyl radicals, has been reported to exceed that of dimethylsilicone grades by up to 50 °C. Correlation of thermoanalytical and viscometric data of aged silicone oils has indicated an even larger figure [5.92]. At higher temperature the formation of siloxyl and silyl radicals via cross-linking leads to the formation of polymer molecules and thus results in gels.

Some iron and cerium compounds are especially suitable as oxidation inhibitors. At elevated temperatures gel formation nevertheless occurs readily in the presence of selenium or tellurium. In the presence of chlorine even explosions are possible.

Si–O bonds can also be broken by hydrolytic attack, but in contrast with silicic ester oils formation of silica or silicic acid gel does not occur.

The surface tension and the foaming tendency of silicone oils are much lower than those of mineral oils, but the thermal expansion of the same order of magnitude; silicone oils are, therefore, ideal defoamers for the hydrocarbons.

The lubricating properties of phenylmethylsilicone oils are better than those of the dimethylsiloxanes; trifluoropropylmethylsilicone oils perform even better [5.93]. Despite this they have been widely superseded by the perfluorinated polyethers.

Silicone oils can be found in all kinds of industrial and military installations. Dimethylsilicone oils are used as lubricants for bearings and gears with rolling friction. In sliding friction their performance depends on the metal pairs, they are used, for example, as lubricants for bronze or brass on aluminum, copper, or zinc at low surface pressures and as lubricants for porous bronze bearings. Silicone oils are among the best lubricants for plastic bearings, but in precision instrument applications spreading should be prevented by use of an epilamization agent. They are suitable lubricants for rubber parts, also, and also serve as switch and transformer oils. Silicone oils with higher phenyl substitution are mainly used for the lubrication of turbines, ball bearings, and all kinds of instrument, especially at high temperatures. Their radiation stability is also remarkable.

Silicone oils can be used as base oils for all kinds of lubricating greases, be it for sealing and damping applications, for reasons of chemical inertness, or for high-performance low- and high-temperature applications [5.94].

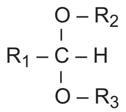
## 5.6
## Other Synthetic Base Oils

There have always been two reasons for the development of new synthetic lubricating oils. The first is to achieve a lower temperature-dependence of the physical properties, i.e. to make either higher or lower temperatures accessible; the second is to obtain better inertness, i.e. to prolong performance time. More recently a third reason must be added, better ecological properties combined, if possible, with physiological inertness.

Below –75 °C the performance of commercial dicarboxylic acid esters and siloxane oils ends and only some branched hydrocarbons, e.g. 3,3-dimethylhexane, seem to have suitable properties (melting points < –125 °C) but at the same time have all the disadvantages of their kind. Above 300 °C the practical performance of commercial perfluorinated polyethers ends and fluids comprising a stable element–carbon nucleus, e.g. C–N in some triazine derivatives and alkylated phenyl or silyl groups, must be considered. Above 400 °C the performance of perfluorinated polyethers ends, even under ideal conditions, and in the triazine derivatives the alkylated phenyl or silyl groups must be replaced by phenoxyphenyl groups. Tailored molecules with no C–H bonds at all can be imagined that will be suitable for temperatures above 500 °C [5.95].

Of the following group of synthetic fluids that have been proposed as base oils for lubricants or that have been developed especially for that use, none has gained yet real economic or commercial importance in that area during the last 30 years, all still wait for their niche application, even though some have really astonishing properties.

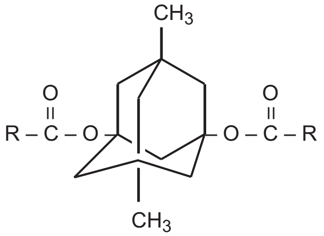
Acetals **5.23** have been investigated because of their high stability in alkaline media; they are, however, sensitive to acidic hydrolysis.



$$\begin{array}{l} \quad O-R_2 \\ \quad | \\ R_1-C-H \\ \quad | \\ \quad O-R_3 \end{array}$$

**5.23**

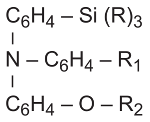
Adamantane derivatives, mainly the esters of 1,3-dihydroxy-5,7-dimethyladamantane, **5.24**, reminding of the extraordinary oxidative stability of adamantane itself, have been reported as being superior to the common dicarboxylic acid esters [5.96].



**5.24**

Alkylated carbosilanes [5.117], molecules with alternating Si and C atoms, are somewhat comparable with the tetraalkylsilanes, although of higher viscosity, and are even more stable thermally. Although known for decades, they are still waiting for suitable niches as lubricants, mainly because of problems with their technical scale synthesis.

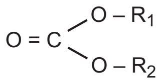
Aromatic amines, in particular their trialkylsilyl-substituted derivatives **5.25** have been considered as lubricants because of their good heat-resistance [5.95].



$$\begin{array}{l} C_6H_4-Si\,(R)_3 \\ | \\ N-C_6H_4-R_1 \\ | \\ C_6H_4-O-R_2 \end{array}$$

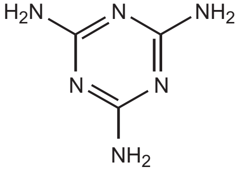
**5.25**

The performance of dialkylcarbonates, the diesters of carbonic acid **5.26**, is similar to that of common dicarboxylic acid esters, and have been reported to be preferable with regard to toxicity and seal compatibility [5.97]. Interesting, also, is that no acid compounds are formed on decomposition.

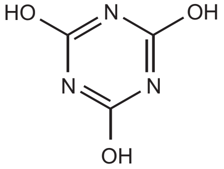


**5.26**

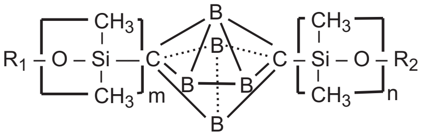
Heterocyclic Boron, Nitrogen, and Phosphorus Compounds are ring systems that do not consist only of carbon. It is replaced partially, for example by nitrogen. Triazine derivatives such as melamine **5.27** and cyanuric acid **5.28** form the nuclei for fire-resistant and high-temperature lubricants [5.95, 5.96]. Carbon can be in the minority as for example in polycarboranesiloxanes **5.29**, that have been reported to be comparable with silicone oils, but can be used at temperatures above 220 °C [5.98] and carbon can be entirely replaced by nitrogen and boron [5.99] or by nitrogen and phosphorus, as in the fluorine-containing phosphazene derivatives **5.30** that have been developed mainly for the use in fire-resistant high-temperature hydraulic fluids [5.100] and for use as lubricity additives in perfluorinated polyethers [5.101, 5.102].



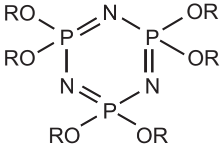
**5.27**



**5.28**



**5.29**

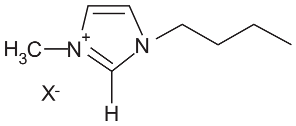


**5.30**

R = $C_6H_4F$, $C_6H_4 - CF_3$, ⋯

Room-temperature ionic liquids, salts that are liquid at room temperature, attracted interest as lubricants when the preparation of these air and moisture-stable, neutral products became possible [5.118]. The vapor pressure of these liquids is too low to measure, and they are non-flammable, thermally stable, have a wide liquid range, and have solvating properties for all kinds of material. Depending on the length of the cation side-chain and on the choice of the anion they can be made miscible with water or with organic solvents.

They consist of bulky organic cations, for example 1-alkyl-3-methylimidazolium **5.33**, and a wide range of anions, from tetrafluoroborate or hexafluorophosphate, to organic anions, for example bis-trifluorosulfonimide or tosylate. 1-Butyl-3-methylimidazolium tetrafluoroborate, for example, is a colorless liquid with a viscosity of approximately 100 $mm^2 s^{-1}$ at room temperature and a freezing point below –80 °C. Ionic liquids can be used – with water as additive – as lubricants for steel-steel contacts [5.119] or for steel-aluminum contacts [5.120], with or without water as heat-transfer fluids [5.121].

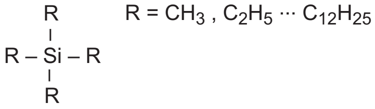


**5.33**

Polyalkylenesulfides, or Polythioether Oils are polyalkylene glycols in which all C–O–C bonds are replaced by C–S–C bonds; they have good oxidation stability, and higher viscosities and lower pour points than the corresponding hydrocarbons [5.103].

Polyphenylsulfides, or polyphenylthioethers, or C-ethers are polyphenylethers in which all C–O–C bonds are replaced by C–S–C bonds. This gives them lower pour points and better boundary lubrication properties, but reduced oxidative and thermal stability [5.104, 5.105].

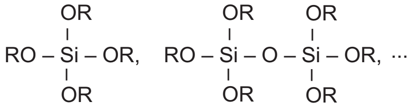
Compared with their hydrocarbon analogs, silahydrocarbons, mainly tetraalkylsilanes **5.31** have lower pour points, lower volatilities, higher VI values up to 155, and superior thermal stability [5.95, 5.106, 5.107]. They respond to antioxidants and antiwear additives in the same way as PAOs [5.108].



$$R-\underset{R}{\overset{R}{\mathrm{Si}}}-R \qquad R = CH_3, C_2H_5 \cdots C_{12}H_{25}$$

**5.31**

Silicate, or (ortho)silicic acid esters **5.32** have been known since the middle of the nineteenth century. They have properties that differ strongly from those of siloxanes. Despite their good thermal stability and their low pour points their use is restricted to hydraulic, heat-transfer, and cooling fluids and to oils for automatic weapons because of their poor hydrolytic stability [5.109]. With the development of polysilicate clusters the shortcomings of the simple esters can, perhaps, be overcome [5.110].



$$RO-\underset{OR}{\overset{OR}{\mathrm{Si}}}-OR, \quad RO-\underset{OR}{\overset{OR}{\mathrm{Si}}}-O-\underset{OR}{\overset{OR}{\mathrm{Si}}}-OR, \cdots$$

**5.32**

## 5.7
## Comparison of Synthetic Base Oils

It is of interest, of course, to compare the performance of synthetic base oils. Because these oils are mostly niche products for very specific applications and their costs are very different, there was some reluctance to publish a general comparison. A performance-related general ranking has, nevertheless, been attempted (Table 5.3) [5.111].

**Tab. 5.3** Performance ranking of base oils.

| | A | B | C | D | E | F | Σ |
|---|---|---|---|---|---|---|---|
| Alkylbenzenes | 2.5 | 1.0 | 2.5 | 3.0 | 2.0 | 3.0 | 2.3 |
| Naphthenic mineral oils | 2.5 | 2.0 | 2.0 | 2.5 | 2.0 | 2.5 | 2.2 |
| Paraffinic mineral oils | 2.0 | 3.0 | 2.0 | 2.5 | 2.0 | 2.0 | 2.3 |
| Hydrocracked mineral oils | 1.5 | 3.0 | 2.0 | 2.0 | 2.0 | 1.5 | 2.0 |
| Polyalphaolefines | 1.0 | 1.0 | 1.0 | 2.0 | 1.5 | 1.5 | 1.3 |
| Polyolesters | 1.5 | 1.5 | 2.0 | 1.0 | 1.5 | 1.5 | 1.5 |
| Poly(alkylene glycol)s | 2.0 | 1.5 | 2.0 | 1.5 | 1.5 | 1.5 | 1.7 |
| Silicones | 1.0 | 1.0 | 1.0 | 1.0 | 1.5 | 1.0 | 1.1 |
| Perfluorinated polyethers | 1.0 | 1.5 | 1.0 | 1.5 | 1.5 | 1.5 | 1.3 |
| Vegetable oils | 3.0 | 2.0 | 3.0 | 1.0 | 1.0 | 2.0 | 2.0 |

1.0 = excellent, 2.0 = fair, 3.0 = poor
A = high temperature, B = low temperature, C = ageing,
D = evaporation loss, E = toxicity, F = VT-behavior

It is beyond question that – when it comes to detail – there is no simple answer. High-temperature performance is an open field and, fortunately, tribochemistry [5.122], after two decades of being kind of a sleeping beauty, has again attracted the attention of lubrication specialists, with regard to comparison of engine oils and the role of micelles, for example [5.123]. Low-temperature performance proves to be equally complex, even with use of low-temperature DSC and NMR as tools [5.124]. And (non)toxicity, now environmental friendliness, is about to affect the results of comparisons more than in the past [5.125].

## 5.8
## Mixtures of Synthetic Lubricants

Today few lubricants contain only one base oil, firstly because mixing two or more base oils with different properties often leads to a lubricant with the desired performance, and secondly because many of the more polar synthetic base oils serve as additives in less polar oils, e.g. esters in hydrocarbons and vice versa. In thickened systems even immiscible base fluids like hydrocarbons and complex esters, perfluorinated ethers, or polyalkylene glycols can be combined.

# 6

# Additives

*Jürgen Braun*

Base fluids – mineral oil and also synthetic products – generally cannot satisfy the requirements of high performance lubricants without using the benefit of modern additive technology. Additives are synthetic chemical substances that can improve lots of different parameters of lubricants. They can boost existing properties, suppress undesirable properties and introduce new properties in the base fluids.

Additives can be classified regarding different aspects. Important and helpful for the understanding of additives is the following differentiation that takes into consideration which part of the tribo system is influenced by the additives. According to this considerations additives can be classified into types that

1) influence the physical and chemical properties of the *base fluids*
    - physical effects: e.g. VT characteristics, demulsibility, low temperature properties, etc.,
    - chemical effects: e.g. oxidation stability
2) affect primarily the *metal surfaces* modifying their physicochemical properties, e.g. reduction of friction, increase of EP behavior, wear protection, corrosion inhibition, ...

Additives are used at treat rates of a few ppm (antifoam agents) up to 20 or even more weight percentages. They can assist each other (synergism) or they can lead to antagonistic effects. Some additives are multifunctional products that decrease the possibility of additives interfering with each other negatively.

Also additives have a very big influence on the performance of lubricants that make it possible to fulfil new performance levels, of course there are some properties that cannot be influenced by additives, e.g. volatility, air release properties, thermal stability, thermal conductivity, compressibility, boiling point etc.

Also well balanced and optimized additive systems can improve the performance of lubricants enormously the formulation of high performance lubricants requires also excellent high quality base fluids. The present trend to use more and more hydrocracked and severely hydrotreated highly refined mineral oils as well as to synthetic esters and PAOs underlines this statement.

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## 6.1
## Antioxidants

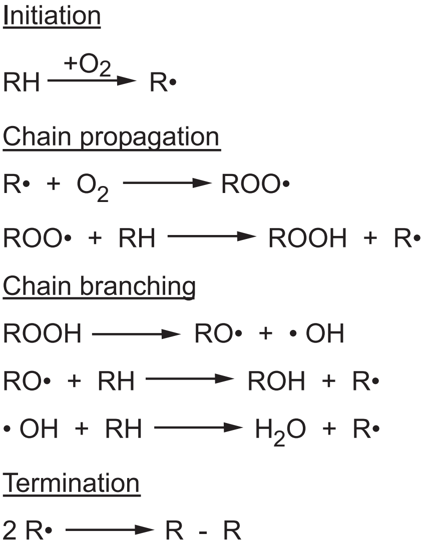
Industrial lubricants must fulfil many different functions, e.g. power transmission and wear protection just to name two of them. Mostly the function of a lubricant is limited by the aging of the lubricant base stock. Typical characteristics of aged lubricants are discoloration and a characteristic burnt odour. In advanced stages the viscosity will begin to rise significantly, acidic oxidation products are build, which in turn may induce corrosion and lubricant problems. This aging process can be delayed tremendously by the use of antioxidants.

### 6.1.1
### Mechanism of Oxidation and Antioxidants

The aging of lubricants can be differentiated into two processes: the oxidation process by reaction of the lubricant molecules with oxygen and the thermal decomposition (cracking) at high temperatures. In practice the oxidative aging of the lubricant is the dominating process which influences significantly the lifetime of the lubricant. Caused by steadily increased power density and reduced lubricant volumes (higher load-to-oil ratio) as well as extended service life in the last years, the thermal stress on the lubricant molecules grows constantly.

The oxidation of hydrocarbons can be described by the well-known free radical mechanism via alkyl and peroxy radicals [6.1]. The main reaction steps are shown in Fig. 6.1.

The initiation of the so-called autoxidation consists in the hydrogen abstraction of the hydrocarbon by oxygen attack that will lead to the formation of an alkyl radical. The alkyl radical can react with oxygen to form an alkyl peroxy radical. The next step



Initiation

$$\mathrm{RH} \xrightarrow{+O_2} \mathrm{R}\bullet$$

Chain propagation

$$\mathrm{R}\bullet + O_2 \longrightarrow \mathrm{ROO}\bullet$$

$$\mathrm{ROO}\bullet + \mathrm{RH} \longrightarrow \mathrm{ROOH} + \mathrm{R}\bullet$$

Chain branching

$$\mathrm{ROOH} \longrightarrow \mathrm{RO}\bullet + \bullet\mathrm{OH}$$

$$\mathrm{RO}\bullet + \mathrm{RH} \longrightarrow \mathrm{ROH} + \mathrm{R}\bullet$$

$$\bullet\mathrm{OH} + \mathrm{RH} \longrightarrow H_2O + \mathrm{R}\bullet$$

Termination

$$2\,\mathrm{R}\bullet \longrightarrow \mathrm{R-R}$$

**Fig. 6.1** Mechanism of autoxidation.

in the chain propagation scheme is the hydrogen abstraction by a peroxy radical from another hydrocarbon that will lead to a hydroperoxide and an alkyl radical which can again react with oxygen as described above. The difference in reactivity of the miscellaneous radicals explains why linear and unbranched hydrocarbons exhibit a much higher oxidation stability compared to branched, aromatic and unsaturated hydrocarbons. Further steps are chain branching (homolytic cleavage of hydroperoxides, increasing number of reactive free radicals, autocatalytic phase of the autoxidation) and the termination of the radical chain reaction by recombination of two radicals to yield unreactive, non-radical species.

The typical oxidation products that will be formed by these oxidation processes are alkylhydroperoxides (ROOH), dialkyl peroxides (ROOR′), alcohols (ROH), aldehydes (RCHO), ketones(RR′C=O), carboxylic acids (RCOOH), esters (RCOOR′) and so forth. By polycondensation processes high molecular weight oxidation products are formed. These products are responsible for the typical viscosity increase of aged oil. Further polycondensation and polymerization of these still oil soluble oxidation products lead finally to oil-insoluble polymers that can be observed as sludge and varnish-like deposits.

Because of the acidic character of most of the oxidation products the danger of corrosion is increased. Also the attack of alkylperoxy radicals on the metal surface may be responsible for corrosive wear. Furthermore such dissolved metals can form salts which also precipitate as sludge.

Non-hydrocarbon based lubricants may behave totally different. Thus the aging of polyalkylene glycols will lead to a decreasing of the viscosity caused by the decomposition of the polymeric structure.

Normally refined mineral base stocks contain traces of nitrogen-, sulfur- and oxygen-containing heterocycles as well as mercaptans (RSH), thioethers(RSR) and disulfides (RSSR) that may act as so-called ‘natural antioxidants’ or as pro-oxidants that will accelerate the oxidation of the lubricant. Usually the antioxidants are dominating thus providing a relatively good oxidation stability of the base oil. On the other hand hydrocracked base stocks (API group II and III) as well as synthetic lubricants like PAOs (API group IV) demand a well balanced antioxidant combination in order to benefit the advantages of that kind of lubricants [6.2–6.4].

Antioxidants can be differentiated as primary antioxidants (radical scavengers) and secondary antioxidants (peroxide decomposers).

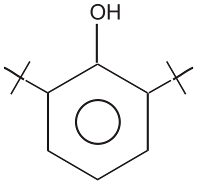
Radical scavengers compete successfully with the lubricant molecules in the reaction with reactive radicals of the propagation process. They react preferably with the radical oxidation products forming resonance-stabilized radicals that are so unreactive that they will stop the propagation of the autoxidation. Peroxide decomposers convert hydroperoxides into non-radical products thus also preventing the chain propagation reaction.

In finding the optimum synergistic antioxidant and metal deactivator combination the maximum delay of oil oxidation is achieved.

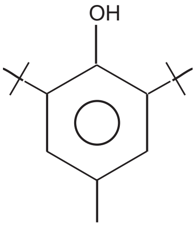
## 6.1.2
## Compounds

### 6.1.2.1 Phenolic Antioxidants

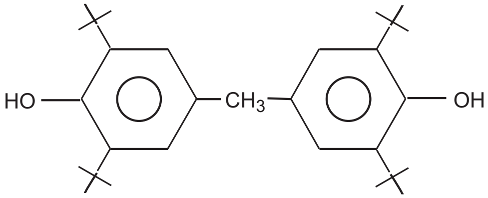
Sterically hindered mono-, di- and polynuclear phenol derivatives belong to the most effective antioxidants acting as radical scavengers and are used in many applications. Typically the phenols are substituted in the 2 and 6 positions with tertiary alkyl groups. The most common substituent is the tertiary butyl group. The most simple derivatives are *2,6-DTB (2,6-di-tert-butylphenol)* **6.1** and *BHT (butylated hydroxytoluene, 2,6-di-tert-butyl-4-methylphenol)* **6.2**. The advantage of polynuclear phenols like *4,4′-methylenebis(2,6-di-tert-butylphenol)* **6.3** or types with high-molecular-mass substituents in the 4 position is the reduced volatility due to the higher molecular weight that makes these products suitable for high temperature applications. Phenolic antioxidants can form degradation by-products (e.g. quinones) with intense yellow or red colors. Hindered phenols with large substituents in the para position are less prone to this effect, because the reaction pathway is more hampered.



**6.1**



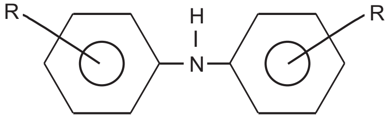
**6.2**



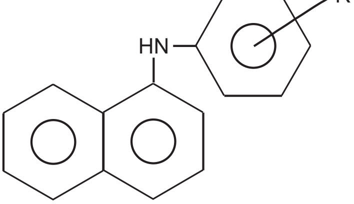
**6.3**

### 6.1.2.2 Aromatic Amines

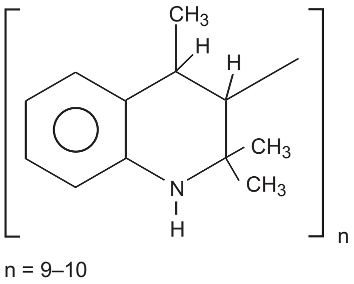
Oil-soluble secondary aromatic amines represent another important class of antioxidants that act as radical scavengers. Typical products are a large number of *alkylated diphenylamines* **6.4**, *N-phenyl-1-naphthylamine (PANA)* **6.5** and the *polymeric 2,2,4-trimethyldihydroquinoline (TMQ)* **6.6**. Because of its poor solubility in mineral oil the latter is commonly used in greases and polar lubricants.



**6.4**



**6.5**



**6.6**

At moderately elevated temperatures (< 120 °C) one diphenylamine molecule can eliminate 4 peroxy radicals whereas a monophenol can only scavenge two peroxy radicals. Under high-temperature conditions it could have been shown that aminic antioxidants are even still more superior to their phenolic counterparts [6.5]. Aminic antioxidants, on the other hand, tend to form strongly colored, dark brown degradation by-products (polyconjugated systems) finally resulting in dark colored oils even after relatively short usage. Aminic antioxidants, especially unsubstituted PANA, also have a greater tendency to form sludge than phenolic antioxidants [6.41].

#### 6.1.2.3 **Compounds Containing Sulfur and Phosphorus**

The most famous representatives of this group of additives are the zinc dithiophosphates which mainly act as radical scavengers [6.5]. Because of their multifunctional properties (antioxidant, antiwear additive, extreme pressure additive, corrosion inhibitor) zinc dithiophosphates retain their dominating role as standard additives since the early days of modern tribology in the beginning of this century.

A survey of the antioxidant properties of primary, secondary and aryl zinc dithiophosphates is given in Section 6.8.

Beside the well established group of metal dithiophosphates there are also a large number of ashless dithiophosphoric acid derivatives, the so-called O,O,S-triesters, which are reaction products of dithiophosphoric acid with olefins, cyclopentadiene, norbornadiene, $\alpha$-pinene, polybutene, unsaturated esters like acrylic acid esters, malenic acid esters and other chemicals with activated double bonds [6.5]. All these additives show antiwear and also antioxidant properties, although their performance as antioxidant is not really as good as that of the metal dithiophosphates.

#### 6.1.2.4 **Organosulfur Compounds**

These products are typical peroxide decomposers. Numerous types of organosulfur compounds have been proposed as antioxidants: dialkyl sulfides, diaryl sulfides, polysulfides, modified thiols, thiophene derivatives, xanthates, thioglycols, thioaldehydes, sulfur containing carboxylic acids, heterocyclic sulfur nitrogen compounds like dialkyldimercaptothiadiazoles, 2- mercaptobenzimidazoles and others.

Zinc- and methylenebis(dialkyldithiocarbamates) have been found to be highly efficient [6.5]. See also Section 6.8.3.

#### 6.1.2.5 Organophosphorus Compounds

Beyond this group triaryl- and trialkylphosphites are the most common types. Moreover they are not only peroxide decomposers, but they also can limit photodegradation. Because of their relatively poor hydrolytic stability their application is restricted to sterically hindered derivatives.

#### 6.1.2.6 Other Compounds

Surprisingly organocopper compounds in combination with peroxide decomposers can act as antioxidants although catalytic quantities of copper ions usually act as pro-oxidants [6.5]. Overbased phenates and salicylates of magnesium and calcium behave as antioxidants at higher temperatures. Many compounds have been proposed as antioxidants but none of these have gained real relevance.

#### 6.1.2.7 Synergistic Mixtures

Commonly several types and combinations of antioxidants are used. Well known is the synergistic effect when aminic and phenolic antioxidants are combined. This effect is based on the ability of the phenol to regenerate the more efficient aminic antioxidant [6.6]. The combination of radical scavengers and peroxide decomposers is called heterosynergism. So the combination of phenolic antioxidants with phosphites is known as highly efficient especially in hydrotreated base stock [6.7]. As metals may act as catalysts in the oxidation process typical rust inhibitors and metal passivators are also used as synergistic compounds (see also Section 6.10).

### 6.1.3
### Testing of the Oxidation Stability

For evaluation of antioxidants several bench tests have been established: turbine oil oxidation stability test (TOST) (DIN 51587, ASTM-D 943, ASTM-D 4310), rotary bomb test (ASTM-D 2272), IP 48, to name just a few. A detailed list of methods can be found in Chapter 18.

All tests are made under severe conditions by increasing the temperature, using catalysts (iron, copper) and high exposure to oxygen in order to shorten the time required to decompose the lubricant. Depending on the given set of oxidation conditions the antioxidants will exhibit different performance in different tests.

Therefore a variety of different oxidation tests is necessary to obtain an actual description of the lubricant's oxidation stability respectively the performance of the antioxidants. On the other hand a surprisingly good correlation of TOST and ROBOT results could have been shown recently for a given steam turbine formulation in different base stocks [6.8].

Basically oxidation tests can be differentiated into two groups: one kind of tests describe the condition of the lubricant after a defined test period by measuring several aging indicating parameters like acid number, viscosity change and sludge formation. The 1000-h TOST test according to DIN 51587 and IP 280 oxidation test are examples of that kind of test. Another group of oxidation tests measures the so-called induction time (time from the beginning of the oxidation to the autocatalytic phase of the autoxidation) by recording constantly or after defined periods of time the aging indicating parameters. Typical examples of this group are the lifetime TOST test according to ASTM-D 943 or the rotary bomb test (ASTM-D 2272) where the drop of oxygen pressure indicates the aging of the lubricant. More recently the high pressure differential scanning calorimetry (HPDSC) as well as the sealed capsule differential scanning calorimetry (SCDSC) has seen more application measuring the stability of different antioxidants and formulations [6.9, 6.10]

## 6.2
## Viscosity Modifiers

### 6.2.1
### Physical Description of Viscosity Index

A fundamental characteristic of every fluid is its viscosity (see Chapter 3). The kinematic viscosity of a fluid is dependent on the external parameters of pressure and temperature. A great number of applications specify viscosity at a defined temperature, in the case of hydraulic equipment at 40 °C and 100 °C, to achieve optimum pump efficiency [6.11].

The effect of temperature on a fluid can be illustrated double-logarithmically according to Walter (see Section 3.2), whereby the gradient defines the viscosity index. The viscosity index is defined exactly by the gradient between 40 and 100 °C.

This simple-to-calculate characteristic has a significant impact on lubrication technology and is practically a standard feature of every specification. The viscosity index defines molecular structure and has been exactly described for defined molecules [6.12].

Depending on their source, conventionally refined base oils display between 80 and 120 VI points and the most common, central European paraffinic solvent cuts have VIs of around 100 points.

As this type of base oil still accounts for the largest part of conventional lubricants (see Chapter 8), the improvement of viscosity index is best described using this example.

### 6.2.2
### VI Improvement Mechanisms

In the simplest of cases, a desired viscosity index can be achieved by mixing fluids with corresponding VIs. Usually, however, the viscosity requirements of modern lubricant specifications can be met only by addition of viscosity modifiers (VMs) also known as viscosity index improvers (VIIs).

As opposed to those present in low-molecular base fluids, viscosity modifiers have a polymer nature [6.13]. These molecules are described as being chain-like molecules whose solubility depends on chain length, structure and chemical composition [6.14].

As a rule, the base oil solubility of these polymer chains deteriorates as the temperature falls and improves with increasing temperature so that an increase in viscosity induced by viscosity modifiers also increases the viscosity index. In 1958 T. W. Selby published a descriptive explanation of the mechanism of VMs [6.42]. Because of poor solubility at low temperatures the chain-like VM molecules form coils of small volume and as the temperature is increased these molecules expand and unravel, resulting in an increasing beneficial effect on high-temperature viscosity.

The absolute increase in viscosity and the VI depends on the type, the molecular weight and the concentration of viscosity modifiers in the formulation [6.15]. In practice and depending on the projected application, molecular weights of 15 000 to 250 000 g $mol^{-1}$ for PAMAs and 2 000 to 5 000 g $mol^{-1}$ for PIBs, to name just two, are used. Concentrations are usually between 3 and 30 % (w/w). As a result of their high molecular weight, viscosity modifiers are always dissolved in a base fluid.

Apart from their thickening effect which is schematically illustrated in Fig. 6.2A as a function of molecular weight, shear stability serves as a second characteristic [6.16]. According to Fig. 6.2B, increasing molecular weight reduces shear stability if the polymer concentration remains constant.

The reason for this effect is mechanically- or thermally-induced chain degradation [6.17]. As opposed to Newtonian fluids, whose viscosity is independent of the rate of shear or the velocity gradient, long-chain compounds subject to high shearing are mechanically broken. Depending on the type and duration of the load, a number of different molecular sizes are created. The resulting drop in viscosity is described by the Permanent Shear Stability Index (PSSI) which describes the percentage loss of the contribution of the polymer to the viscosity (viscosity increase by the VM compared with base oil viscosity), Fig. 6.3.

Under conditions of high shear stress, if the relaxation time for the polymer chains is short ($10^{-6}$ s) the high-molecular-weight molecules adopt a temporary alignment. In Selby's model the polymer coils are deformed in the direction of the shear force and lose part of their original contribution to the viscosity. This reversible drop in viscosity is described by the temporary shear stability index (TSSI).

Both values are of great importance to automotive applications, especially to engine, gear and hydraulic oils because the specified characteristics of such an oil

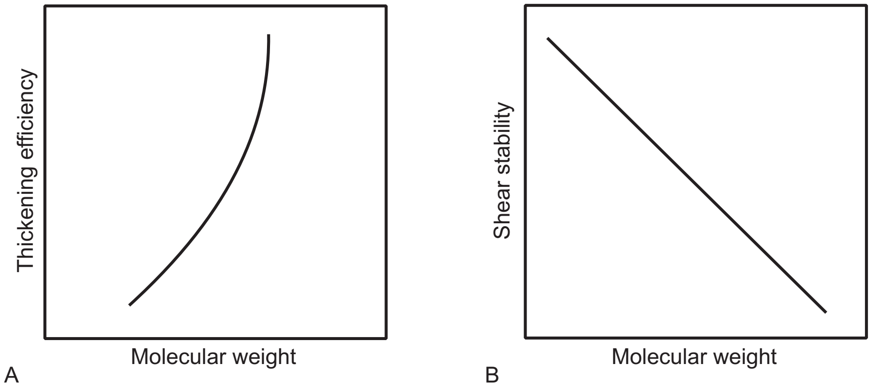
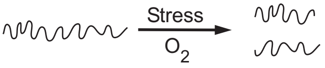


**Fig. 6.2** Thickening efficiency and shear stability of VMs.

**1. Degradation of polymers**: Mechanical stress
Oxidative bond cleavage



**2. Shear loss (permanent):**

$$\frac{[\text{KV before shearing}] - [\text{KV after shearing}]}{[\text{KV before shearing}]}$$

e. g. $$\frac{11\ \text{cSt} - 8\ \text{cSt}}{11\ \text{cSt}}$$

**3. Shear stability index (SSI):**

$$\frac{[\text{KV before shearing}] - [\text{KV after shearing}]}{[\text{KV before shearing}] - [\text{KV of the base oil}]}$$

e. g. $$\frac{11\ \text{cSt} - 8\ \text{cSt}}{11\ \text{cSt} - 5\ \text{cSt}}$$

**Fig. 6.3** Mechanism and description of the shear loss.

do not just apply to the fresh oil but should remain throughout the drain interval. Although the reduction in molecular weight is, in practice, overshadowed by oxidation and other effects, a series of laboratory tests has been established to characterize shear stability [6.18]. The tests are shown in Fig. 6.4; they are listed in order of test severity which increases in the order Bosch diesel injection pump test, 30 cycles < Bosch diesel injection pump, 250 cycles < tapered roller bearing test, 4 h ~ sonic shear test, 40 min < tapered roller bearing test 20 h.

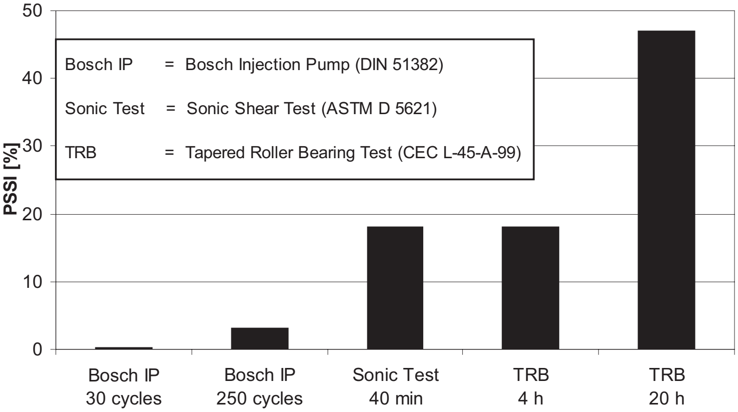
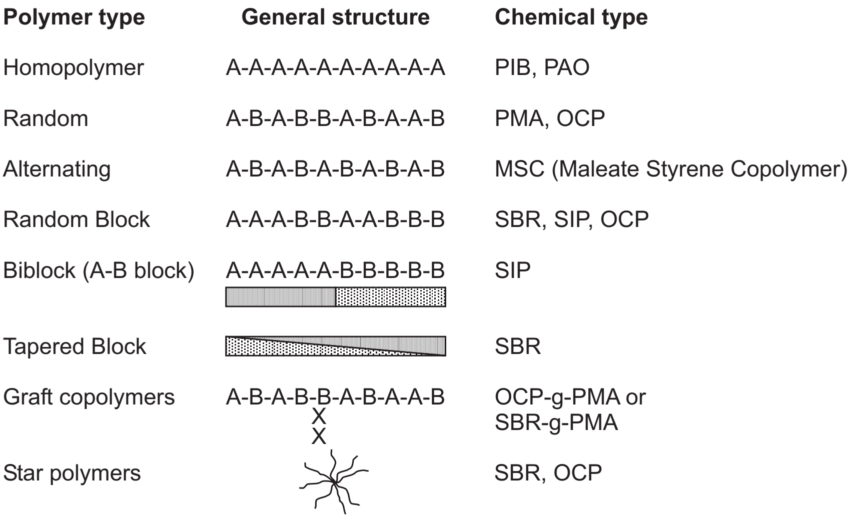


**Fig. 6.4** Laboratory shear-stability tests.

### 6.2.3
### Structure and Chemistry of Viscosity Modifiers

Apart from the fundamental description of thickening effect and shear stability, the expert differentiates viscosity modifiers by the molecular structure, composition and chemical nature of the individual chain links (monomers).



| Polymer type | General structure | Chemical type |
|---|---|---|
| Homopolymer | A-A-A-A-A-A-A-A-A-A | PIB, PAO |
| Random | A-B-A-B-B-A-B-A-A-B | PMA, OCP |
| Alternating | A-B-A-B-A-B-A-B-A-B | MSC (Maleate Styrene Copolymer) |
| Random Block | A-A-A-B-B-A-A-B-B-B | SBR, SIP, OCP |
| Biblock (A-B block) | A-A-A-A-A-B-B-B-B-B | SIP |
| Tapered Block | | SBR |
| Graft copolymers | A-B-A-B-B-A-B-A-A-B<br>X<br>X | OCP-g-PMA or<br>SBR-g-PMA |
| Star polymers | | SBR, OCP |

**Fig. 6.5** Viscosity modifiers, chain structures, and monomer make-up.

The most important monomers for viscosity modifiers are shown in Table 6.1 as structural formulas in the typical nomenclature for homo- and copolymers.

As a listing according to monomer units would not be worthwhile due to the vast number of combination possibilities, a series of polymer types have found favor for certain applications (Table 6.1).

It is also possible to sort these polymers according to their supramolecular chain structure, independently of the base monomers used (Fig. 6.5).

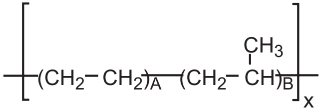
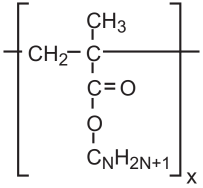
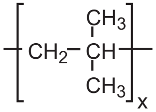
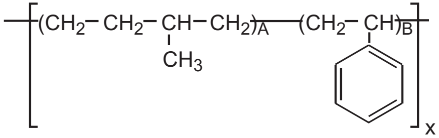
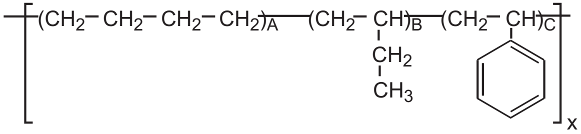
In this case, a differentiation is made between linear and branched chains. Chain branching can create comb- or star-shaped structures. Depending on the polarity of the monomer, the viscosity modifiers are either dispersing or non-dispersing. Dispersing viscosity modifiers are in principle the link to ashless dispersants which are discussed in Section 6.4.3.

## 6.3
## Pourpoint Depressants (PPD)

With the exception of polyalkylated naphthalenes, pour-point depressants (PPD) are closely linked to a series of viscosity modifiers. The major difference of these polymers is their application concentration and the selection of monomer building blocks [6.19]. Molecular weight and thickening efficiency only play a subordinate role in a band from 0.1 to max. 2 %.

An additional thickening effect is always welcome but is usually limited by solubility thresholds.

**Tab. 6.1** Types of polymeric viscosity modifier.

| ***VM Type*** | | ***Description*** | ***Main applications*** |
|---|---|---|---|
| OCP |  $[(CH_2-CH_2)_A-(CH_2-CH(CH_3))_B]_x$ | Olefin Copolymers | Engine and hydraulic oils |
| PAMA |  $[CH_2-C(CH_3)(C(=O)-O-C_NH_{2N+1})]_x$ | Polyalkyl(meth)acrylates | Gear and hydraulic oils |
| PIB |  $[CH_2-CH(CH_3)(CH_3)]_x$ | Polyisobutylene | Gear oils, raw material for ashless dispersants |
| SIP |  | Hydrogenated styrene–isoprene copolymers | Engine oils |
| SBR |  | Hydrogenated styrene–butadiene | Engine oils |

As is generally the case with flow improvers, although paraffin crystallization cannot be suppressed, the crystalline lattice and thus the morphology of the paraffin crystals can be significantly altered. While polyacrylates and ethylene vinyl acetate copolymers are used for crude oils and petroleum, special polyalkyl methylacrylates are normally used for mineral oil-based formulations. The fundamental background for this is the previously-mentioned co-crystallization of paraffinic components in the base oil and the polymer chain. This interaction results in an alteration of the crystal morphology. Instead of the needle-like paraffin crystals which rapidly cause paraffin gelation, densely packed, round crystals are formed which hardly effect flowing properties even at temperatures below the pour point. The corresponding PAMA-PPDs are in principle, comb-like and contain $C_{12}$–$C_{24}$ paraffinic side chains [6.20]. By determining the polymer solubility and crystallization of paraffinic components in the base oil, the optimum PPD-mix and concentration can be determined for every base oil mixture. However, as a wide range of base oils are used in practice, standard products are often used which cover almost the entire spectrum. In such cases, efficiency is controlled via PPD concentration.

## 6.4
## Detergents and Dispersants

Detergents and dispersants, often called DD or HD (heavy-duty) additives have been indispensable for the development of modern engine oils for gasoline and diesel combustion motors. These lubricants are especially severely stressed due to the high temperatures that they are exposed to and the additional influence of aggressive blow-by gases of the combustion process. DD additives keep oil-insoluble combustion products in suspension and also prevent resinous and asphalt-like oxidation products from agglomerating into solid particles. The overbased metal-containing compounds additionally are able to neutralize acidic combustion products as well as oxidation products by their alkaline reserve. Thus, DD additives prevent oil thickening, sludge and varnish deposition on metal surfaces and corrosive wear.

The original definition of detergents refers to their cleaning properties similar to the detergents in washing agents although their function appears to be more the dispersing of particulate matter like abrasive wear and soot particles rather than cleaning up existing dirt. Historically these kind of additives have been metal-containing compounds, often with high alkaline reserve.

To meet the dramatically increased requirements of modern high-performance engine oils new ashless dispersants with improved dispersing properties have been developed. As these ashless compounds possess also cleansing properties in fact there is no real difference between detergents and dispersants and it is more an arbitrary definition to call the metal-containing compounds as detergents and the ashless types as dispersants. Thus it seems to be more appropriate to speak of metal-containing and ashless DD or HD additives [6.21, 6.22].

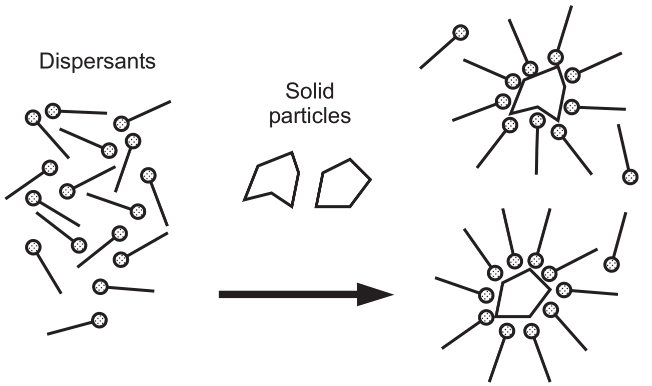


Fig. 6.6 Function of dispersants – peptidization process.

### 6.4.1
### Mechanism of DD Additives

Detergents and dispersants are generally molecules having a large oleophilic hydrocarbon 'tail' and a polar hydrophilic head group. The tail section serves as a solubilizer in the base fluid, while the polar group is attracted to contaminants in the lubricant. A multitude of dispersant molecules are able to envelope solid contaminants forming micelles whereby the non-polar tails prevent the adhesion of polar soot particles on metal surfaces as well as the agglomeration into larger particles. Fig. 6.6 illustrates this process that is generally known as peptidization.

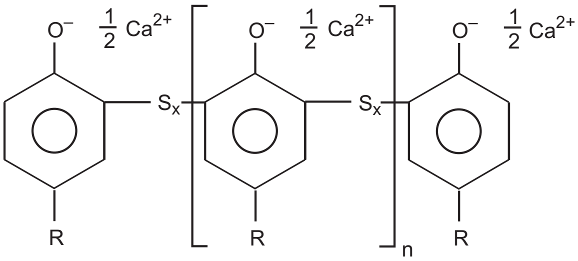
### 6.4.2
### Metal-containing Compounds (Detergents)

#### 6.4.2.1 Phenates

Phenates represent an important class of detergents which are synthesized by reaction of alkylated phenols with elemental sulfur or sulfur chloride followed by the neutralization with metal (calcium, magnesium, barium) oxides or hydroxides. Calcium phenates **6.7** are currently the most widely used types. Basic calcium phenates **6.8** can be produced by using an excess of the metal base. Beside their good dispersant properties they also possess greater acid neutralization potential.

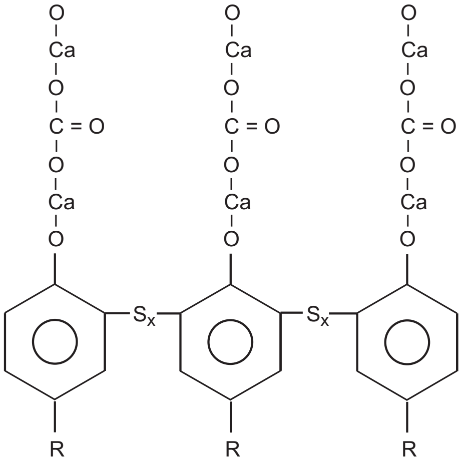
#### 6.4.2.2 Salicylates

Salicylates **6.9** are generally prepared by carboxylation of alkylated phenols with subsequent metathesis into divalent metal salts. Typically also these products are overbased by an excess of metal carbonate (calcium and magnesium) to form highly basic detergents that are stabilized by micelle formation. Salicylates exhibit additional antioxidant properties and have proven effective in diesel engine oil formulations.

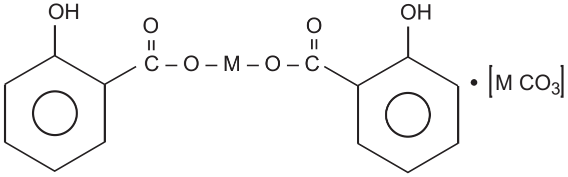


**6.7**

R = Alkyl group
X = 1–2
n = 0–2



**6.8**



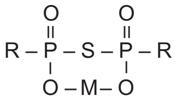
**6.9**

M = Ca, Mg

### 6.4.2.3 Thiophosphonates

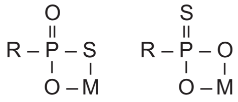
These representatives of the detergents are produced by the reaction of polybutene (molecular weight from 500 to 1000) with phosphoruspentasulfide followed by hydrolysis and formation of metal (calcium, formerly also barium) salts.

The reaction products consist mainly in thiopyrophosphonates **6.10** combined with thiophosphonates **6.11** and phosphonates **6.12** [6.23]. Overbased products have almost vanished from use.



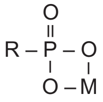
Thiopyrophosphonate

**6.10**



Thiophosphonates

**6.11**



Phosphonate

**6.12**

#### 6.4.2.4 **Sulfonates**

Sulfonates are metal salts of long-chain alkylarylsulfonic acids which can be divided into the petroleum and synthetic types. For more details see Section 6.10.1. Beside their excellent anticorrosion properties neutral and especially so-called overbased sulfonates with colloidally dispersed metal oxides or hydroxides (Fig. 6.7) additionally exhibit an excellent detergent and neutralization potential that makes them very cost-effective multifunctional DD additives for engine oils.

Calcium sulfonates are relatively cheap products with good general performance. Magnesium compounds distinguish by excellent anticorrosion properties but tend to form hard ash after thermal decomposition. Deposits of hard ash can lead to bore polishing. Barium sulfonates are hardly used anymore due to toxicological concerns.

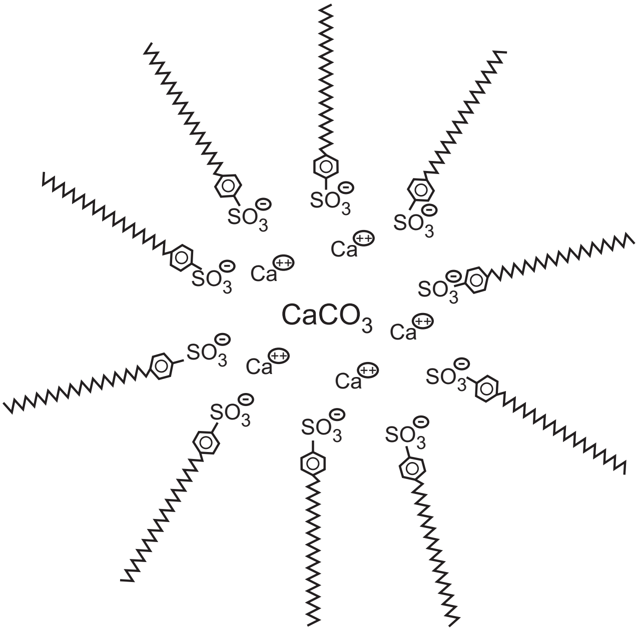


**Fig. 6.7** Model of the structure of overbased sulfonates.

### 6.4.3
### Ashless Dispersants (AD)

As opposed to detergents containing metals, ADs are by definition free of metals. They are generally derived from hydrocarbon polymers. The best known and economically most interesting raw material group are polybutenes with molecular weights of 500 to 3000 g $mol^{-1}$ [6.24].

As a result of the cationic polymerization of a C4 cut, polybutenes (PIB) with double bonds are formed, which can be thermally coupled with maleic anhydride (MA), to give PIBSA (polyisobutene succinic acid anhydride). In addition to the thermal synthesis of PIBSA a chlorine-catalyzed production process is also still in use. In a further reaction step oligomeric aminoalkylenes are added to the anhydride to form thermally stable imides. By using suitable raw materials and reaction control, this two-stage, single vessel reaction can produce a variety of products. The reaction schematic in Fig. 6.8 shows just one possible reaction and, for illustration purposes only, just one PIBSI (polyisobutene succinimide) as the final product. In general, there are mono and bis succinimides.

The amine functions of PIBSI and free unreacted, low-molecular-weight amines in particular, participate in undesirable reactions with fluorocarbon elastomers. Different methods are used to reduce the aggressiveness of amine groups, e.g. blocking

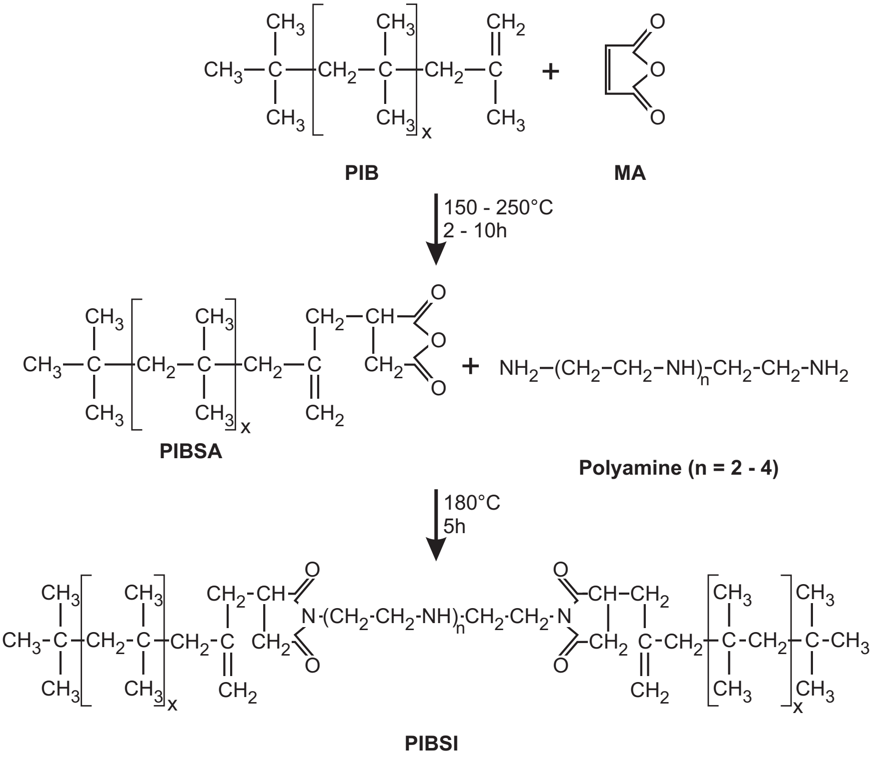
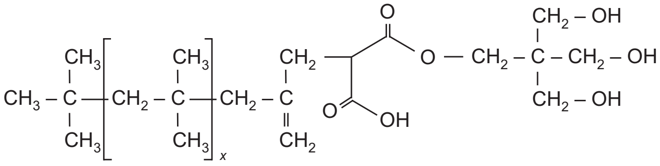


Fig. 6.8 Reaction mechanism for the manufacture of ashless dispersants.

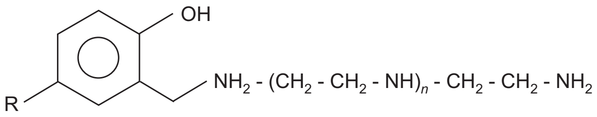
by chemical reaction with boric acid. In addition to bifunctional amines (triethylene tetramines, tetraethylene pentamines), alcohols such as pentaerythritols are used as polar groups to improve thermal stability and elastomer compatibility. These polyisobutene succinic esters (PIBSE) **6.28** are often used in combination with PIBSIs, because they are less efficient as dispersants than PIBSIs. Intramolecular combinations (polyisobutene succinic acid ester amides) are also known.



$X$ = 8–45, typically 18

**6.28**

Alkylphenolamines (Mannich bases) **6.29** have lost importance, because they are very aggressive toward fluorocarbon elastomers.



R = long-chain alkyl

**6.29**

$n$ = 1–4

Because advanced PCMO formulations contain almost twice the amount of ADs as in the past, the search is on for highly efficient alternatives. Dispersion of oxidation, nitration, and soot particles can be controlled by selection of the appropriate product mixture and by use of components of adequate purity.

The disadvantage of conventionally-manufactured PIB-based oligomers is their limited functionality and their wide molecular weight distribution. Interesting developments [6.25, 6.26] have lead to hydrocarbon oligomers with a high percentage of double bonds of the end of the chain. This can be achieved by the cationic polymerization of isobutylenes or by the metallocene oligomerization of α-olefins [6.27]. The raw materials can be further altered using gentle, chlorine-free processes.

Equally chlorine-free alternatives are low-molecular-weight, dispersant PAMAs. Contrary to hydrocarbon-based oligomers, the dispersing units are spread along the chains, whereby the type of dispersing groups can vary greatly [6.28]. Current research reports advantages with regard to soot dispersing and improved fuel efficiency characteristics [6.29].

## 6.5
## Antifoam Agents

The foaming of lubricants is a very undesirable effect that can cause enhanced oxidation by the intensive mixture with air, cavitation damage as well as insufficient oil transport in circulation systems that can even lead to lack of lubrication. Beside negative mechanical influences the foaming tendency depends very much on the lubricant itself and is influenced by the surface tension of the base oil and especially by the presence of surface-active substances such as detergents, corrosion inhibitors and other ionic compounds.

Very important for the understanding of foaming effects is the difference between the so-called surface foam and the inner foam.

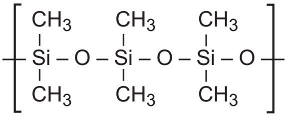
*Surface foam* can be controlled by antifoam agents. Effective defoamers possess a lower surface tension compared to the lubricant base oil, are usually not soluble in the base oil and therefore have to be finely dispersed in order to be sufficiently stable even after long-term storage or use. The particle size of the dispersed defoamers should be smaller than 100 μm or even smaller than 10 μm [6.30].

The *inner foam* refers to finely dispersed air bubbles in the lubricant that can form very stable dispersions. Unfortunately the common defoamers dedicated to control the surface foam tend to stabilize the inner foam. Generally the air release properties of lubricants cannot be improved by additives. In the contrary lots of additives have a negative influence. Lubricants which need excellent air release properties e.g. turbine oils have to be formulated using specially selected base oils and additives.

Air is not only present in the lubricants in form of dispersed air bubbles (surface and inner foam) but can also be truly physically dissolved up to 9 % (*v*/*v*) in mineral oil. Also this air can cause severe problems like cavitation ('diesel effect') but this effect cannot be controlled by additives.

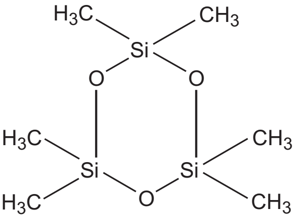
### 6.5.1
### Silicon Defoamers

Liquid silicones, especially linear **6.13** and cyclic polydimethylsiloxanes **6.14**, are the most efficient antifoam agents at very low concentrations of 1 to max. 100 mg $kg^{-1}$. To guarantee a stable dispersion silicones usually are predissolved in aromatic solvents.



$$\left[\begin{array}{c}CH_3\\|\\-Si-O-\\|\\CH_3\end{array}\begin{array}{c}CH_3\\|\\Si-O-\\|\\CH_3\end{array}\begin{array}{c}CH_3\\|\\Si-O-\\|\\CH_3\end{array}\right]$$

**6.13**



**6.14**

Compared to other additives silicone defoamers have the disadvantage of being particularly easily carried out of the lubricant due to their insolubility and their strong affinity to polar metal surfaces.

### 6.5.2
### Silicone-free Defoamers

Nowadays silicone-free defoamers are used more and more in many applications. Especially in metalworking processes cutting fluids as well as hydraulic fluids used close by have to be silicone-free to guarantee the subsequent application of paints or lacquers on the workpieces. Silicones have caused lots of problems in this application.

The main representatives of silicone-free defoamers are special poly(ethylene glycols) (polyethers), polymethacrylates, and miscellaneous organic copolymers. Also tributylphosphate has been proposed as antifoam agent.

## 6.6
## Demulsifiers and Emulsifiers

### 6.6.1
### Demulsifiers

Most of the industrial oils in circulation systems (hydraulic, gear, turbine and compressor oils) require good or excellent demulsification properties to separate water contamination from the lubricating system. Without demulsifiers lubricating oils can form relatively stable water-in-oil emulsions.

In principle, all surface-active substances are suitable demulsifiers. One of the first known types have been alkaline-earth metal salts of organic sulfonic acids particularly barium and calcium dinonylnaphthenesulfonates. Nowadays special polyethylene glycols and other ethoxylated substances have proved to be highly efficient demulsifiers which are often part of many additive packages.

Surprisingly the same class of chemical substances is used as emulsifiers. Here the molecular weight, the degree of ethoxylation and the treat rate are very important to guarantee the demulsifying properties.

### 6.6.2
### Emulsifiers

Because of their importance for the formulation of water-based metal working fluids, types and mechanism of emulsifiers are described in Chapter 14.

## 6.7
## Dyes

For marketing, identification or leak detection purposes some lubricants contain dyes which are classified according to the International Color Index [6.31]. Most of these substances are solids which often are suspended in mineral oil or dissolved in aromatic solvents to make their handling easier.

Generally oil-soluble azo dyes are used. Some of these products may be removed in the near future because they have to be labeled as potentially carcinogenic.

Fluorescent dyes are typically used to detect leaks under UV light when the coloration of lubricants is not appreciated.

## 6.8
## Antiwear (AW) and Extreme Pressure (EP) Additives

When two contacting parts of a machinery start to move and the hydrodynamic lubrication has not yet build up or in the case of severe stress and strong forces the lubricating system runs in the area of mixed friction. In this case antiwear (AW) and extreme pressure (EP) additives are necessary in any metalworking fluid, engine oil, hydraulic fluid or lubricating grease to prevent welding of the moving parts respectively to reduce wear.

### 6.8.1
### Function of AW/EP Additives

Because of their polar structure these additives form layers on the metal surface by adsorption or chemisorption that guarantees their immediate availability in the case of mixed friction conditions. When the hydrodynamic lubricating film is not yet or no longer present, temperature will increase and the AW and EP additives can react with the metal surface forming tribochemical reaction layers (iron phosphides, sulfides, sulfates, oxides and carbides – depending on the chemistry of the additive) that will prevent direct contact between the sliding metals. These friction reducing, slideable reaction layers can smooth the asperity of the metal surface by plastic deformation and reduce wear that would occur due to microwelding processes respectively avoid real welding of the moving parts under extreme pressure conditions.

Layers formed by only physically adsorbed polar substances like fatty oils, fatty acids and others exhibit only poor or moderate high pressure properties. These kind of additives are called friction modifiers (see Section 6.9). More effective and more stable are chemically reactive products (AW and EP additives) that can form tribochemical reaction layers.

AW additives are mainly designed to reduce wear when the running system is exposed to moderate stress whereas EP additives are much more reactive and are used when the stress of the system is very high in order to prevent the welding of the moving parts that otherwise would lead to severe damage. Typically EP additives increase wear effects due to their high reactivity.

This differentiation cannot be precise and there are many additives that can be related to both groups.

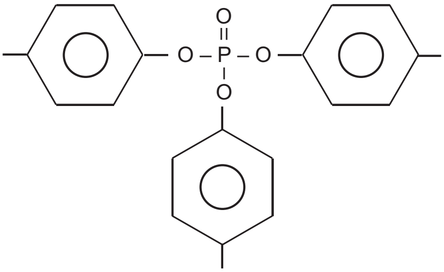
## 6.8.2
## Compounds

### 6.8.2.1 Phosphorus Compounds

Under condition of medium stress organic phosphorus compounds work excellent as antiwear additives. Some of them are highly efficient FZG boosters.

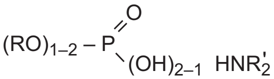
Most of these additives are neutral and acidic phosphoric acid ester derivatives, their metal or amine salts or amides. As the acidic form of these compounds is the most reactive one, the reactivity decreases with the degree of neutralization.

*Trialkyl- and triarylphosphates* represent the neutral phosphoric acid triesters, where the *tricresylphosphate (TCP)* **6.15** is the most known species. For toxicological reasons TCP should be free of *o*-cresol.



**6.15**

*Amine neutralized mixtures of mono- and dialkyl phosphoric acid partial esters* **6.16** are highly efficient FZG boosters and widely used in ashless hydraulic oil formulations. Some types exhibit additional anticorrosion properties.



$$(RO)_{1-2}-P(=O)(OH)_{2-1}\ HNR'_2$$

**6.16**

*Ethoxylated mono- and dialkylphosphoric acids* are even more polar by their hydrophilic structure what makes them more efficient.

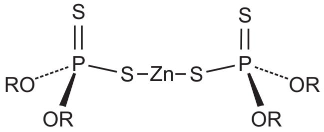
Also *phosphites* have generated much interest. Because of the inherent hydrolytic instability of this chemical group in practice only sterically hindered derivatives like triarylphosphites and long chain trialkylphosphites are used for some applications. Beside their antiwear properties they are able to scavenge free sulfur by forming thiophosphates by which their use in gear oils is based.

*Phosphonates and phosphines,* the derivatives of the phosphonic and phosphinic acid, have also been proposed as antiwear additives but are not commonly used [6.32].

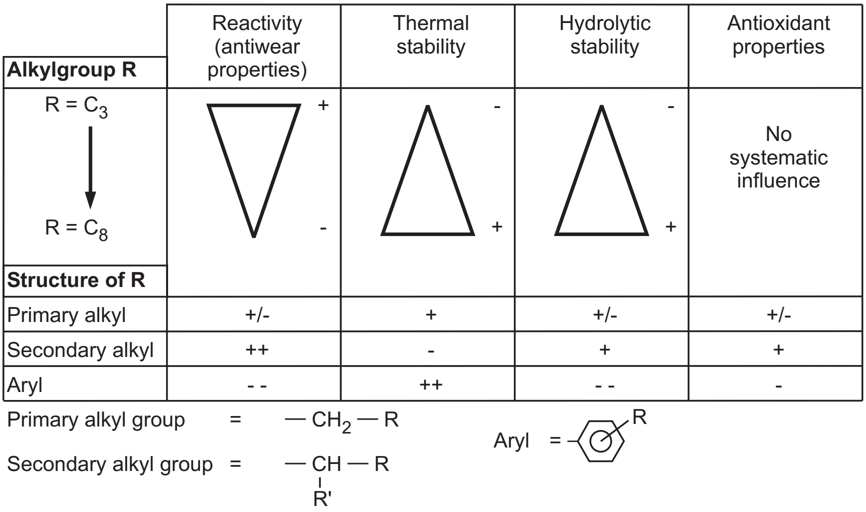
### 6.8.2.2 Compounds Containing Sulfur and Phosphorus

The most important and well known additive group of the sulfur–phosphorus compounds are the *zinc dialkyldithiophosphates (ZnDTP)* [6.33]. They are synthesized by reaction of primary and secondary alcohols ($C_3$–$C_{12}$) as well as alkylated phenols with phosphorus(V)sulfide followed by neutralization of the resulting dialkyldithiophosphoric acids with zinc oxide. Usually this last reaction step is done in mineral oil solution but it is also possible to make mineral oil free compounds by using solvents that can be removed afterwards by distillation. Beside the neutral species also basic ZnDTPs can be obtained when the neutralization is done with an excess of zinc oxide.

ZnDTPs based on isopropanol or *n*-butanol are solids, whereas mixtures of short and long chain alcohols are liquid. The thermal and hydrolytic stability of ZnDTPs and thus their reactivity (AW/EP-performance) can be influenced by the structure of the alkyl groups. So the thermal stability increases with the chain length of the alkyl groups and their structure in the sequence secondary, primary and aromatic. By carefully directed alcohol composition the specific requirements of different applications can selectively be adjusted. Beside excellent antiwear and extreme pressure properties ZnDTPs are also efficient antioxidants and even metal passivators. These



Zinc-dialkyldithiophosphate/
Zinc-diaryldithiophosphate



| Alkylgroup R | Reactivity (antiwear properties) | Thermal stability | Hydrolytic stability | Antioxidant properties |
|---|---|---|---|---|
| R = $C_3$<br>↓<br>R = $C_8$ | +<br>- | -<br>+ | -<br>+ | No systematic influence |
| **Structure of R** | | | | |
| Primary alkyl | +/- | + | +/- | +/- |
| Secondary alkyl | ++ | - | + | + |
| Aryl | - - | ++ | - - | - |

Primary alkyl group = — $CH_2$ — R

Secondary alkyl group = — CH — R, R'

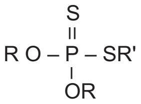
Aryl = R

**Fig. 6.9** Influence of alcohol composition and structure on the properties of ZnDTPs.

multifunctional properties makes them the widest spread cost effective additive group that is used nowadays in huge quantities in engine oils, shock absorber oils and hydraulic fluids. The influence of alcohol composition and structure on the properties of zinc dithiophosphates is shown in Fig. 6.9.

Beyond ZnDTPs also *ammonium, antimony, molybdenum and lead dialkyldithiophosphates* are known, which the latter no longer has real significance because of toxicological and ecotoxicological concerns. MoDTPs are effective antiwear additives with remarkable friction reducing properties and excellent antioxidant behavior.

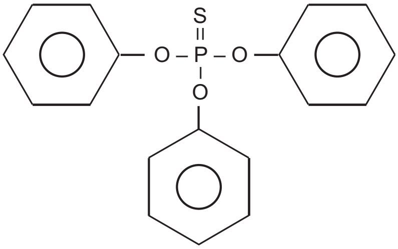
*Ashless dialkyldithiophosphoric acid-O,O,S-triesters* **6.17** distinguish by improved hydrolytic stability in comparison to metal salts of the dithiophosphoric acid. On the other side their antioxidant properties are reduced. Similar to the ZnDTPs the reactivity can be influenced by variation of the organic substituents.



$$\mathrm{RO-\overset{\overset{\displaystyle S}{\|}}{\underset{\underset{\displaystyle OR}{|}}{P}}-SR'}$$

**6.17**

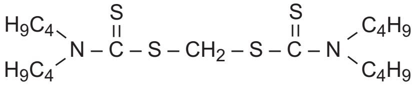
The *triphenylphosphorothionate (TPPT)* **6.18** belongs to the neutral thiophosphoric acid esters and is distinguished by its extraordinary thermal stability what makes it suited for high temperature lubrication.



**6.18**

### 6.8.2.3 Compounds Containing Sulfur and Nitrogen

*Zinc-bis(diamyldithiocarbamate)* and the ashless *methylene-bis(di-n-butyldithiocarbamate)* **6.19** are highly effective EP additives and excellent antioxidants. Beside these main species also antimony and tungsten derivatives are known. Dithiocarbamates are predominantly used in lubricating greases and to some extent in gear oil formulations.



**6.19**

*Dialkyl-2,5-dimercapto-1,3,4-thiadiazole (DMTD) derivatives* are usually known as metal passivators and sulfur scavengers, but they also exhibit excellent EP properties. The use of thiadiazoles and dithiocarbamates is limited by their relatively high price level. The chemical structure of DMTD is given in Section 6.10.

### 6.8.2.4 **Sulfur Compounds**

From the early days of lubrication until now elemental sulfur has been added directly to mineral oil (up to 1.5 %) to improve the EP properties of metal working fluids. Oil-soluble organic sulfur compounds, the so-called sulfur carriers, of the general formula $R{-}S_x{-}R$ offer improved solubility and better control over the reactivity of the sulfur.

Fundamentally there can be differentiated between inactive and active sulfur carriers. The inactive types with predominantly disulfide bridges ($x = 2$) possess relatively stable C–S bonds which will react at elevated temperatures only. The active forms with $x$ between 3 and 5 (so-called pentasulfides) are much more reactive as the sulfur of the relatively labile polysulfide bridges can easily be made available even at low temperatures. Moreover numerous sulfur carriers with specific distribution of the different polysulfide bridges ($x = 1$–$5$) are used to cover the whole field of application with its varying stress requirements. The mechanism of sulfur carriers under EP conditions can be described as to begin with physical adsorption followed by chemisorption and finally cleavage of the sulfur and its reaction with the metal surface (Fig. 6.10). Generally this reaction takes place at temperatures over 600 °C [6.32].

Active sulfur carriers are excellent EP additives that will prevent welding by a kind of controlled wear when the slideable reaction layers are removed continuously under severe loads. Because of the high reactivity with non-ferrous metals active sulfur carriers cannot be used in the machining of non-ferrous metals respectively when non-ferrous metals are incorporated in an engine or other aggregates. Inactive sulfur carriers need higher temperatures to set the sulfur free. Therefore they are much more compatible with non-ferrous metals and show to some extent antiwear properties.

Contrary to this well-known behavior active sulfur carriers exhibit surprisingly good antiwear properties in unsaturated esters or fatty oils like trimethylolpropanetrioleate or rape seed oil [6.34].

The polarity and thus the affinity to the metal surface is determined by the organic substituents. The polarity increases in the sequence sulfurized hydrocarbons, esters, alcohols and fatty acids whereas the solubility in mineral oil decreases in the same order.

*Sulfurized hydrocarbons* **6.20** are obtained by direct sulfurization of olefins (e.g. diisobutylene, terpenes) with elementary sulfur in the presence of $H_2S$, their reaction with dichlorodisulfide or oxidation of mercaptans to disulfides. The first method will lead to products with high sulfur contents up to 45 %. The other will

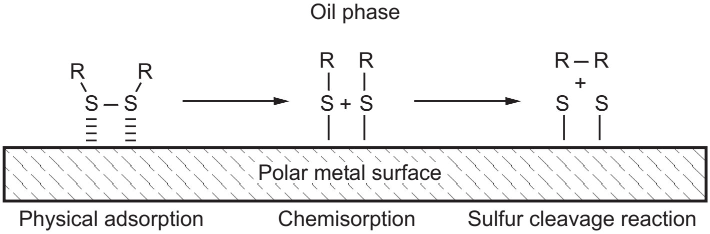
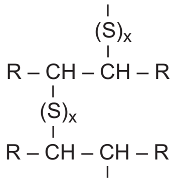


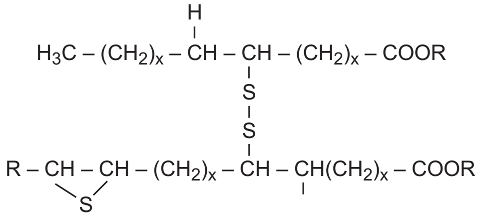
**Fig. 6.10** Mechanism of sulfur carriers under extreme pressure conditions.

form inactive sulfur carriers that are predominantly used in gear oils and mild metalworking fluids (cutting fluids). Active pentasulfides are used for severe machining processes e.g. broaching.



**6.20**

*Sulfurized fatty oils and fatty acid esters* **6.21** are produced by sulfurization of the unsaturated raw materials with sulfur (dark colored products) or with sulfur in the presence of $H_2S$ to obtain light colored products that are used predominantly in modern lubricants instead of the formerly used sperm oil products. The combination of sulfur with the friction reducing properties of the fatty raw materials leads to excellent EP additives with high load carrying capacity in the four ball testing machine. Sulfurized fatty acid methyl esters will lead to products with low viscosities that are used e.g. for deep-hole drilling.



**6.21**

### 6.8.2.5 **PEP Additives**

Overbased sulfonates, especially calcium and sodium salts, can be used as highly efficient boosters in combination with active sulfur carriers to formulate metal working fluids with extremely high load carrying behavior. These overbased products are called PEPs (passive EP).

### 6.8.2.6 **Chlorine Compounds**

The excellent AW/EP properties of chlorine compounds have been conventionally explained by their ability to coat the metal surface with a slideable metal chloride film under the influence of high pressure and in the presence of traces of moisture. Because of the formation of hydrogen chloride alkaline buffers have to be added to avoid severe corrosion. Another approach refers to the formation of high pressure stable adsorption layers due to the affinity of the additive's heteroatom (Cl) showing outstanding lubricity at low machining speed and moderate temperature but reduced efficiency with increasing temperature (speed) [6.35].

For environmental and toxicological reasons concerning the disposal of the chlorinated used fluids chlorine compounds are increasingly replaced although this additive technology has been extremely successful in metalworking fluids. Beside the corrosivity of their decomposition products, primarily hydrogen chloride, the biggest problem is the possible formation of highly toxic dioxins at the incineration of used oil especially when the incineration temperatures are not high enough.

Typical additives have been *chlorinated paraffins* with a chlorine content of 35 to 70 % and a hydrocarbon base of 10 to 20 carbon atoms as well as *chlorinated sulfur carriers.*

### 6.8.2.7 **Solid Lubricating Compounds**

Mainly finely ground powders of *graphite* and *molybdenum disulfide* respectively their dispersions are used as solid additives. They are distinguished by excellent emergency running properties when the oil supply is breaking down. Also for extreme high temperature applications solid lubricating compounds are used due to their high thermal stability. Other compounds that are used especially in lubricating greases are *polytetrafluoroethene, calcium hydroxide* and *zinc sulfide*. For more detailed information see Chapter 17.

## 6.9

## Friction Modifiers (FM)

In the case of liquid (hydrodynamic) lubrication friction can be reduced only by the use of base oils with lower friction coefficients and lower viscosity respectively high viscosity indices. In the area of low slide velocities, moderately increased loads and low viscosities at higher temperatures the liquid lubrication can easily proceed to mixed friction conditions. In this case so-called friction modifiers have to be used to prevent stick–slip oscillations and noises by reducing frictional forces. They work at temperatures where AW and EP additives are not yet reactive by forming thin monomolecular layers of physically adsorbed polar oil-soluble products or tribochemical friction reducing reaction layers that exhibit a significantly lower friction behavior compared to typical AW and EP additives [6.36, 6.37]. Therefore friction modifiers can be regarded as mild AW or EP additives working at moderate temperatures and loads in the area of beginning mixed friction.

Friction modifiers can be classified into different groups regarding their function [6.38]: mechanically working FMs (solid lubricating compounds, e.g. molybdenum disulfide, graphite, PTFE, polyamide, polyimide, fluorinated graphite), adsorption layers forming FMs (e.g. long chain carboxylic acids, fatty acid esters, ethers, alcohols, amines, amides, imides), tribochemical reaction layers forming FMs (saturated fatty acids, phosphoric and thiophosphoric acid esters, xanthogenates, sulfurized fatty acids), friction polymer-forming FMs (glycol dicarboxylic acid partial esters, dialkyl phthalic acid esters, methacrylates, unsaturated fatty acids, sulfurized olefins) and organometallic compounds (molybdenum compounds like molybdenum dithiophosphates, molybdenum dithiocarbamates, their synergistic combination with ZnDTPs [6.43], copper containing organic compounds).

A widely spread group of friction modifiers are the adsorption forming agents whose effect increases with increasing molecular mass in the order alcohol < ester < unsaturated acid < saturated acid. The main products are carboxylic acids with 12 to 18 carbon atoms, fatty alcohols and synthetic (methyl, butyl) or natural esters of fatty acids (glycerides).

Typical applications of friction modifiers are modern fuel economy oils, slide way oils, automatic transmission fluids (ATF) that contain so-called anti-squawk additives and lubricants for limited-slip axles that contain so-called anti-chatter additives.

## 6.10
## Corrosion Inhibitors

Corrosion inhibitors are used in nearly every lubricant to protect the metal surface of any machinery, metalworking tool or work piece from the attack of oxygen, moisture and aggressive products. These mostly acidic products may be formed by the thermal and oxidative decomposition of the lubricant (base oil and additives), brought in directly from the environment (acid atmosphere) or caused by the specific application (aggressive blow-by gases in internal combustion engines). The base oil itself forms a kind of protective layer on the metal surface. But in general this will not be sufficient especially when highly refined oils without natural inhibitors are used. Then highly efficient anticorrosion additives are necessary.

### 6.10.1
### Mechanism of Corrosion Inhibitors

The mechanism of anticorrosion additives is relatively simple. Anticorrosion additives are molecules with long alkyl chains and polar groups that can be adsorbed on the metal surface forming densely packed, hydrophobic layers as shown in Fig. 6.11. The adsorption mechanism can base on a physical or chemical interaction of the polar anticorrosion additive with the metal surface [6.39].

Because of this high surface activity, anticorrosion additives compete with other polar additives like antiwear and extreme pressure additives for the metal surface

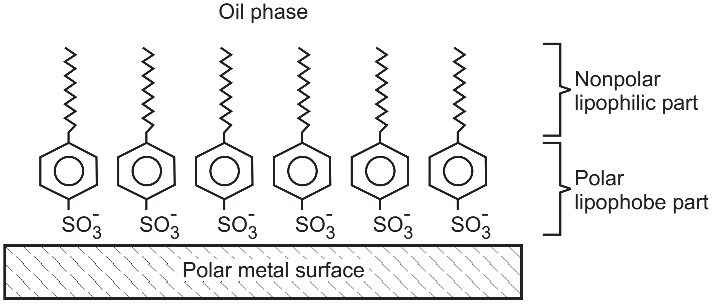


**Fig. 6.11** Function of rust inhibitors.

and can therefore reduce their efficiency. Thus a FZG damage load stage of > 12 can be reduced down to a damage load stage of 8 if highly efficient and polar anticorrosion additives are added.

Corrosion inhibitors can be divided into 2 main groups: antirust additives for the protection of ferrous metals and metal passivators for non-ferrous metals.

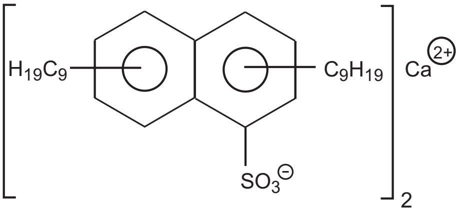
## 6.10.2
## Antirust Additives (Ferrous Metals)

### 6.10.2.1 **Sulfonates**

*Petroleum sulfonates* (mahogany sulfonates) are by-products at the production of white oils by treatment with oleum. The resulting acid tar contains long chain alkylarylsulfonic acids that can be neutralized with lyes. Sodium sulfonates with low molecular weights (below approx. 450) are typically used as low-priced emulsifiers and detergents with additional anticorrosion properties in water based metal working fluids, engine oils and rust preventatives. Sulfonates with higher molecular weights distinguish as highly efficient corrosion inhibitors especially when based on divalent cations like calcium, magnesium and barium. The importance of the barium compounds is going to decrease constantly due to toxicological and ecotoxicological concerns.

Nowadays *synthetic alkylbenzene sulfonates* are used preferably in spite of a higher price level due to their higher and more constant quality. They are reaction products of specifically designed monoalkylbenzenesulfonic acids (typically $C_{24}$ alkyl groups) and dialkylbenzenesulfonic acids (typically two $C_{12}$ alkyl groups) with alkaline and earth alkaline metal hydroxides.

A special group of synthetic sulfonates are the *dinonylnaphthenesulfonates* **6.22** of which the neutral calcium and barium salts distinguish by additional demulsifying properties and a good compatibility with EP additives.



**6.22**

Beside the neutral or only slightly basic sulfonates *overbased sulfonates* with high alkaline reserve (TBN 100 to 400 mg KOH $g^{-1}$) play an important role especially in the formulation of engine oils. There they exhibit detergent properties and can neutralize acidic oxidation products. Moreover in metal working fluids they act as so called passive EP additives (see Section 6.8) to prevent welding under high pressure conditions.

### 6.10.2.2 **Carboxylic Acid Derivatives**

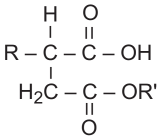
Many different long-chain carboxylic acid derivatives have been proposed as corrosion inhibitors at which the carboxylic group acts as polar part that can easily be adsorbed on the metal surface.

*Lanolin* (wool fat) and salts of the lanolin fatty acid mostly in combination with sulfonates have long been known as corrosion inhibitors in rust preventatives.

*Oxidized paraffins* with their high polarity because of the high content of hydroxy and oxo carboxylic acids are still used for that purpose.

*Zinc naphthenates* are especially used in lubricating greases, whereas lead naphthenates are not used anymore for toxicological reasons.

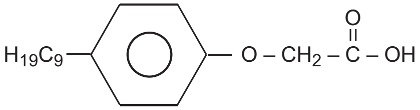
*Alkylated succinic acids*, their partial esters **6.23** and half amides are known as highly efficient, not emulsifying antirust additives even at very low treat rates of 0.01 to 0.05 %. Therefore these additives are used preferably in turbine oils and hydraulic fluids.



$$\begin{array}{l} R-\overset{H}{\overset{|}{C}}-\overset{O}{\overset{\|}{C}}-OH \\ \quad H_2\overset{|}{C}-\underset{O}{\underset{\|}{C}}-OR' \end{array}$$

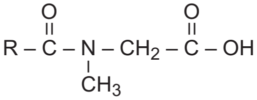
**6.23**

*4-Nonylphenoxyacetic acid* **6.24** and derivatives have a similar performance.



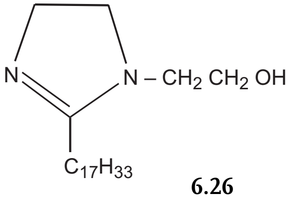
**6.24**

Another wide spread group are *amides and imides* as reaction products of saturated and unsaturated fatty acids with alkylamines and alkanolamines. The most known product of this type is *N-acylsarcosine* **6.25** that shows a strong synergistic effect with *imidazoline derivatives* **6.26**. Additionally these additives have good water-displacing properties.



$$R-\overset{O}{\overset{\|}{C}}-\underset{CH_3}{\underset{|}{N}}-CH_2-\overset{O}{\overset{\|}{C}}-OH$$

**6.25**



**6.26**

#### 6.10.2.3 Amine Neutralized Alkylphosphoric Acid Partial Esters

Some special amine salts of mono- or dialkylphosphoric acid partial esters exhibit excellent anticorrosion properties in addition to their highly efficient antiwear properties [6.40]. Because of the well-known antagonism of anticorrosion and antiwear additives this behavior makes them one of the mostly used components in ashless industrial oils.

#### 6.10.2.4 Vapor Phase Corrosion Inhibitors

Vapor phase corrosion inhibitors (VCIs) for closed systems are substances with high affinity to metal surfaces and relatively high vapor pressure to guarantee their availability on parts that are not steadily in direct contact with the corrosion inhibited lubricant. The mostly used product group for this application are amines. *Morpholine, dicyclohexylamine* and *diethanolamine* have proved to be highly efficient for that purpose. Because of toxicological concerns that refer mainly to the nitrosamine forming potential of secondary amines, these products are going to be partly substituted by tertiary amines like *diethanolmethylamine* and similar products.

Another group of oil soluble VCIs are low molecular weight *carbonic acids* (*n*-$C_8$ *to n*-$C_{10}$).

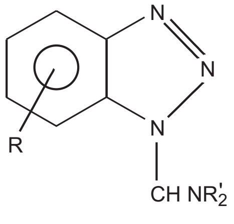
## 6.10.3
## Metal Passivators (Non-ferrous Metals)

The metal passivators can be classified into three groups: *film forming compounds, complex forming chelating agents* and *sulfur scavengers.*

The fundamental function of the film forming types consists in the building of passivating protective layers on the non-ferrous metal surface thus preventing the solubilization of metal ions that would work as pro-oxidants. The complex forming agents are able to build oil-soluble complexes with significantly reduced catalytic activity regarding the influence of non-ferrous metal ions on the oxidative aging process of lubricants. Sulfur scavengers are even able to catch corrosive sulfur by integrating sulfur into their molecular structure. Mechanism and function of these three types of metal passivator are shown in Figs 6.12–6.14.

Metal passivators in combination with antioxidants show strong synergistic effects as they prevent the formation of copper ions respectively suppress their behavior as pro-oxidants. Thus these additives are used in nearly every formulation of modern lubricants.

The mostly used metal passivators are *benzotriazole* and *tolyltriazole* as well as their alkylated liquid derivatives **6.27** with improved solubility. Typical treat rates of these film forming passivators are between 0.005 and 0.03 %.



**6.27**

Furthermore *2-mercaptobenzothiazole* (reduced importance) and especially *2,5-dimercapto-1,3,4-thiadiazole* derivatives are used as highly efficient film forming passivators. The later can also act as sulfur scavenger by building in sulfur into the alkyl–sulfur–thiadiazol bonds (see Fig. 6.14).

*N,N′-disalicylidenealkylendiamines* (where ‘alkylen’ is ethylene or propylene) belong to the group of chelating agents. Beside their EP and antiwear properties *zinc dialkyldithiophosphates* and *dialkyldithiocarbamates* have some metal-passivating properties (see Section 6.8).

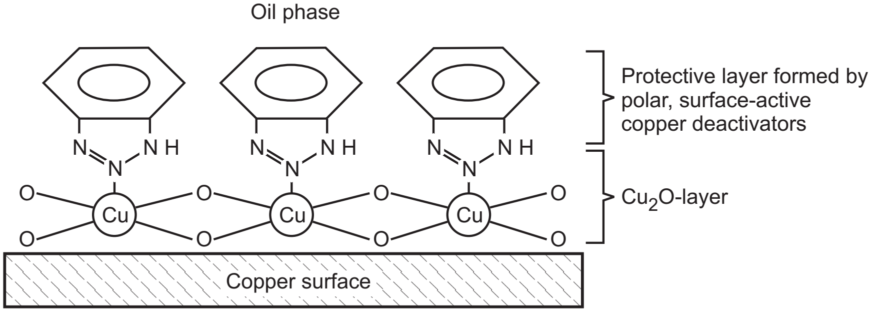


**Fig. 6.12** Function of benzotriazole as film forming agent.

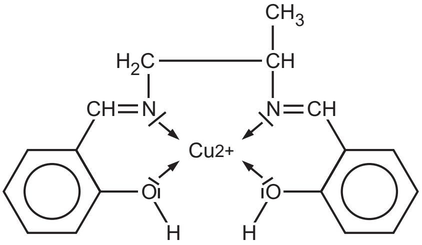


**Fig. 6.13** Function of *N*,*N′*-disalicylidene-propylenediamine as chelating agent.

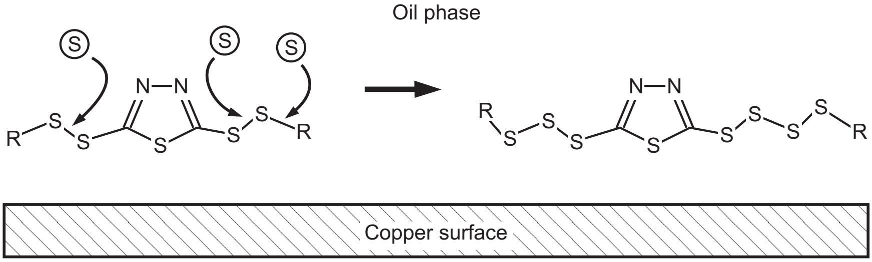


**Fig. 6.14** Function of dialkyl-2,5-dimercapto-1,3,4-thiadiazole as sulfur scavenger.

# 7
# Lubricants in the Environment

*Rolf Luther*

## 7.1
## Definition of 'Environmentally Friendly Lubricants'

Ecological aspects are gaining importance in our society. Bearing in mind that our environment is being increasingly contaminated with all kinds of pollutants, any reduction is welcome. From an environmental point of view and compared to a number of other chemical products, lubricants are not particularly problematic. A large proportion of the lubricants pollute the environment either during or after use. This can be technically desired (total-loss lubrication) or a result of mishaps such as leaks, emissions, spillages or other problems.

Lubricants and functional fluids are omnipresent due to their widespread use and they thus pollute the environment in small, widely-spread amounts and rarely in large, localized quantities.

The terminology used in connection with 'environmental compatibility' has to be split between the subjective and the objective:

Subjective criteria (non-measurable):

- Environmentally friendly
- Environmentally compatible

Objective criteria (measurable or provable), for example:

- Biodegradability
- Water solubility, water pollution
- Ecological toxicity and physiological safety
- Performance, approvals, oil change intervals
- Efficiency improvements, lower energy consumption
- Emission reduction in use
- Compatibility with conventional lubricants and materials
- The use of renewable raw materials
- Environmental awards.

Normally biodegradability of at least 60 % according to OECD 301 or 80 % according to CEC L-33-A-93 is considered as main objective criteria for 'Bio-Lubricants'. But the other criteria will get more and more importance in the near future.

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## 7.2
## Current Situation

Worldwide mineral oil and its derivatives are dominating the lubricants market. But this triumphant progress is limited to the last century. In former days of mankind the friction and wear decreasing properties of natural oils and fat were well known and used absolutely in many different ways. In this respect the development in the last 25 years to formulate biodegradable lubricants based on natural oils followed the tradition – even if the market share today only amount to few percent.

The statistical data and the main legislation in this chapter apply to experiences in Germany, which has – beside some countries in Western Europe (Holland, Austria, Switzerland) and Scandinavia – the highest amount of biodegradable lubricants worldwide.

### 7.2.1
### Statistical Data

Every year, about 40–50 % of the approximately 5 million tonnes of used lubricants in Europe end up polluting the environment. This interface with the environment and the resulting pollution of the atmosphere, the ground or water is either technically desired (total-loss lubrication) or follows leaks or other problems.

The environmental damage caused by mineral oil-based lubricants is largely caused by the approximately 40 % of lubricants which are not properly disposed of. This figures included total-loss applications, the residual oil in about 90 million oil cans and 20 million oil filters, spillages during topping-up, leaks, drips from separated oil-line and hydraulic couplings, accident losses and all manner of emission losses (CONCAWE Report no. 5/96, see Fig. 7.1). CONCAWE is the oil companies' European organisation for environment, health and safety.

Even though mineral oil products can be relatively rapidly biodegraded by the microorganisms present in nature, these natural degrading systems are overwhelmed by the volume of the losses. In recent years, rapidly biodegradable total-loss lubricants based on esters, polyglycols and natural oils have significantly eased lubricant-based pollution.

In the past, mineral oil-based products were used almost exclusively. Leakages and other causes allowed a part of these lubricants to pollute the environment. Soil and ground water is severely contaminated by lubricants and particularly some of the ingredients in such products. The problem has spurred society and politics into action. This has been reflected by diverse legislation and recommendations as well as the issuing of environmental awards to products which cause less damage. The aim of this all is to avoid or, at least, reduce environmental pollution. The use of environmentally friendly materials and substances will play an increasingly important part in the scope and procedures of the EU Eco Audit. The most effective measure to prevent environmental pollution is and remains the avoidance of physical containment by lubricants. The technical prerequisites for this already exist and should be fully utilized.

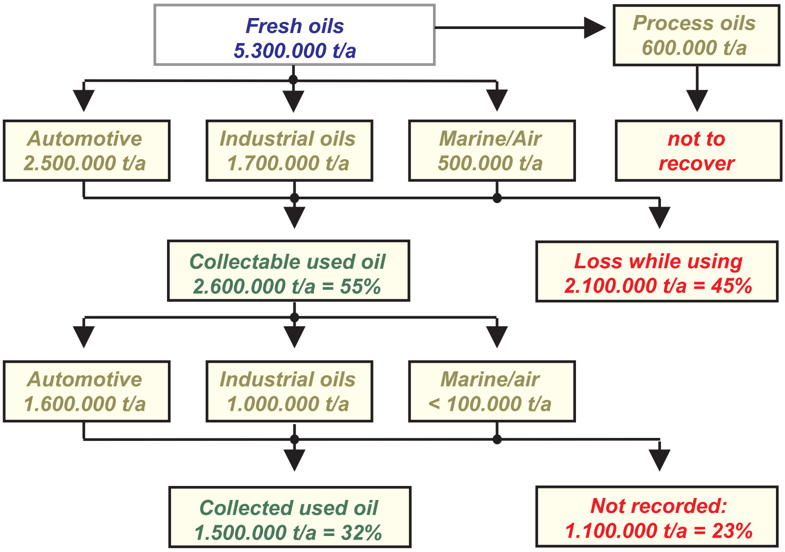


**Fig. 7.1** Lubricants, and residual amounts in Europe

Technically, and disregarding development and overall costs, it is accepted that more than 90 % of all lubricants could be made rapidly biodegradable. However, this change will only take place if the ecological benefits of using harvestable raw materials is made clear and best of all, quantified in monetary terms.

### 7.2.2
### Economic Consequences and Substitution Potential

Now that corresponding laws and guidelines are in place, environmental legislation has economic repercussions. In addition to national laws, European guidelines are gaining relevance in the different European countries. Accordingly, new production facilities must comply with EU machinery guidelines which came into force on January 1, 1995. For example, this supercedes previously applicable German Legislation concerning safety and the protection of human health and, along with thorough documentation, includes mandatory measures to reduce operator stress and fatigue. Compared to older plant, this provides the operators of new machinery with greater safety. The implementation of these regulations in practice requires the use of environmentally harmless lubricants.

In Germany the selection of rapidly biodegradable lubricants, especially those classified as ‘not water pollutant’ reduce the expense of oil spillages or disposal as well as the cost of complying with health and safety regulations to a minimum. Compared to mineral oil-based products, the selection of low-evaporation ester and vegetable oil-based lubricants can allow lower viscosities to be used. This enables faster machining and cuts drag-out losses. This, in turn, increases economy, increases productivity and generates product optimization.

Active environmental protection is steadily gaining importance. The trend is towards clean and environmentally friendly factories in which people like to work and which combine the requirements of advanced industrial technologies with environmental protection considerations. This is a major objective for a number of companies in a variety of industries. In the field of metalworking, for example, ecologically friendly cutting fluids can significantly cut the costs of:

- Disposal (see Chapter 14 'Water-miscible Cutting Fluids')
- Complying with water laws
- Maintenance (see Chapter 14)
- Downtime
- Washing operations between machining steps
- Water-reconditioning in splitting plants.

Furthermore, environmentally harmless products are generally non-toxic and skin compatibility is mostly much better than mineral oil products. This creates ecological and economic points of view which promote the use of environmentally friendly lubricants.

The vast majority of rapidly biodegradable lubricants currently on the market are based on saturated or unsaturated ester oils. The acids required for esterification are mostly from vegetable oils. Compared to equivalent mineral oils, ester oils are significantly more expensive. This is a major barrier to the wider acceptance of such oils. But the overall cost of a lubricant results from the interaction between the following characteristics (in alphabetical order):

- Aging and temperature stability
- Disposal costs
- Machine compatibility (multifunctional oil)
- Machine investments
- Maintenance measures
- Oil change intervals
- Operating costs
- Reconditioning possibilities
- Reduction in storage costs
- Reduction in emission reduction measures
- Reduction in the cost of preventative health and safety measures
- Risk minimization and lower clean-up costs after spillages.
- Simpler plant license procurement.

Completely new mix of economic aspects can result from every new application. In particular, the use of fully saturated synthetic esters offers a number of opportunities. Pure vegetable oil formulations are now only used for lower demands, for example total-loss chain saw or mold release oils and some types of greases.

Users are greatly interested in the potential life of the new Generation of environmentally friendly lubricants. Rapeseed oil-based hydraulic fluids are normally used in lightly-stressed systems and changed at the same intervals as mineral oils. Increasing use is being made of ecologically friendly fluids based on saturated and unsaturated synthetic esters. These satisfy the technical demands which are defined as higher performance, longer life, low oil volume and more rapid oil circulation times. At the same time, the disposal of ester-based lubricants is generally unproblematic.

### 7.2.3
### Agriculture, Economy, and Politics

Analysis of the economics of renewable raw materials often includes high EU agricultural subsidies, of rapeseed oil, for example, as a critical factor. This is relatively insignificant for the manufacture of bio-lubricants. EU vegetable oil prices depend on world market prices. The world market price of vegetable oils is largely affected by the price of soybeans. Soya accounts for a large fraction of total vegetable oils (Table 7.1). Nearly half of the world's soybean production is in the USA; the volume of soybean oils produced in South America (Brazil, Argentina) is increasing. Vegetable oil prices, and thus also oil acid or oil ester prices, are, therefore, heavily dependent on the size of the US soya harvest.

**Tab. 7.1** Production of important oil seeds.

| *Oil seed* | *World production in 2005 (major producers)* | *Largest producer* | *Production of largest producer in 2005* |
|---|---|---|---|
| Soy | 190,857,900 t | USA | 82,820,048 t |
| Oil palm | 168,378,200 t | Malaysia | 75,650,000 t |
| Coconut | 51,392,800 t | Indonesia | 16,300,000 t |
| Rapeseed | 41,326,800 t | China | 11,300,010 t |
| Sunflower | 26,168,300 t | Russia | 6,280,000 t |

Source: FAO Statistical Databases, 2005

Increasing correlation of the prices of vegetable oils with the price of crude oil must be recognized in some respects, because the use of vegetable oils in combustion engines, both for energy production and by traffic, is increasing. One driving force is the European Directive 2003/30/EC of 8 May 2003 on promotion of the use of bio-fuels or other renewable fuels for transport.

Minimum proportions of bio-fuels and other renewable fuels have been laid down as strategic commercial targets. A reference value for these targets was 2 % by the end of 2005 and 5.75 % by end of 2010, calculated on the basis of the energy content of all petrol and diesel for transport purposes. If these European targets are to be achieved, domestically grown oil crops will not be sufficient to support the fuel production (mainly bio-diesel and ethanol) necessary. Global markets will be able to provide sufficient quantities of oil seed and oils, but with a consequent effect on the prices of vegetable oils.

In Germany, currently the leading country in the use of bio-diesel (the methyl ester of rapeseed oil), approximately 1.5 million tonnes of bio-diesel were produced in 2005.

Technical crops can replace the reduction in the over-production of food and animal feed products in Europe. Rapeseed oil and derived oleochemical products are especially key products for rapidly biodegradable lubricants. In Europe, rapeseed and sunflower oils as lubricant base oils have been in the forefront, for several reasons:

- they are abundantly available, growing well in central European climates;
- their thermal oxidation stability is acceptable for some applications;
- their flowing properties are better than those of other vegetable oils; and
- their oleochemical derivatives combine performance with good environmental properties.

When subjected to severe thermal exposure, especially, pure vegetable oils have application boundaries comparable with similar mineral oil products. The main disadvantage of these oils is that their oxidation, hydrolytic, and thermal stability is not adequate to enable their use in circulating systems.

Apart from pure vegetable oils, oleochemical esters are being used increasingly, because their stability to thermal oxidation compared with that of rapeseed oil opens up a wider field of applications. These products are, however, substantially more expensive, a factor which, in turn, limits their use.

Environmentally harmless lubricants based on renewable material have good potential, even if prices of the raw materials are also rising. In addition to direct environmental advantages, synthetic esters have technical advantages for lubricants in comparison with conventional base oils.

In 2005, approximately 20,000 hectares of rapeseed were cultivated in Germany solely for application in lubricants. This corresponds to approximately 20,000 tonnes rapeseed oil per year, used both as bio-lubricants and as (lubricity) components of conventional lubricants. It can be assumed that approximately 20,000 to 25,000 tonnes of oleochemical esters are also used in lubricants every year. In Germany the percentage of biodegradable lubricants amounts to 4–5 % of all lubricants, approximately 47,000 t (2005).

### 7.2.4
### Political Initiatives

In Germany, currently the most important market for biodegradable lubricants, several political initiatives were started in the last decade of these, some were included in two comprehensive reports submitted to the German parliament.

- Resolution dated June 16, 1994: Measures to promote the use of rapidly biodegradable lubricants and hydraulic fluids
- Resolution dated November 28, 1995: News from the Federal Ministry for Food, Agriculture, and Forestry: “Report on the use of rapidly biodegradable lubricants and hydraulic fluids and measures taken by the Government” (New Edition 1999)

As a consequence, the German Ministry of Agriculture launched the Market Introduction Program for bio-based fuels and lubricants in the year 2000, the focal point of which was lubricants. Using targeted promotional activity, the Ministry hopes that special, vegetable oil-based lubricants will gain a stronger footing in the German market.

Technically, rapidly biodegradable alternatives based on harvestable raw materials are available as replacements for most mineral oil lubricants. Bio-based lubricants are also available for particularly demanding applications, for example engine lubrication. Nevertheless, bio-based lubricants have failed to achieve market penetration reflecting their technical performance and ecological potential, mainly because of their high price.

To improve this situation, the Market Introduction Program for bio-based lubricants was started in Germany. This program promotes changing from mineral oil-based lubricants to rapidly biodegradable products made from renewable raw materials. The principal condition is application is in the ecologically-sensitive areas of agriculture, forestry, and water supply. This thus exploits the risk reduction potential of bio-based lubricants without the need for special environmental legislation.

After five years of experience with this Market Introduction Program it can be stated that the approach was a success under difficult conditions. The program is ongoing.

Research and development on ecologically harmless lubricants is sponsored by European and national projects. The government offers research sponsorship for projects aimed at improving and demonstrating the performance of biodegradable lubricants, for example producing lubricants which are thermally and hydrolytically stable, and stable to oxidation, without significantly altering their biodegradability.

At a European level the "European Renewable Resources and Materials Association" (ERRMA), a representative body promoting the use of renewable raw materials in technical applications, focuses its efforts on bio-lubricants and bio-plastics, because of the forecasts for these product groups.

## 7.3
## Tests to Evaluate Biotic Potential

The possibility of measuring the environmental impact of lubricants was often discussed. This issue is to differentiate in tests regarding the biodegradability in the natural environment (water, soil) and in measuring the ecotoxicological potential for damage the health of creature.

### 7.3.1
### Biodegradation

The limit for rapid biodegradation is not exactly defined, but the most legislation and recommendations are working with these numbers for not water soluble lubricants:
Degradation > 80 % accord. CEC L-33-A-93 or
Degradation > 60 % accord. OECD 301B.

The most important tests are shown in the next Table 7.2 – beyond these tests are many ISO, ASTM and national procedures with more or less similar methods and restrictions.

The CEC L-33-A-93 test becomes more and more obsolete, no important regulation refers to this test any longer. It is normally substituted by an OECD 301 procedure.

**Tab. 7.2** Most important methods for testing the biodegradation of lubricants.

| *Test* | *Short description – relation to other tests* | *Limit for 'rapid degradation'.* |
|---|---|---|
| OECD 301B | 'Modified Sturm test', aerobic degradation, ultimate biodegradation | ≥ 60 % in 28 days |
| OECD 301C | 'Modified MITI Test', aerobic degradation, measurement of $O_2$ consumption, for volatile components | ≥ 60 % in 28 days |
| OECD 301D | 'Closed Bottle Test', aerobic degradation, preferred for water soluble products, but possible for not water solubles substances | ≥ 60 % in 28 days |
| OECD 301F | 'Manometric Respirometry Test'; for water soluble and not water soluble substances | ≥ 60 % in 28 days |
| OECD 302B | 'Zahn–Wellens/EMPA Test', assesses inherent biodegradability for water soluble substances | |
| CEC L-33-A-93 | 'Inherent degradation', aerobic degradation, water soluble part of degradation not considered, only for bad water soluble substances. Because of primarily degradation the values are higher than OECD 301 results – up to 20 %. | ≥ 80 % in 21 days |
| BODIS test, ISO 10708 | Two phases 'Closed Bottle' test, similar OECD 301D | |

### 7.3.2
### Ecotoxicity

To develop environmentally acceptable lubricants, toxicological criteria must be considered. The aim is to protect life in various areas, especially in water (aquatic area) and in the non-aquatic area (terrestrial area). The following, especially in the eco-labelling systems used test procedures are of importance:

- Bacteria toxicity, according to DIN 38 412-8; this determines cell-multiplication inhibiting ($EC_{10}$ and $EC_{50}$ values). The Pseudomonas type used for this test is found in waste water and soil.
- The bacteria test, according to OECD 209 or ISO 8192, determines acute toxicity through the inhibiting of oxygen consumption; results of this test are $EC_{50}$ values.
- The algae toxicity test according to OECD 201 or DIN 38 412-9 is yet another test for aquatic systems (measurement of chlorophyll fluorescence and determination of $EC_{10}$ and $EC_{50}$ values).
- One of the most important test procedures in German legislation concerning the aquatic area is a test on small living organisms called the 'Daphnia test' (daphnia magna STRAUS, water flea, small crustacean) according to DIN 38 412-11 or OECD guideline 202.

- In the aquatic area, fish toxicity, according to OECD 203 or DIN 38 412-15, performed on the Goldorfe (*Leuciscus idus*), is of importance. Test results are the $LC_0$, $LC_{50}$ and $LC_{100}$ values.
- Fish toxicity, according to OECD guideline 204, is incorporated in the German eco label 'Blue Angel'. Possible pollution of the non-aquatic terrestrial area, i.e. soil and plants, is evaluated with the plant growth test according to OECD guideline 208 (i.e. wheat, cress and rape seeds).

Naturally, toxicity testing for environmental protection purposes must also include mammal and human toxicity and all the more occupational safety aspects.

### 7.3.3
### Emission Thresholds

Every evaporating lubricant pollutes the atmosphere with its emission.

The evaporation loss of rapidly biodegradable lubricants is generally lower than that of conventional oils. This feature of biodegradable lubricants can help to meet such emission limits as known from cutting oils or engine oils.

### 7.3.4
### Water Pollution

Because the national legislation in Germany is very ambitious, the principles of environmental relevant laws will be shown exemplary for this European country.

#### 7.3.4.1 The German Water Hazardous Classes

The German Water Law serves to protect waters and, indirectly, the ground. A law specifically aimed at protecting the ground, a German Ground Law, is currently being drafted.

Among other points, the Water Law defines the water pollution potential of substances and mixtures with Water Hazardous Categories (WHCs).

The measurements and calculations should be performed by a certified laboratory. In Germany, the figures thus gained are submitted to a 'Commission for the Evaluation of Water Polluting Substances'. A substances evaluation and classification in the catalog of water-polluting substances is then performed by this Government commission. In 1996, new provisions concerning water-polluting substances were implemented and the substances classified in one of four 'Water Pollution Categories' or 'Water Hazardous Classes' (WHC 1–4). In addition, these provisions contained a legally-binding directive on how the Water Pollution Category of mixtures should be calculated.

These 1996 provisions were revised on June 1, 1999. The most important change was the linking of Water Pollution Categories to the R-phrases in European Hazardous Substances Legislation with the ultimate objective of harmonizing the evaluation of substances. The principal weakness of the previous system, the poor transferability to international regulations, has thus been eliminated. Water Pollution Categories neither evaluate the correct use of substances nor what is finally disposed of into sewers or waterways.

The procedure is as follows:

- Classification of the points to each R-phrases (in correlation of the meaning for the safety aim and the previous classification procedure)
- Definition of basis data (acute oral toxicity, one aqueous toxicity, biological degradation, bio accumulation); distribution of default-values, in case these characteristics have not been determined
- Derivation of a WHC from the total amount of points

In the latest version of the WHC system the former WHC 0 no longer exists; it was replaced by the new category 'not water hazardous' ('nwh') – on the one hand with more restrictions but on the other hand with more advantages in use. The WHC's are defined as

**WHC 1** = Slightly water polluting (e.g. mineral oils without additives or simple oils)
**WHC 2** = Water polluting (e.g. mineral oils with additives)
**WHC 3** = Highly water polluting (e.g. water-miscible cutting fluids, lubricants with emulsifiers, special lubricants).

The determination of WHC of formulated lubricants, that means blends, is described in the 'mixture regulation' (Table 7.3). For WHC 1 to 3 both the classification of the overall formulation and the classification according the mixture regulation is possible. For 'not water hazardous' blends only the mixture regulation is provided.

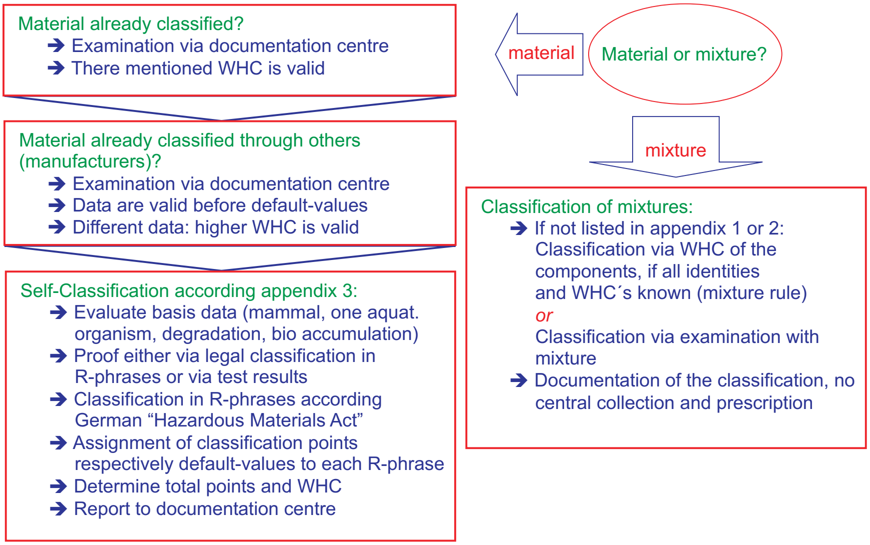
**Tab. 7.3** Mixture regulation.

| ***Containing materials*** | | | | |
|---|---|---|---|---|
| WHC 3 | $\geq 3\,\%$ | $0.2$–$3\,\%$ | $< 0.2\,\%$, as supplement | $< 0.2\,\%$, no supplement allowed |
| WHC 2 | – | $\geq 5\,\%$ | 0.2 bis 5 % | $< 0.2\,\%$ |
| WHC 1 | | | $\geq 3\,\%$ | $< 3\,\%$ |
| Not water hazardous | | | | |
| R 45 phrase | $\geq 0.1\,\%$ | $\geq 0.1\,\%$, but WHC 2 | $< 0.1\,\%$, as supplement | $< 0.1\,\%$, no supplement allowed |
| ⇒ Result: | WHC 3 | WHC 2 | WHC 1 | Not water hazardous |

The principle means of generating the WHC is shown in Fig. 7.2.

### 7.3.4.2 German Regulations for Using Water-endangering Lubricants (VAwS)

Water hazard classes have a major influence on the storage and handling of these substances. The German regulation for using water-endangering lubricants (VAwS) considerably will take greater care with highly water polluting substances than for non-water-hazardous substances (Table 7.4). Hazard potential based on Water Hazard Class and quantity is divided into 3 groups when manufacturing, processing, storing or application of water polluting substances is planned. The highest level is achieved when only 1 m$^3$ of water-miscible cutting fluid (WHC 3) is stored.



Material already classified?
- ➔ Examination via documentation centre
- ➔ There mentioned WHC is valid

Material already classified through others (manufacturers)?
- ➔ Examination via documentation centre
- ➔ Data are valid before default-values
- ➔ Different data: higher WHC is valid

Self-Classification according appendix 3:
- ➔ Evaluate basis data (mammal, one aquat. organism, degradation, bio accumulation)
- ➔ Proof either via legal classification in R-phrases or via test results
- ➔ Classification in R-phrases according German "Hazardous Materials Act"
- ➔ Assignment of classification points respectively default-values to each R-phrase
- ➔ Determine total points and WHC
- ➔ Report to documentation centre

Classification of mixtures:
- ➔ If not listed in appendix 1 or 2: Classification via WHC of the components, if all identities and WHC´s known (mixture rule) *or* Classification via examination with mixture
- ➔ Documentation of the classification, no central collection and prescription

**Fig. 7.2** Scheme for determination the WHC

On the other hand, a rapidly biodegradable lubricant with a nwh-certificate must no longer fulfil the requirements of WHC 1 to WHC 3.

**Tab. 7.4** German regulations for use of water-endangering lubricants.

| ***Volume of the stock plant ($m^3$)*** | ***Non-water hazardous*** | ***WHC 1*** | ***WHC 2*** | ***WHC 3*** |
|---|---|---|---|---|
| ≤ 1 | Non-water hazardous materials and preparations are not subject to the VAwS | F0 + R0 + I0 | F0 + R0 + I0 | F1 + R2 + I0 |
| > 1 to ≤ 10 | | F1 + R0 + I1 | F1+ R1 + I1 | F2 + R2 + I0/<br>F1 + R3 + I0 |
| > 10 to ≤ 100 | | F1 + R1 + I1 | F1 + R1 + I2/<br>F2 + R1 + I1 | F2 + R2 + I0/<br>F1 + R3 + I0 |
| > 100 | | F1 + R1 + I2/<br>F2 + R1 + I1 | F2 + R2 + I0/<br>F1 + R3 + I0 | F2 + R2 + I0/<br>F1 + R3 + I0 |

Ground areas: F0 – no special requirements, internal company requirements are valid
F1 – impervious areas
F2 – like F1, but with proof
Storage capability: R0 – no special requirements, internal company requirements are valid
R1 – storage capability is sufficient up to taking effect of the security precautions
R2 – storage capability equal the volume of the liquid
R3 – double wall with leak detector
Infra structure: I0 – no special requirements, internal company requirements are valid
I1 – independent signalling unit in connection with permanent occupied production sites
I2 – Alarm and measurement plan

Therefore it is one objective for ester based products in Germany, especially in metalworking, to reach the classification 'not water hazardous' – the better classification creates an 'added value' for the higher cost of those lubricants.

## 7.4
## Environmental Legislation 1: Registration, Evaluation and Authorization of Chemicals (REACh)

On 13 February 2001 the European Commission adopted the White Paper "Strategy for a Future Chemicals Policy". This proposes a wide-ranging fundamental overhaul of EU chemical control legislation, i.e. the Dangerous Substances Directive (DSD), including the notification scheme for new substances, the Dangerous Preparations Directive (DPD), the Existing Substances Regulation, and the Marketing and Use Directive. In essence, legislation for new and existing substances would be merged. The current EU chemical control measures result in too great disparity between new and existing substances, with the high cost of notification of new substance stifling innovation. Furthermore, existing substances account for >99% by volume of chemicals in commerce, but are poorly assessed and controlled in comparison.

The European Commission published the first draft of legislation intended to implement the White Paper on 7 May 2003. This is the Registration, Evaluation, and Authorization for Chemicals (REACh). The next step was an eight-week Internet consultation on the "workability" of this legislation; approximately 6,400 contributions were received. A revised version emerged in September 2003, jointly from the Enterprise and Environment Directorates of the European Commission. After consultation within the Commission, the final proposed Regulation was presented on October 29, 2003. These formal legislative proposals had to be discussed by the Council of Ministers and the European Parliament, under the Co-decision Procedure. The European Parliament approved a compromise text on 17 November 2005 and the Competitiveness Council reached political agreement on December 13, 2005. This paves the way for REACh to become law in the summer of 2007, with the European Chemicals Agency (ECA) as administration body, and to become fully operational a year later in Helsinki, Finland. Meanwhile, work on the REACh Implementation Projects (RIPs) continues, with the EU Joint Research Center (JRC) getting ready to take a greater role in preparing the technical guidance documents. The new and existing substances regimes will continue until REACh starts to become operational.

REACh will place a duty on companies that produce, import, and use chemicals to assess the risks arising from their use, and to take the necessary measures to manage any risks identified. Hence the burden of proof that commercial chemicals are safe will be transferred from the regulators to industry. Testing results will have to be shared, to reduce animal testing, and registration of information on the properties, uses, and safe use of chemical substances will be an integral part of the new system. These registration requirements will vary, depending on the volume of a substance produced and on the likelihood of exposure of humans or the environment. A phase-in system lasting up to 11 years is planned. High-tonnage substances would require the most data, and would have to be registered first; lower-tonnage substances would require less data and be registered later.

Tighter controls will be introduced for the chemicals of highest concern; thus carcinogens, mutagens, and reproductive toxicants (CMRs), persistent, bioaccumulative, and toxic substances (PBTs), and very persistent and very bioaccumulative substances (vPvBs) will require authorization and hence will be registered early. Other substances of concern, for example endocrine disrupters, will be included on a case-by-case basis within the authorization system. Substances subject to authorization will have to be approved for a specific use, with decisions based on a risk assessment and consideration of socio-economic factors.

The first reading of the proposed REACh Regulation by the European Parliament occurred on November 17, 2005, and considered over 1,000 tabled amendments. A compromise text was approved. Pre-registration of all existing "phase-in" substances will be in a single phase 18 months after the regulation comes into force, but with a further six months allowed for SMEs and downstream users. Earlier registration will be required for PBT and vPvB substances. Full safety data will only be required for registration of substances at 1 to 10 tonnes per annum if they are suspected of being carcinogenic, mutagenic, or toxic for reproduction (CMR) or are assessed as classified as dangerous to human health or the environment and are for diffusive use, particularly if used by consumers. Reproduction toxicology data will normally be needed at 100 tonnes per annum and above instead of at 10 tonnes per annum. Waiving of studies on the grounds of low exposure is introduced for specific tests at all tonnages. The "one substance one registration" (OSOR) requirement was agreed, but with the possibility of opting out if the cost would be disproportionate, where there would be a breach of confidentiality, or if there is disagreement about the hazardous properties, but sharing of animal testing results would still be mandatory, as also would sharing of results from non-animal testing if requested by one potential registrant. Authorization of very-high-concern substances will be subject to periodic review.

The European Commission considers that work on existing substances under the Existing Substances Regulation will be completed. New substance notifications made in the run up to REACh will be handled as their equivalents under REACh. Similarly, classification and labeling work will continue, but with a shift toward using the REACh documentation from 2006, and the 31st Adaptation to Technical Progress (ATP) of the Dangerous Substances Directive is planned for end of 2006.

The European Commission plans to bring into force a new regulation implementing the Global Harmonized Classification Scheme (GHS) at the same time as REACh becomes operational; this would be in accord with the stated political intention to implement GHS by 2008. A draft regulation to implement the GHS is in preparation.

### 7.4.1
### Registration

All substances manufactured in or imported into the EU at ≥ 1 tonne per annum will be registered with the ECA, who will assign a registration number and perform a completeness check using an automated process, normally within three weeks. The registrations are forwarded to Member State competent authorities and entered on to a database of registered substances. Under the compromise text, at least 5 % of registration dossiers are to be checked in more detail by the ECA.

Registration will be needed before new substances are manufactured or imported. Manufacture or import of new substances can begin three weeks after the registration date, unless the ECA informs the registrant that the registration is incomplete.

All "phase-in" substances, i.e. those listed in the European Inventory of Existing Chemical Substances (EINECS) or manufactured in the EU 15 years before the regulation comes into force, must be registered in a prioritized review. In 1981 ca 100,000 existing chemical substances were known. The deadlines for registration of such "phase-in" substances are based on the date the new regulation comes into force (Table 7.6). The compromise text requires earlier registration of substances classified as very toxic to aquatic organisms that may cause adverse effects in the aquatic environment (i.e. labeled with R50/53). A new manufacturer or importer of a phase-in substance can participate in the review and enter the EU market under the compromise text. It is important to note that new substances already notified under the current DSD scheme are regarded as registered under the new REACh system, but further information is required under REACh if the manufacture or import quantities are triggered. It is expected that ca 30,000 substances will be registered, with at least 10,000 requiring new testing.

### 7.4.2
### Evaluation

It is estimated that approximately 80% of registered substances will not proceed to the next stage of evaluation. Registration information for the ca 5,000 substances exceeding a manufacture or import volume of 100 tonnes per annum will, however, have to be evaluated. The registration dossier for substances at 100 tonnes per annum includes a proposal for so-called "Annex VII testing", and some substances at levels below this tonnage will also have additional proposed testing. For new substances, the ECA will evaluate the proposal and produce a draft decision within 180 days. Evaluation of testing proposals for phase-in substances must be completed as shown in Table 7.6, with priority being given to substances with CMR, PBT, vPvB, or sensitizing properties. The registrant is set a deadline to submit the additional studies for examination by the ECA. At 1,000 tonnes per annum an equivalent procedure is followed for "Annex VIII" testing.

There is also a procedure for evaluating substances. The ECA select substances for evaluation on the basis of a risk-based approach, taking into account the hazardous properties, including of analogous substances, exposure, and tonnage, including aggregated tonnage from all registrants. Such substances are evaluated by Rapporteur Member States, who select substances from the EU rolling action plan. The outcome may be EU-harmonized classification, restrictions, or adding to the list for authorization.

**Table 7.6** Deadlines for registration of "phase-in" substances.

- Register new substances at ≥ 1 t.p.a. before manufacture or import
- Pre-registration of all phase-in substances within a 6-month period, beginning 12 months after the regulation comes into force
- Registration for phase-in substances (from the date the regulation is in force):

| | |
|---|---|
| CMRs (>1 t.p.a.) | 3 years |
| >100 t.p.a. (R50/53) | 3 years |
| >1,000 t.p.a. | 3 years |
| >100 t.p.a. | 6 years |
| >1 t.p.a. | 11 years |

- Draft decisions for phase-in substances for further testing:

| | |
|---|---|
| CMRs (>1 t.p.a.) | 5 years |
| >100 t.p.a. (R50/53) | 5 years |
| >1,000 t.p.a. | 5 years |
| >100 t.p.a. | 9 years |
| >1 t.p.a. | 15 years |

### 7.4.2
### Authorization

Substances of very high concern will have to be authorized before being used for specific purposes demonstrated to be of negligible risk. It is estimated that ca 1,400 substances will be subject to authorization. There will be a published list of these very-high-concern substances that are candidates for authorization. Very high concern substances are substances classified as category 1 or 2 carcinogens, mutagens or toxic for reproduction (CMRs), persistent, bioaccumulative, and toxic (PBT), or very persistent and very bioaccumulative (vPvB). The PBT and vPvB criteria given in Annex XII of the Regulation are summarized in Table 7.7. Endocrine disruptors not covered by these criteria will be added to the list of very high concern substances on an ad hoc basis.

**Table 7.7** Criteria for Identification of PBT and vPvB

- BCF is bioconcentration factor, NOEC is no-observed effect concentration, and CMR is a substance classified as carcinogenic, mutagenic, or toxic for reproduction
- For marine environmental risk assessment, half-life data in freshwater sediment can be superseded by data obtained under marine conditions
- Substances are classified when they fulfill the criteria for all three inherent properties for P, B, and T. There is some flexibility, however, for instance when one criterion is marginally not fulfilled but the others are exceeded substantially.

| Criterion | PBT criteria | vPvB criteria |
|---|---|---|
| **P** | Half-life >60 days in marine water, or >40 days in fresh or estuarine water, or >180 days in marine sediment, or >120 days in fresh or estuarine water sediment, or >120 days in soil | Half-life >60 days in marine, fresh, or estuarine water, or >180 days in marine, fresh, or estuarine water sediment, or >180 days in soil |
| **B** | BCF >2,000 in fresh or marine aquatic species | BCF >5,000 |
| **T** | Chronic NOEC <0.01 mg $L^{-1}$ for fresh or marine water organisms, Category 1 or 2 carcinogen or mutagenic or Category 1, 2, or 3 toxic for reproduction or chronically toxic (i.e. classified as T or Xn with R48) | Not applicable |

The first step is to identify existing substances, or particular uses of substances, requiring authorization, and to decide on a deadline for authorization and any uses exempted from authorization. When additional very-high-concern substances are identified, largely from testing for registration and evaluation, they will be fed into the authorization system.

Particular uses of very-high-concern substances will be authorized in the second step on the basis of a risk assessment covering all stages of the life-cycle for that particular use submitted by industry. The risk assessment will focus on exposure assessment in use, and no new studies would usually be required. There is the possibility of authorization based an adequate control of exposure, but not for PBTs, vPvBs, and “non-threshold” CMRs. The ECA can, however, take into account socio-economic factors when deciding if use of the substance can nevertheless be authorized in the EU. The compromise text gives greater emphasis to the substitution principle, and applications for authorization must be accompanied by analysis of possible alternatives with their risks and the technical and economic feasibility of substitution.

The compromise text introduced an amendment to require authorizations to be subject to time-limited review, to enable further consideration of alternative substances. Authorization is also reviewed if information on possible substitutes is submitted to the ECA.

Restrictions for persistent organic pollutants (POPs) required under the Stockholm Convention will also be implemented through the restrictions provisions of the Regulation.

### 7.4.2
### Registration Obligations

Substances manufactured or imported, either neat or in a preparation, at >1 tonne per annum have to be registered, unless exempted. There is the option for a non-EU manufacturer to appoint an EU representative to register the substance an behalf of the EU importer(s).

Some substances in articles are subject to registration, and the provisions have been clarified under the compromise text. Substances in articles must be registered if they are intended to be released from the article and are supplied at >1 tonne per annum. Instead of registration, a less onerous procedure of notification applies to substances present in articles at >0.1 % unless release of the substance is excluded. Under the compromise text, articles manufactured in the EU are treated the same as imports. The ECA can, however, require a substance in an article to be registered if it poses a risk to human health or the environment.

Substances notified under the DSD are regarded as having been registered, as are active substances used only for products covered by the Plant Protection Products Directive or the Biocidal Products Directive (98/8/EC) or for coformulants of Plant Protection Products.

The regulation as a whole does not apply to radioactive substances, substances under customs supervision, substances in transit, and non-isolated chemical intermediates or waste. Substances are also exempt from registration if regulated by equivalent EU legislation (human and veterinary pharmaceuticals, food additives and flavorings, animal feed, and substances used in animal nutrition). Some categories of substance are, furthermore, exempt from registration (Table 7.8).

**Table 7.8** Categories of substances exempt from registration.

- Specific substances listed in Annex II of the Regulation
- Substances covered by Annex III:
  Substances produced by environmental degradation
  Substances produced by degradation during storage
  Substances produced during use
  Products from reaction with additives
  By-products
  Hydrates, if the anhydrous forms are registered
  Non-dangerous natural substances
  Hydrogen, oxygen, nitrogen and the noble gases
  Minerals, ores, and ore concentrates, natural gas, crude oil, and coal
- Monomers bound in polymers, but note that registration is required if the monomer is present at > 2 % (*w*/*w*) in the polymer and is at > 1 tonne per annum
- Polymers
- Food and food ingredients
- Waste and some recycled materials.

Substances needed in the interests of defense and non-isolated chemical intermediates do not have to be registered. Site-isolated intermediates at > 1 tonne per annum are registered with information about the identity of the manufacturer and substance, classification, and available test data. Registration also applies for isolated intermediates, which are transported between or supplied to other sites under contractual control (including for toll or contract manufacture) and for which there are strict conditions for manufacture and use to ensure only limited exposure. When transported at ≥ 1 tonne per annum these are registered with the same information as site-isolated intermediates, but at >1,000 tonnes per annum the basic Annex V test data are needed.

Although polymers do not have to be registered, if a polymer contains a monomer or other starting substance at ≥ 2 % (*w/w*) in the chemically bound form at ≥ 1 tonne per annum that has not been registered by another registrant, this monomer or starting substance must be registered by the polymer manufacturer or importer.

Substances used only for process-orientated research and development (PORD) are exempt from registration for five years (further extendable in exceptional circumstances). The manufacturer or importer must inform the ECA of the identity of the substance, its labeling, and its quantity, and must list the customers using it. Those customers only can use the PORD substance and it cannot be supplied to the public.

The registration dossier and chemical safety report is specified in Annexes IV to VIII of the regulation. The general technical, commercial, and administrative information needed for all registrations for the technical dossier is specified in Table 4 of Annex IV.

The technical dossier, including robust summaries of the study reports, for registration of chemicals under REACh is to be submitted to the ECA electronically using the International Chemical Information Database (IUCLID) format, which is an established database format for communicating and storing information an chemicals.

## 7.5
## Globally Harmonized System of Classification and Labeling (GHS)

The use of chemical products to enhance and improve life is a widespread practice worldwide. Alongside the benefits of these products, however, there is also the potential for adverse effects on people or the environment. As a result, several countries and organizations have developed laws or regulations over the years that require information to be prepared and transmitted to those using chemicals by use of labels or safety data sheets (SDS). Given the large number of chemical products available, individual regulation of all of them is simply not possible for any entity. Provision of information gives those using chemicals the identities and hazards of these chemicals, and enables the appropriate protective measures to be implemented locally.

Although these laws or regulations are similar in many respects, differences are sufficiently significant to result in different labels or SDS for the same product in different countries. As a result of variations in definitions of hazards, a chemical may be regarded as flammable in one country, but not in another. Or it may be believed to cause cancer in one country, but not in another. Decisions on when or how to communicate hazards on a label or SDS thus vary around the world, and companies wishing to be involved in international trade must employ many experts who can follow the changes in these laws and regulations and prepare different labels and SDS. In addition, given the complexity of developing and maintaining a comprehensive system for classifying and labeling chemicals, many countries have no system.

Given the reality of the extensive global trade in chemicals and the need to develop national programs to ensure their safe use, transport, and disposal, it was recognized that an internationally harmonized approach to classification and labeling would provide the foundation for such programs. When countries have consistent and appropriate information on the chemicals they import or produce in their own countries, the infrastructure to control chemical exposures and protect people and the environment can be established comprehensively.

In this sense, the new "Globally Harmonized System of Classification and Labeling of Chemicals" (GHS) addresses the classification of chemicals by types of hazard and proposes harmonized hazard-communication elements, including labels and safety data sheets. Its objective is to ensure that information on physical hazards and toxicity of chemicals is available to enhance the protection of human health and the environment during the handling, transport, and use of these chemicals. The GHS also provides a basis for harmonization of rules and regulations on chemicals at national, regional, and worldwide levels, an important factor also for trade facilitation.

The GHS was developed as a consequence of the "Rio Earth Summit" in 1992 and provides a basis for classifying and communicating the hazards of chemical products at different stages of their lifecycle from raw materials to recycling or disposal. The target beneficiaries of the new system include consumers, workers, and emergency responders and transport workers. The World Summit on Sustainable Development in 2002 requested all countries to implement the GHS and have it fully operational by 2008. Heads of Governments have signed up to this, and progress is being monitored by the UN.

Although governments, regional institutions, and international organizations are the primary audiences for the GHS, it also contains sufficient context and guidance for those in industry who will ultimately be implementing the requirements which have been adopted.

The European Commission has outlined its timetable to implement the GHS in the EU. This envisages that it will be implemented at the same time as REACh, which is currently foreseen for mid 2007. The GHS will replace the current EU classification legislation and will be the basis for classification under REACh.

The GHS will be implemented in the EU by means of a regulation, which will be separate from REACh but will provide the basis for classification for REACh. The European Commission is currently finalizing the draft text of the proposed legislation.

It is expected that the GHS will be implemented in two phases, with, first of all, substances having to be reclassified according to the GHS by the time registration under REACh commences. When this is complete, reclassification of preparations/mixtures will start.

There will be benefits for industry resulting from the global adoption of the GHS, because companies that trade multi-nationally or globally should eventually have a common basis for the classification and labeling of their products, irrespective of the countries in which they operate or with which they trade. The GHS thus provides the potential to facilitate international trade and simplify some of the business operations of companies.

The GHS contains two elements:

- harmonized criteria for classifying substances and mixtures according to their health, environmental, and physical hazards; and
- harmonized hazard communication elements, including requirements for labeling and safety data sheets.

The information in the SDS will use sixteen headings:

1. Identification
2. Hazard(s) identification
3. Composition/information on ingredients
4. First-aid measures
5. Fire-fighting measures
6. Accidental release measures
7. Handling and storage
8. Exposure controls/personal protection
9. Physical and chemical properties
10. Stability and reactivity
11. Toxicological information
12. Ecological information
13. Disposal considerations
14. Transport information
15. Regulatory information
16. Other information.

**Table 7.9** REACh and GHS – a comparison.

| ***REACh*** | ***GHS*** |
|---|---|
| Registration, evaluation, authorization | Classification, labeling, SDS |
| Risk (probably hazard-based within authorization) | Hazard |
| Substances produced | Substances marketed |
| Hazardous and nonhazardous | Hazardous |
| > 1 t per manufacturer/importer | Any volumes |
| Harmonized classification (CMRs at EU level) | Self classification |
| European Union | Global |
| Supply | Supply and transport |

## 7.6
## Environmental Legislation 2: Dangerous Preparations Directive (1999/45/EC)

The “Dangerous Preparations Directive” 1999/45/EC (DPD) effectively came into force in the European Community on July 30th 2002 and requires preparations to be evaluated for classification as “Dangerous for the Environment” (DFE) and, if necessary, to carry the “dead fish/dead tree” hazard symbol, based on the amount of DFE classified components they contain, or their intrinsic properties. DFE classifications have applied to substances since 1994, with the introduction of the 18th Amendment to Technical Progress.

Until now only single substances have had to be labeled as “dangerous for the environment” (symbol “N”), and no preparations. This has now changed – preparations must also be assessed according to exact regulations. Depending on the amount of hazardous substances it contains the preparation must be declared with relevant R-phrases or – in the worse case – classified as “dangerous to the environment”.

Thus directive 99/45/EC effects Material Safety Data Sheets for preparations. Article 14 extends the scope for the provision of Safety Data Sheets (SDS), extending the right of professional users to request SDSs containing “proportionate” information for some nondangerous preparations. In any event, the relevant R-phrases, respectively the symbol “N”, must be mentioned. Some details of the regulations are given below.

With directive 1999/45/EC limits for the concentrations of dangerous substances in mixtures are introduced:

| ***Labeling of substance*** | ***Concentration (%)*** | ***Labeling of mixture*** |
|---|---|---|
| N, R50-53 | > 25 | N, R50/53 |
| | 2.5–24.9 | N, R51/53 |
| | 0.25–2.4 | R52/53 |
| N, R51-53 | > 25 | N, R51/53 |
| | 2.5–24.9 | N, R52/53 |
| R52/53 | > 25 | R52/53 |

| | |
|---|---|
| R50 | Very toxic to aquatic organisms |
| R51 | Toxic to aquatic organisms |
| R52 | Harmful to aquatic organisms |
| R53 | May cause long-term adverse effects in the aquatic environment |

R52, R53 and the combination R52/53 are not given a symbol (for example “N”).

The limits for mentioning under point 2 in the SDS are given by:

0.1% for R50, R51, R53 (in combination with R50 or R51)
1.0% for R52, R53.

This is the "calculation respectively conventional method". Alternatively the health and environmental hazards of a preparation can be evaluated by testing the preparation using experimental methods, for example OECD. In general, classification derived using test data of the finished product override those given by the "conventional method", although there are several of exceptions to this. Any preparation containing more than the specified amount of a component classified as a carcinogen, mutagen, or reproductive toxicant must be classified using the conventional method – testing of preparations for biodegradability or bioaccumulation is not allowed. For acute toxicity, preparations must, furthermore, be tested on all three aquatic species (e.g. OECD 201, 202, 203) with the limit of $LC/EC_{50} > 100$ mg $L^{-1}$. After successful testing such a preparation is not awarded a symbol, even if the calculation scheme would require labeling.

Responsibility for determining whether the labeling must change as a result of the new directive lies with the supplier of a material. Individual companies are not obliged to require updated SDS from their supplier and they can legally rely on the SDS they hold in hands for raw materials purchased within the last year.

## 7.7
## Environmental Legislation 3: Regular use

All industrialized countries have laws which are designed to protect waters, the ground, working places and the air from pollution. However, the only ban on the use of mineral oil-based lubricants exists in Austria and this only for chain saw oil. Austria banned mineral oil-based chain saw oils effective May 1, 1992, following a resolution (No. 647) passed on October 16, 1990. Germany and a number of other countries have implemented provisions or quasi-legal procedures which are designed to promote the use of this new generation of products (e.g. the Environmental Seal in Germany). There is also political pressure to channel considerable funds into environmental protection. The following summarizes the laws and legislative initiatives in Germany which have an effect on the development and use of lubricants. Even if European legislation more and more dominates national legislation, a lot of individual laws and directives are to be considered.

The fundamental point is that "if lubricants are released into the environment during or after use, water, the ground and air are endangered".

The relevant ecological laws and regulations especially in Germany are:

- The German Water Law
- The Drinking Water Directive
- The German Ground Law (in draft)
- The Federal Emissions Law

- The Recycling and Waste Law
- The Chemicals Law
- The Environmental Liability Law
- German Parliament initiatives
- The 'Blue Angel' environmental seal.

The following chapters concentrate on German legislation.

### 7.7.1
### Environmental Liability Law

Following the introduction of the Environmental Liability Law in Germany, the environmental risks for a company have increased significantly.

This law states that causer is liable for environmental pollution regardless of blame. This ensures that it is easier for the 'victim' can claim compensation.

The law names about 100 types of factory which pose a potential risk in terms of liability, e.g. warehouses, machinery, vehicles, gearboxes, etc.

Important Articles of the law are:

- Article 6 Causer principle
- Article 7 Cause assumption (Liability on suspicion)
- Article 8 Obligation to inform
- Article 17 Funding of clean-ups.
- Article 19 Preventative obligations

The effects on lubricants are the reversal of the innocent until proven guilty principle increases the risks for operators using 'doubtful' lubricants. This risk can be reduced by the use of environmentally friendly products.

### 7.7.2
### The Chemicals Law, Hazardous Substances Law

The German Chemicals Law is oriented to safety-at-work considerations while its links with other environmental laws are still wanting. The European harmonization of the toxicological evaluation of chemicals, which is well advanced, has led to the labeling of lubricants if hazardous substances are contained and the uniform design of EU Safety Data Sheets.

According to the Hazardous Substances Law, the Definition of hazardous substances and mixtures (threshold concentrations of chemicals and additives) leads, in practice, to an increasing rejection of lubricants which contain hazardous substances even if the concentration of such is below the corresponding threshold. This means that hazardous substances are also rejected for mixtures (e.g. lead compounds in lubricants have largely been replaced by new non-toxic additives).

A recent addition to the German Chemicals Law also defines hazardous substances and mixtures according to hazard parameters. New to the list is the Parameter 'Environmentally dangerous'. Article 4 of the Hazardous Substances Law explains this as follows: “Substances or mixtures are environmentally hazardous if they or their decomposition products can alter the nature of water, the ground, the air, animals, plants or microorganisms in such a way that they suffer immediate or delayed harm”. Additives which display these features are thus also unsuitable for the development of ecologically friendly lubricants.

The effects on the use of lubricants are, for example:

- A reduction in the substances which pollute workplaces.
- The use of fewer ‘risky’ substances (e.g. oil changing, maintenance and production materials, the use of lubricants as production materials, contact with lubricants, during the servicing of machinery, etc.)

This limitation in the Chemicals Law and those in the Water Law severely restricts the number of additives which are suitable for environmentally friendly, rapidly biodegradable lubricants.

A further Chemicals Law restriction on additives for rapidly biodegradable lubricants results from the complicated approval procedures which are necessary for new substances.

### 7.7.3
### Transport Regulations

In Germany, these regulations focus primarily on the Transport of hazardous substances by road and rail.

These hazardous substances regulations classify hazardous substances into various groups. Category 3 is of greatest importance to lubricants. This category includes ignitable fluids with a flashpoint less than 100 °C (e.g. low-viscosity mineral oils). Environmentally friendly products based on rapeseed or ester oils normally have a considerably higher flashpoint than equi-viscous mineral oils.

### 7.7.4
### Disposal (Waste and Recycling Laws)

The Law on Waste Oils had a special status in Germany before it was integrated into the Wastes Law in 1986. This was then combined with a Recycling Law in 1996. Separate regulations determine the Waste Codes for waste products.

The integration into the Wastes Law obliged lubricant manufacturers to take back used engine and gear oils from consumers. As a rule, the lubricant manufacturers subcontracted the work to waste oil collection companies. In practice, the lubricant consumer pays for the collection and disposal or recycling. Moreover, the law has defined a number of waste oil categories with chlorine content (0.2 %) and PCB content (less important today) as major classification parameters. The great expense of disposing of products containing chlorine led to the development of chlorine-free products.

The most important methods and options for waste are listed in Table 7.10.

**Tab. 7.10** Methods and options for waste.

| *Product group* | *Methods + options* |
|---|---|
| Mineral oil-based hydraulic oils | Reconditioning (disposal) |
| Ester-based hydraulic oils | Reconditioning (disposal) |
| Mineral and ester oil-based engine and gear oils | Reconditioning (disposal) |
| Vegetable oils | Special waste, disposal |

Biodegradable lubricants based on synthetic esters are included in the term waste oil while vegetable oil-based products are not and are thus discriminated against by the law. As they are not included in the recycling and the associated waste oil laws, they have to be treated differently as their Waste Disposal Code indicates. The Code 12102 requires very careful monitoring and such wastes have to be stored and transported separately. In the reality of collecting used lubricants and hydraulic fluids, a certain normalization has taken place as regards vegetable oil-based products. However, the waste oil collectors still charge significantly more to dispose of vegetable oils than they charge for mineral oil-based products.

The chances are good that a revision of the Law on Wastes and other subordinate provisions and regulations will include vegetable oil-based lubricants in the term waste.

### 7.7.5
### Disposal Options for 'Not water pollutant' Vegetable Oils

The waste catalog presents the following options:

- Wastes which can be burned or dumped together with household wastes.
- Industrial or factory wastes which cannot be disposed of in household waste disposal plants but in special waste disposal plants.
- Wastes, which due to their toxicity, can only be incinerated in special plants fitted with exhaust filters or at sea.

A used, vegetable oil-based hydraulic oil cannot be allocated to any of these three groups because vegetable oil used as lubricants are not included in the term 'waste oils'. Instead, used vegetable oil-based lubricants have to be disposed of in line with the waste catalog. This catalog again defines three groups of wastes. The first includes wastes which can be disposed of or dumped together with household waste. However, used vegetable oil-based lubricants have been allocated Waste Code 12102 and thus are covered by the second group of wastes. This means that they cannot be disposed of together with household waste but must be incinerated in special plants (Waste Code 13503 applies to non-contaminated animal and vegetable oils). As regards their collection and storage, they are also special. This is because as lubricating oils (e.g. hydraulic oils), vegetable oils are not subject to the law on waste oils but to the general law on wastes and cannot at present be mixed with other waste oils which are used, for example, for heating purposes. In practice, this means that mineral based oils and vegetable oils have to be separately collected and stored.

Disposal possibilities for rapeseed-based hydraulic fluids:

- Burning for heating purposes
- Use as a concrete mould release oils
- Flux oils for bitumens (e.g. roofing felt and sealants)

Official reconditioning methods to convert used vegetable oil-based lubricants into a type of re-refined oil do not exist at present so that the above-mentioned disposal possibilities for such re-refined oils like flux oils or concrete mould release oils are not available until suitable refining technologies have been developed for used vegetable oil-based lubricants.

## 7.8
## Environmental Legislation 4: Emissions

### 7.8.1
### Air Pollution

Most air pollution is caused by the fossil fuels we burn in our vehicles, homes, thermal power plants and factories.

Many chemicals have been identified in urban air pollution. A small number of these have been found to contribute to a range of air quality problems. These pollutants include nitrogen oxides ($NO_x$), carbon monoxide (CO), sulfur dioxide ($SO_2$), particulate matter (PM) and volatile organic compounds (VOC). When some of them combine, they produce smog or acid rain. Ground-level ozone, the major component of smog, is formed when nitrogen oxides ($NO_x$) and volatile organic compounds (VOC) react in the presence of warm temperatures and sunlight. Another key element of smog is particulate matter.

### 7.8.2
### Water Pollution

The quality of freshwater and marine areas is affected by three important water pollution problems: toxic substances, excess nutrients, and sedimentation.

Toxic substances from industrial, agricultural and domestic use are some of the main pollutants in our water. These include trace elements, polychlorinated biphenyls (PCBs), mercury, petroleum hydrocarbons, dioxins, furans and some pesticides. Some of these substances accumulate through the food chain rather than break down in the environment.

These substances enter the water in a variety of ways, including: industrial sources such as mining, steel production, the generation of electricity and chemical production; accidents such as oil or chemical spills; municipal wastewater effluents; and agricultural run-off.

Excess nutrients such as nitrogen and phosphorous compounds come mainly from municipal sewage and farm run-off containing fertilizers and animal waste. These nutrients can cause excess growth of aquatic plants, which then die and decay, depleting water of dissolved oxygen and killing fish.

Sedimentation is an increase in the amount of solid particles in water, caused primarily by human activities such as forestry, farming and construction. When sediment settles, it can smother the feeding and spawning grounds of fish and kill aquatic organisms (that means: Toxic substances in the food chain).

The impact of lubricants to the environment, that means to soil, water and air, is restricted by different types of laws. Again the German situation will give an example for other countries.

### 7.8.3
### German Law for Soil Protection

In March 1999, a German Federal law to protect the ground should came into force. This law is a framework law, similar to the Water Law and the Federal Emissions Law and implementation will be the responsibility of the Federal States. While the Water Law only has an indirect effect on the ground (in the sense of clean water does not pollute), the Ground Law should avoid the build-up of ecologically harmful substances in the ground.

The law is aimed at private and industrial landowners along with the operators of factories (avoidance and preventative, clean-up and re-cultivating measures). The law will provide greater precision regarding environmental damage and a further restriction on the use of ecologically harmful substances.

Effects on the use of lubricants are: As a significant proportion of lubricants pollute the ground, the Soil Law will have profound effects on lubricant applications. For example the clean-up measures for mineral oil-polluted ground:

Because threshold values are not part of national laws designed to protect the ground, most European countries use the ‘Holland List’ which stipulates when mineral oil contamination requires cleaning-up. According to this list, the thresholds which then require clean-up measures are:

1. > 500 mg $kg^{-1}$ in residential areas and in Protected Water Zones
2. > 1000 mg $kg^{-1}$ for general cleaning-up
3. In individual cases and when confirmed by an independent expert, up to 5000 mg $kg^{-1}$ can be tolerated (such as in industrial areas with no ground water relevance).

lt is assumed that the necessary disposal and cleaning of 1 $m^3$ earth costs ca. 1000 €. This fact also promotes the development of environmentally friendly products which may generate far lower clean-up costs.

### 7.8.4

### German Water Law

German water law offers direct protection for waters and only indirect protection for the ground. At present, a law (titled: German Soil Law) directly aimed at protecting the ground is being drafted.

Important articles of the German Water Law are:

- ***Article 19 G***– Plant and equipment for handling water-polluting substances (mineral oils as well as their derivatives): "Plants and equipment designed to store, fill, manufacture or treat water-polluting substances as well as plant and machinery using water-polluting substances in trading companies and the public sector must be designed, constructed, installed, maintained and operated in such a way that no contamination of waters or any other disadvantageous changes to the characteristics of the water can occur. The same applies to pipework within the boundaries of a company".
- ***Article 22***– Liability for changes to the characteristics of the water: "Any party which allows substances to contact water which change the physical, chemical or biological characteristics of the water is liable for any damage caused. If more than one party is involved, then all are liable''.

These articles and German building regulations are important guidelines for lubricant manufacturers and customers alike. Applying these guidelines to the storage and use of lubricants could lead to restrictions if larger quantities of oil are involved (central warehouses, large mobile plant etc.). In the past, these regulations were handled differently in different parts of Germany. The States working party on water, which may form the basis of a national model, created a uniform definition of Hazard Categories and the accompanying preventive measures. Applying building regulations to the design and spillage protection of production plants would be enormously expensive.

According to the Environmental Agency's 'Guideline on Handling Water-Polluting Substances', the potential hazard depends on the volume of the plant and the Water Pollution Category of the substances used therein (see Chapter 7.3.4). The aim is to reduce this potential and thus avoid the massive cost of renewing plant and equipment by:

- Setting-up a plant and equipment register
- Securing and sealing flooring
- Setting-up containment capacity
- Infrastructure measures.

This is only possible with biodegradable lubricants. The German Water Law details biotest procedures (e.g. bacteria–algae toxicity) which are mandatory for the monitoring of wastes routed into public sewers. In addition, threshold concentrations of heavy metals are measured in wastes routed into public sewers (these can originate from cutting fluid emulsions which are contaminated with hydraulic oils containing zinc).

### 7.8.5
### Waste Water Charges

The charges for directly routing wastes into public sewers depends on the degree of contamination. The corresponding law defines:

- the definition of hazardous substances, e.g. heavy metals such as zinc and barium in lubricant additives, organic halogen compounds, etc.
- the charges for hazardous substances.

During use, such as in machine tools, lubricants come into contact with metalworking fluids, slideway oils and hydraulic oils. When water-miscible cutting fluids are split, parts of the hydraulic oil's additives can remain in the aqueous phase and in unfavorable circumstances, this can lead to increased waste water charges. This problem has lead to the development of zinc- and ash-free hydraulic oils. The use of ecologically friendly lubricants can also make a positive contribution to reducing pollution as well as lowering waste water charges.

### 7.8.6
### Clean Air: German Emissions Law

The German Emissions Law focuses on keeping the air clean and has had an increasing influence on the manufacture, use and disposal of lubricants in recent years.

The Emission Law affects lubricants if total emissions from a plant reach a critical level. lt should be remembered that in certain applications, over 10 % of the lubricant can evaporate or form oil mist. This in turn, can exhaust into the atmosphere as emissions. Lubricants which evaporate or mist significantly less than conventional products are already views as being environmentally friendly. Compared to equiviscous mineral oils, rapidly biodegradable, rapeseed oil- or ester-based products reduce emissions by up to 90 %. According to the Water Law, if the state-of-the-art is technically and economically feasible, all methods, installations and procedures should be employed to reduce emissions. According to the Emissions Law, all methods, installations or procedures which are practicable should be used to limit emissions. Regarding the determination of the state-of-the-art, comparable methods, equipment and processes were selected and tested for practical success.

### 7.8.7
### Drinking Water Directive

The cleanliness of drinking water is already an objective of the Water law, particularly with regard to the restrictions applicable in Protected Water Zones. An important aspect of laws concerning the safety of machinery is the classification of lubricants as combustible fluids. These laws lay-down measures for the storage of flammable fluids.

A lubricant which is not classified as being a combustible fluid and which is ‘not water hazardous’ is the least costly to store. This applies to a number of environmentally friendly lubricants.

## 7.9
## Standardization of Environmentally Compatible Hydraulic Fluids

The most known standardization of environmentally compatible lubricants concerns to hydraulic fluids because this was the application with the greatest amount of biodegradable alternatives up to now. The most important regulations for environmental compatible hydraulic fluids are shown in Table 7.11.

### 7.9.1
### The German Regulation VDMA 24568

The well known minimum requirements of mineral oil-based hydraulic fluids of DIN 51 524 do not satisfy the specifications of rapidly biodegradable hydraulic fluids. In order to do justice to the technical performance of different fluids, the VDMA (Association of German Machinery and Plant Manufacturers) created the specifications 24568 (Minimum Technical Requirements) and 24569 (Change-Over Guidelines). These specifications detail the minimum technical requirements of the ecologically safe product families; HETG (environment-compatible hydraulic fluids based on triglycerides, i.e. vegetable oils), HEES (based on synthetic esters) and HEPG (based on polyglycols).

### 7.9.2
### ISO Regulation 15380

The increasing importance of environmentally friendly fluids is reflected at an international level by the ISO (International Standards Organization): ISO 15380 “Lubricants, industrial oils and related products (class L) – Family H (hydraulic systems) – Specification for categories HETG, HEES, HEPG, HEPR” (Table 7.12). The European activities of CETOP (European Committee for Oil Hydraulics and Pneumatics) also reflects this activity.

The ISO standard 15380 deals with ecologically harmless fluids (ref. VDMA 24568 and 24569, enlarged to HEPR). It includes, in addition to technical requirements (Table 7.11), specific demands regarding the environmental impact of the hydraulic fluid.

ISO 15380 is the “technical standard” for European Eco-Labelling of Lubricants and for the German “Blue Angel”.

**Tab. 7.11** Regulations for environmentally compatible hydraulic fluids – minimum requirements HEES 46.

| | ***ISO 15380*** | ***EU Margerite 2005/360/EC*** | ***Blue Angel RAL-UZ 79*** | ***White Swan Version 4.2*** | ***Swedish Standard 15 54 34 ed. 4*** |
|---|---|---|---|---|---|
| ***Technical requirements*** | The technical criteria of EC Directive 2005/360/EC ('EC Ecolabel for Lubricants'), RAL-UZ 79 ('Blue Angel') and Nordic Ecolabelling for Lubricants (Version 4.2, 'White Swan') for hydraulic fluids refer to ISO 15380; the German VDMA Guideline 24568 is obsolete. | | | | |
| ***Foam*** | ISO 6247<br>(24/93/24) | ISO 6247<br>(24/93/24) | ISO 6247<br>(24/93/24) | ISO 6247<br>(24/93/24) | ISO 6247<br>(24/93/24) |
| Requirement | 150/0, 75/0, 150/0 | 150/0, 75/0, 150/0 | 150/0, 75/0, 150/0 | 150/0, 75/0, 150/0 | 150/0, 75/0, 150/0 |
| ***Air release*** | ISO 9120 | ISO 9120 | ISO 9120 | ISO 9120 | ISO 9120 |
| Requirement | max 10 | max 10 | max 10 | max 10 | max 10 |
| ***Seal swell*** | ISO 1817 | ISO 1817 | ISO 1817 | ISO 1817 | ISO 1817 |
| Requirement | Volume –3% to +10%<br>Elongation –30%<br>Tensile Strength –30%<br>Hardness +/–10% | Volume –3% to +10%<br>Elongation –30%<br>Tensile Strength –30%<br>Hardness +/–10% | Volume –3% to +10%<br>Elongation –30%<br>Tensile Strength –30%<br>Hardness +/–10% | Volume –3% to +10%<br>Elongation –30%<br>Tensile Strength –30%<br>Hardness +/–10% | Volume –3% to +10%<br>Elongation –50%<br>Tensile Strength –50%<br>Hardness +/–10% |
| ***Hydrolytic stability*** | not specified | not specified | not specified | not specified | not specified |
| Requirement | none | none | none | none | none |
| ***Cu corrosion*** | ISO 2160<br>(100 °C, 3h) | ISO 2160<br>(100 °C, 3h) | ISO 2160<br>(100 °C, 3h) | ISO 2160<br>(100 °C, 3h) | ISO 2160<br>(100 °C, 3h) |
| Requirement | rating max 2 | rating max 2 | rating max 2 | rating max 2 | rating max 1b |
| ***Rust prevention*** | ISO 7120,<br>procedure A | ISO 7120,<br>procedure A | ISO 7120,<br>procedure A | ISO 7120,<br>procedure A | |
| Requirement | pass | pass | pass | pass | |

**Tab. 7.11** Regulations for environmentally compatible hydraulic fluids – minimum requirements HEES 46 (continued).

| | ***ISO 15380*** | ***EU Margerite 2005/360/EC*** | ***Blue Angel RAL-UZ 79*** | ***White Swan Version 4.2*** | ***Swedish Standard 15 54 34 ed. 4*** |
|---|---|---|---|---|---|
| ***Demulsification*** | ISO 6614 | ISO 6614 | ISO 6614 | ISO 6614 | ISO 6614 |
| Requirement | none | none | none | none | max 30 min |
| ***Oxidation stability I*** | ASTM D 943 – dry | ASTM D 943 – dry | ASTM D 943 – dry | ASTM D 943 – dry | ASTM D 943 – dry |
| Requirement | report | report | report | report | min 1000 h |
| ***Oxidation stability II*** | DIN 51554-3<br>(95 °C/72h) | DIN 51554-3<br>(95 °C/72h) | DIN 51554-3<br>(95 °C/72h) | DIN 51554-3<br>(95 °C/72h) | ASTM D 943<br>DIN 51554-3 |
| Requirement | visc. max +20% | visc. max +20% | visc. max +20% | visc. max +20% | visc. max +20% |
| ***Low temperature properties*** | ISO 3104<br>(–20 °C/7d) | ISO 3104<br>(–20 °C/7d) | ISO 3104<br>(–20 °C/7d) | ISO 3104<br>(–20 °C/7d) | ISO 3104<br>(–20 °C/3d) |
| Requirement | no precipitates/<br>no particles | no precipitates/<br>no particles | no precipitates/<br>no particles | no precipitates/<br>no particles | max. 2400 mm$^2$/s |
| ***Filterability*** | not specified | not specified | not specified | not specified | ISO 13357-2 |
| Requirement | none | none | none | none | none |
| ***FZG gear test*** | DIN 51354 | DIN 51354 | DIN 51354 | DIN 51354 | DIN 51354 |
| Requirement | min 10 | min 10 | min 10 | min 10 | min 10 |
| ***Vane pump*** | ASTM D2882/IP 281 | ASTM D2882/IP 281 | ASTM D2882/IP 281 | ASTM D2882/IP 281 | DIN 51389 (=ASTM) |
| Requirement | 120/30 | 120/30 | 120/30 | 120/30 | 120/30 |
| ***Shear stability*** | not specified | not specified | not specified | not specified | DIN 51350-6/<br>ASTM D445 |
| Requirement | none | none | none | none | >6.5mm$^2$/s @100 °C |

**Tab. 7.11** Regulations for environmentally compatible hydraulic fluids – minimum requirements HEES 46 (continued).

| | ***ISO 15380*** | ***EU Margerite 2005/360/EC*** | ***Blue Angel RAL-UZ 79*** | ***White Swan Version 4.2*** | ***Swedish Standard 15 54 34 ed. 4*** |
|---|---|---|---|---|---|
| ***Occupational safety aspects*** | | | | | |
| ***Requirements concerning single substances*** | | not permitted if appearing in OSPAR list, organic halogen + nitrite compounds, metals or metallic compounds with the exception of sodium, potassium, calcium and magnesium | not permitted if classified as<br>• T+, T, R-45, R-46, R-48 *or* R-68 *or*<br>• accord. to MAK 905, TRGS *or* EC cat.1,2,3<br>– carcinogenic or<br>– mutagenic or<br>– toxic to reproduction | • base fluids should not be carcinogenic | • R-42 or R-43 are only permitted up to 1% |
| ***Requirements concerning the final formulated product*** | | product shall not have been assigned any R-phrase indicating environmental and human health hazards according to EC Directive 99/45/EC | • not classified as Xi or N accord. to EC Directive 99/45/EC<br>• max 50% concentration leading to classification of the formulation as Xn accord. to 99/45/EC | formulated hydraulic oil should not<br>• be classified as N<br>• no health hazard<br>• no explosion hazard | formulated hydraulic oil should not<br>• be harmful |
| ***Environmental aspects*** | | | | | |
| ***Base Fluids*** | | Base Fluids (> 5%) each<br>• aquatic toxicity in OECD 201 and 202 ≥100 mg/l | Base Fluids (> 5%) each<br>• >70% BOD/ThOD resp. CO$_2$ within 28d<br>• >80% in BODIS test<br>• LC/EC$_{50}$ not <100mg/l | Base Fluids each<br>• readily degradable *and* not R-50/53, not R-51/53, not R-50, not R-52/53, not R-53 | Base Fluids (> 5 %) each<br>• >60% BOD/ThOD resp. CO$_2$ within 28d (if solubility <100 mg/l)<br>• >70% COD in 28d (if solubility >100 mg/l)<br>• LC/EC$_{50}$ >100 mg/l |
| ***Additives*** | • no limitation on individual substances | • cumulative mass concentration of substances has to be:<br>– ≥90% ultimately biodegradable (f.e. ≥60% in OECD 301)<br>– ≤5% inherently biodegradable (f.e. >70% in OECD302C)<br>– ≤5% non-biodegradable<br>• substances that are non-biodegradable and bioaccumulative are not permitted | • max 5% inherently or not biodegradable substances, max 2% non-biodegradable<br>• not R-51/53 and bioaccumulative<br>• not R-52/53 and bioaccumulative<br>• not EC$_{50}$ <100mg/l in OECD 208<br>• not IC$_{50}$ <100 mg/l in OECD 209 | • no limitation on individual substances, see below | • no limitation on individual substances, see below |

**Tab. 7.11** Regulations for environmentally compatible hydraulic fluids – minimum requirements HEES 46 (continued).

| | ***ISO 15380*** | ***EU Margerite 2005/360/EC*** | ***Blue Angel RAL-UZ 79*** | ***White Swan Version 4.2*** | ***Swedish Standard 15 54 34 ed. 4*** |
|---|---|---|---|---|---|
| ***Polymers*** | | substance does not bioaccumulate if its MM >800 or has a molecular diameter >1,5 nm | • not mobile<br>• LC/$EC_{50}$ >100mg/l in OECD 201, 202 or 203<br>• $EC_{50}$ >100 mg/l in OECD 208 | | |
| ***Formulated product*** | • biodegradability accord. to ISO 14593 or ISO 9439: >60%<br>• Fish ISO 7346-2: $LC_{50}$ >100mg/l<br>• Daphnia ISO 6341: $EC_{50}$ > 100mg/l<br>• Inhibition ISO 8192: $EC_{50}$ >100mg/l | aquatic toxicity in all 3 OECD tests 201, 202 and 203 ≥100 mg/l. Alternatively: special requirements for each constituent substance | • max 2% non degradable<br>• no AOX, $NO_2$-salts, metals, except 0.1% Ca | • max 3% R-53 or R-52/53<br>• max 2% R-50 or R-50/53<br>• max 1% R-51/53 | • max 1% of components with $LC_{50}$ <1 mg/l<br>• max 5% of components with $LC_{50}$ = 1–100 mg/l |
| ***Renewable material*** | no requirements | >50% in hydraulic fluids | no requirements, but the German Market Introduction Program requires 50% renewable components | min 65% renewable substances in the product required | no requirements, but the environmental criteria laid down in SS 155434 correspond to the B level of the “Clean lubrication” definition of the City of Göteborg. The A level of the “Clean lubrication” definition of the City of Göteborg requires that only material from renewable resources are used |

**Table 7.12** Rapidly biodegradable hydraulic fluids according to ISO 15380

| | | |
|---|---|---|
| HEPG | Polyalkylene glycols soluble in water | Hydrostatic drives, e.g. locks, “water hydraulics”, to <90 °C |
| HETG | Triglycerides (vegetable oils), not soluble in water | Hydrostatic drives, e.g. mobile hydraulic systems, –20 to <70 °C |
| HEES | Synthetic esters, not soluble in water | Hydrostatic drives, mobile and industrial hydraulic systems, –30 to <90 °C |
| HEPR | Polyalphaolefins and/or related hydrocarbons, not soluble in water | Hydrostatic drives, mobile and industrial hydraulic systems, –35 to <80 °C |

Numerous specifications have also been issued by leading hydraulic component manufacturers and their direct customers (Mannesmann–Rexroth, Sauer Sundstrand, Caterpillar, Komatsu, etc.).

## 7.10
## Environmental Seal

To combine the environmental behavior and the technical properties of lubricants a lot of countries have introduced so called ‘eco-labels’ or ‘eco-logos’. The labels should give a sense of security to the users of environmental compatible products. Amongst many household appliances the most countries include lubricants in the system of eco-labeling.

### 7.10.1
### Global Eco-labeling Network

The Global Eco-Labeling Network (GEN) is a new international initiative of the national environmental labeling organizations with the purpose of creating a forum for information exchange and promotion of eco-labeling. GEN is a non-profit association of organizations from around the world. To date, eco-labeling agencies from Spain, the United States, Canada, Sweden, Finland, Norway, Taiwan, Japan and the UK have committed to membership. India and Greece are also in the process of commitment.

### 7.10.2
### European Eco-label

The current EU Eco-Label (“EU Margerite”) award scheme has been in operation since 1993, when the first product groups were established. For all product groups the relevant ecological issues and the corresponding criteria have been identified on the basis of comprehensive studies of environmental aspects related to the entire life-cycle of these products.

With publication in the Official Journal of the European Community on 5 May 2005 the directive 2005/360/EC came into force, establishing ecological criteria and related assessment and verification requirements for the award of the Community Eco-Label to Lubricants. The "Competent Body" for the eco-labeling process was the Netherlands' Stichting Milieukeur, the task of preparing the criteria was given to the consulting-bureau IVAM.

The eco-labeling process started two years ago with discussions with the stakeholders. Because lubricants vary substantially, depending on their applications, it was clear from the very beginning that this product group had to be divided in special subgroups. Final selection of the first version of the EU Eco-Label for Lubricants was driven by the environmental relevance of the products:

- hydraulic fluids
- chainsaw oils, concrete release agents, other total-loss lubricants
- two-stroke engine oils
- lubricating greases.

Automotive lubricants were not included, because the most relevant environmental issues for these types of lubricant differ from those selected – according to some Life Cycle Assessments fuel efficiency improvement by use of advanced engine and gear oils is of greater importance to the environment than biodegradability. This does not exclude the possibility of developing criteria for automotive lubricants in the future.

The ecological criteria for the product group "Lubricants" is valid until 31 May 2009. National "Competent Bodies" are responsible for implementation. The seven criteria for European eco-labeling of lubricants are described briefly below.

**Criterion 1:** The product shall not have been assigned any R-phrase at the time of applying for the eco label, indicating environmental and human health hazards according to European Preparation Directive 1999/45/EC.

**Criterion 2:** For aquatic toxicity **two** approaches are allowed – one for the fully formulated preparation and one for the single components.

1. Requirements for preparations:
   Aquatic toxicity data shall be provided for the fully formulated product and the main components:
   - main components are all substances > 5 %, i.e. base oils and thickener;
   - additives are allowed up to a limit of 5 %.

| *Criterion 2.1: Aquatic toxicity, first approach* | *Hydraulic fluids* | *Greases* | *Chainsaw oils, concrete release agents, other total-loss lubricants* | *Two-stroke engine oils* |
|---|---|---|---|---|
| Aquatic toxicity for the fully formulated product in all 3 of the acute toxicity tests – OECD 201, 202 and 203 | >100 mg $L^{-1}$ | >1000 mg $L^{-1}$ | >1000 mg $L^{-1}$ | >1000 mg $L^{-1}$ |
| Aquatic toxicity for each individual main component in OECD 201 und 202 | >100 mg $L^{-1}$ | >100 mg $L^{-1}$ | >100 mg $L^{-1}$ | >100 mg $L^{-1}$ |

Greases can be evaluated in this way only if:

- biodegradation of the thickener is >70 % in OECD 302 C; or
- biodegradation is > 20 % but < 60 % after 28 days in OECD 301 B or F.

2 Requirements for single components:
Aquatic toxicity data shall be provided for **each** constituent substance according to OECD 201 and 202 – without differentiation of the main components and additives. Single component and constituent substance means any substance which has been deliberately added and which constitutes more than 0.1% of the product's content, as measured both before and after any chemical reaction has occurred between the substances mixed to produce the lubricant preparation.

| *Criterion 2.2: Aquatic toxicity, second approach* | *Maximum treat rates of substances with some aquatic toxicity: Cumulative mass concentration of substances [%] in:* | | | |
|---|---|---|---|---|
| | *Hydraulic fluids* | *Greases* | *Chainsaw oils, concrete release agents, other total-loss lubricants* | *Two-stroke engine oils* |
| 10 mg $L^{-1}$ < acute toxicity* ≤ 100 mg $L^{-1}$ | ≤ 20 | < 25 | ≤ 5 | ≤ 25 |
| 1 mg $L^{-1}$ < acute toxicity* ≤ 10 mg $L^{-1}$ | ≤ 5 | ≤ 1 | ≤ 0.5 | ≤ 1 |
| Acute toxicity* < 1 mg $L^{-1}$ | ≤ 1 | <0.1 | <0.1 | <0.1 |

*EC50/LC50/IC50 in accordance with OECD 201/202

**Criterion 3:** Requirements with regard to biodegradability must be measured for each component. For this criterion determination for the fully formulated product is not of interest. Stipulated amounts of some nonbiodegradable substances are allowed, in principle, but only if they are not bioaccumulative. *Not bioaccumulative* are higher molecular weight or minor amounts of bio-active substances (molecular weight > 800 *or* molecular diameter > 1.5 nm *or* log $K_{ow}$ < 3 or > 7 *or* BCF ≤ 100). In other words, substances which are both nonbiodegradable and bioaccumulative are not permitted.

| ***Criterion 3: Biodegradability and bioaccumulative potential*** | ***Cumulative mass concentration of substances [%] in:*** | | | |
|---|---|---|---|---|
| | ***Hydraulic fluids*** | ***Greases*** | ***Chainsaw oils, concrete release agents, other total-loss lubricants*** | ***Two-stroke engine oils*** |
| Non biodegradable | ≤ 5 | ≤ 10 | 0 | ≤ 10 |
| Inherently aerobically biodegradable | ≤ 5 | ≤ 20 | ≤ 5 | ≤ 20 |
| Ultimately aerobically biodegradable | > 90 | > 75 | > 90 | > 75 |

**Criterion 4:** Specific substances to be excluded as intentionally added ingredients from lubricants applying for the EU Eco-Label.

| ***Criterion 4: Exclusion of specific substances*** | Intentionally added ingredients shall not be:<br>• substances appearing in the Community list of priority substances in the field of water policy and the OSPAR List of Chemicals<br>• organic halogen compounds and nitrite compounds<br>• metals or metallic compounds with the exception of sodium, potassium, magnesium, and calcium. Lithium and/or aluminum compounds also may be used in thickeners |
|---|---|

**Criterion 5:** The demand for a minimum content of renewable raw material within the finished lubricant is motivated by the European Climate Change Program (ECCP). In the final product the carbon content must reach the limits:

| ***Criterion 5*** | ***Hydraulic fluids*** | ***Greases*** | ***Chainsaw oils, concrete release agents, other total-loss lubricants*** | ***Two-stroke engine oils*** |
|---|---|---|---|---|
| Carbon content derived from renewable raw materials (%, $m/m$) | ≥ 50 % | ≥ 45 % | ≥ 70 % | ≥ 50 % |

**Criterion 6:** At the beginning of the eco-labeling process it was recognized that minimum technical requirements are essential for acceptance of bio-lubricants. Thus, for hydraulic fluids, chain-saw oils, and two-stroke engine oils the technical basis is described by accepted standards; for greases and total-loss lubricants, only, a general “fit for use” is required.

| ***Criterion 6*** | ***Hydraulic fluids*** | ***Greases*** | ***Chainsaw oils*** | ***Concrete release agents, other total-loss lubricants*** | ***Two-stroke engine oils*** |
|---|---|---|---|---|---|
| Technical requirements | ISO 15380 | "Fit for use" | "Blue Angel" UZ 48 | "Fit for use" | NMMA TC-W3 |

**Criterion 7:** This criterion is related to the special appearance of the eco-label with regard to the product group "Lubricants". The information appearing in the "Box 2" of the eco-label shall contain the text: "Reduced harm for water and soil during use; reduced $CO_2$ emissions".

In summary, the main target of the (voluntary) EU Eco-Label for lubricants is to draw attention to products which have the potential to reduce negative environmental impact. A special European approach for ending the confusion of different national eco-labels in Europe is not yet recognizable.

### 7.10.3
### The German 'Blue Angel'

The world's first eco-labeling program, the German 'Blue Angel', was created in 1977 to promote environmentally sound products, relative to others in the same group categories. The authorities hoped it would be seen as a positive step not only for individual consumers but for the retailers and manufacturers as well. Anyone can propose product groups for the 'Blue Angel' award. It is encouraged, apparently, "in the interests of global warming management".

The institution responsible for assessing such proposals is the Umweltbundesamt (German Environmental Protection Agency, UBA) guided by an Ecolabel Jury which considers these proposals. The Jury is composed of representatives from industry, environmental organizations, consumer associations, trade unions, the Churches and public authorities, in order to ensure that the interests of various groups in society are taken into consideration.

The label is composed of a blue figure with outstretched arms surrounded by a blue ring with a laurel wreath. A standard inscription, 'Umweltzeichen' (environmental label) is at the top of the logo, and a second inscription for the individual product group is found at the bottom, for lubricants it is "... because rapid biodegradable".

This eco-label relies on information and voluntary cooperation, as well as on the motivation and the willingness of each individual to make a contribution towards environmental protection. The Blue Angel is addressed at all market players, enabling retailers and consumers to make deliberate choices in favor of environmentally sound alternatives. Once approved, eco-labeled products are reviewed every two or three years to reflect state-of-the-art developments in ecological technology and product design.

The Eco-Label Jury scrutinizes product groups twice yearly. The criteria for awarding the Blue Angel includes: the efficient use of fossil fuels, alternative products with less of an impact on the climate, reduction of greenhouse gas emission, and conservation of resources.

The German environmental award should highlight products which are more environmentally compatible than others. The following environmental awards have been issued for lubricants:

- RAL UZ 48 for Chainsaw oil (1988)
- RAL UZ 64 for Rapidly biodegradable lubricants and shuttering oils (1991)
- RAL UZ 79 for Rapidly biodegradable hydraulic oils (1996)

For example RAL UZ 79 for rapidly biodegradable hydraulic oils focuses on the points:

- *Objective*: To reduce environmental pollution
- *Application*: Hydraulic systems
- *Requirements*: Formulation, ingredients, disposal, performance
- *Proof*: Formulation, expert evaluation, statement, ISO 15380
- *Issued by*: Product manufacturer, RAL, Federal Environment Agency
- *Award use*: Contract, duration
- *Wording*: Awarded because rapidly biodegradable.

Together with the German Ministry for the Environment, Nature Protection and Nuclear Safety, the awarding committee has decided that the conditions for issuing this award will consider results obtained by the RAL organization consisting of a commission of consumer experts, manufacturers and Federal Environment Agency. The actual issuing of the award is performed by the RAL organization.

The third issuing guideline represents a tightening-up of the first two guidelines in that it covers the use of hazardous substances (additives). The guideline combines demands originating in chemical law, the law on water, biodegradability and eco-toxicological evaluations.

The 'Blue Angel' combines the performance level of the requirements of ISO 15380 with environmental relevant criteria, which are not compatible with the requirements of the European Eco-label.

### 7.10.4
### Nordic countries (Norway, Sweden, Finland, Iceland) – 'White Swan'

The Nordic Swan Label claims to be neutral, independent, and the world's first multinational ecolabeling scheme. Only products that satisfy strict environmental requirements on the basis of objective assessments are allowed to display the environmental product label.

The criteria for the eco labeling of lubricating oils (Nordic Ecolabeling 05.09.1997) encompass lubricating oils with a lubricating and pressure-transmitting effect. The product group encompasses engine oil, transmission oil, hydraulic oil, lubricating grease, two-stroke oil, metal cutting fluid, saw chain oil and mold oil.

Eco labeled lubricating oils must not be subject to classification under regulations concerning hazards to health, environmental hazards, fire hazards or explosion hazards, except products classified in accordance with the 'Mättligt hälsoskadlig' system in Sweden.

The packaging must not contain chlorinated plastics and must be designed in such a way that as little oil as possible is able to remain in the packaging. The type of plastic used in plastic parts must be marked on the parts concerned.

In the case of the products engine oil, transmission oil, hydraulic oil, lubricating grease and two-stroke oil strict requirements are imposed with regard to the permitted level of non-renewable raw materials, re-refined oil, environmentally harmful components and potentially non-degradable components. These parameters are assessed on an overall basis so that a product with a high score in one area may score somewhat lower in another area.

Separate requirements are imposed with regard to saw chain oil, mold oil and metal cutting fluids. Products of this type can not be based on mineral oil.

Documentation must be provided on lubricating oils demonstrating that the lubricating oil in question is of the same quality as an average of competing products available on the market.

Environmentally adapted lubricating oils are produced on the basis of either vegetable or synthetic base oils. Traditional mineral base oils will not satisfy the requirements in the criteria since they have a higher toxic effect on waterborne organisms and poorer biological degradability compared with vegetable and synthetic base oil.

Vegetable base oils and certain synthetic base oils which are extracted from renewable resources (plants) are rewarded in the criteria.

The policy in Norway is that used lubricating oils should be collected and to this end the Government has established a tax refund scheme on returned waste oil. According to this scheme people or organizations in possession of waste oil receive oil delivered free of charge or are paid for each liter that is returned. As a result of this system considerably lower quantities of oil go astray.

A similar return waste oil scheme is being established on Iceland according to which waste oil is returned to the oil companies for incineration under controlled conditions (cement production plant). Finland, Sweden and Denmark have no corresponding return oil scheme but do have systems for collecting waste oil.

The criteria also take account for the fact that certain lubricating oils require a higher percentage of components that are considered environmentally hazardous. Permitting a higher percentage of environmentally harmful components in certain products can be justified on the grounds that these components may improve the performance of the products.

Weights have been ascribed to the parameters on the basis of their environmental significance. For a product to qualify for eco-labeling it must attain a minimum score.

#### 7.10.4.1 Requirements Concerning Renewable Resources

The environmental matrix regulates the use of renewable and non-renewable sources by means of a maximum permitted quantity (% *w*/*w* of the product).

The purpose of this requirement is to permit some synthetic base oils.

#### 7.10.4.2 Requirements Concerning Re-refined Oil

The environmental matrix permits the use of a limited proportion re-refined waste oil in motor oil and transmission oil.

#### 7.10.4.3 Requirements Concerning Environmentally Harmful Components

Components which are classified as environmentally harmful according to current regulations in Denmark, Finland, Iceland, Sweden or Norway or EU Directive 67/548/EEC 18th adaptation with risk allocations R50, R53, R50 + R53, R51 + R53, or R52 + R53 are subject to limits in the environmental matrix with a maximum threshold value for each individual product.

The same requirements concerning environmental harmfulness apply to substances that have been shown to form persistent environmentally harmful degradation products in relevant conditions.

#### 7.10.4.4 Requirements for Hydraulic Fluids, Mould Oil, Metalworking Fluids

##### Requirements A

Data sheet/confirmation to show which components constitute renewable resources.

##### Requirements C

Test results for all components in the product (base oil and additives) performed in accordance with the following OECD test methods:

- Rapid biodegradability: OECD 301 A–F
- Bioaccumulation: OECD 107, 117 or if applicable 305
- Ecotoxicity: OECD 201, 202 and 203.

If corresponding test methods are used, the similarities/differences of the method must be verified by an independent body. This is necessary to ensure credible test results.

Requirements concerning potential degradability (hydraulic fluids, mould oil): The test result for all components in the product (base oil and additives) performed in accordance with OECD test method 302 A–C.

### 7.10.5
### The Canadian ‘Environmental Choice’ (Maple Leaf)

In 1994, total sales of lubricant products in Canada equaled almost 1 billion liters, of which 200 000 L were vegetable oil-based lubricants.

In all the usage of vegetable oil based lubricants is increasing in Canada because many companies prefer environmentally friendly products. By 2000, sales of vegetable oil lubricants are expected to one million liters annually.

Vegetable oil lubricants are priced twice as high as conventional petroleum lubricants. The willingness of Canadian companies to pay very high prices for vegetable oil lubricants indicates a preference for these products in environmentally sensitive areas.

The ‘Environmental Choice Program’ (ECP), Environment Canada’s ecolabeling program, provides a market incentive to manufacturers and suppliers of environmentally preferable products and services, and thereby helps consumers identify products and services that are less harmful to the environment. Established in 1988, the ECP was the second national ecolabeling initiative undertaken.

Canada’s ‘Environmental Choice’ Eco-Logo symbol of certification features three stylized doves intertwined to form a maple leaf, representing consumers, industry and government working together to improve Canada’s environment.

A product or service may be certified because it is made or offered in a way that improves energy efficiency, reduces hazardous by-products, uses recycled materials or because the product itself can be reused. Product manufacturers, importers or purveyors of services may apply for a license to use the Eco-Logo once a guideline containing criteria relevant to the product or service type has been approved. Environmental Choice guidelines are based on the best information available at the time and are upgraded as new information and technology make higher standards possible. Guidelines are developed in consultation with industry, environmental groups, universities and independent technical and scientific advisors.

Environmental Choice issues kits to potential licensees, and Environment Canada’s independent Technical Agency assists companies through the application process. Agreements granting use of the Eco-Logo are renewed annually and continued compliance with the guideline is monitored. Certified products and services must continue to meet all applicable safety and performance standards; specifically, they must be as good in every other respect as is generally expected of that type.

Currently, Environmental Choice has more than 1400 approved products, with 119 licensees and 29 guidelines under which companies may be licensed and their products certified. In the area of lubricants there are listed anticorrosion products, automotive engine oil, synthetic industrial lubricants, vegetable-based industrial lubricants.

‘Vegetable-based industrial lubricants’ must fulfil the criteria:

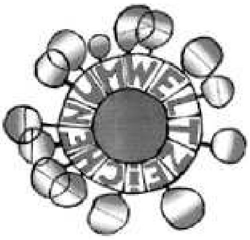
- Not be toxic to fish by demonstrating an $LC_{50}$ not lower than 40 000 mg $L^{-1}$ when tested according to the Acute Lethality Test Using Rainbow Trout, Report EPS 1/RM/9, July 1990, Environment Canada, biological test method
- Be biodegradable, according to CEC L-33-A-93 or one of OECD 301 A–F
- Not contain more than 5 % (*w*/*w*) additives

- Not contain more than 3 % (*w*/*w*) of an additive that is not proven to be biodegradable
- Not contain petroleum oil or additives containing petroleum oil as confirmed by EPA TPH 418.1 with a measured reading no greater than 10.6 g $kg^{-1}$
- Not contain organic chlorine or nitrite compounds or lead, zinc, chromium, magnesium, or vanadium
- Not have to be labeled according to Class D, Poisonous and Infectious Material, as set out in the Controlled Products Regulations of the Hazardous Products Act
- Yield pass results when tested against ASTM D 665 (Standard Test Method for Rust Preventing)
- Characteristics of inhibited mineral oil in the presence of water
- Not have a flash point lower than 200 °C, if ISO grade VG 32 and higher, and not lower than 190 °C, if ISO grade VG 15-22
- Produce a minimum fire point of 311 °C as per ASTM D 92
- Demonstrate a viscosity index of at least 200 as per ASTM D 2270
- Demonstrate a capacity to produce a peroxide value no greater than 15 milliequiv. after 1000 h

The Canadian Environmental Protection Act (CEPA) gives support to the use of labeled lubricants. The CEPA was proclaimed into law in 1988 and is designed to protect Canadians from pollution caused by toxic substances. It provides the power to regulate the entire life cycle of toxic substances.

### 7.10.6
### Other Eco-labels

#### 7.10.6.1 **Austria**



The Austrian eco-label was created by Friedensreich Hundertwasser. It is the sign for products and services with low impact to the environment for product lifetime.

The only relevant lubricant guideline is published under no UZ 14 (January 1997): ‘Chain saw oils based on vegetable oil’. This guideline is usable for chain saw oils with an amount of vegetable oil of at least 95 %. It is allowed to add 5 % additives.

#### 7.10.6.2 **France**

The ‘NF Environnement mark’ is the Eco-Logo for France. Created in 1992, it features a single leaf covering a globe.

The mark means that the product has less impact on the environment while achieving the same level of service as other products on the market. Industries who wish to highlight their environmental efforts can voluntarily apply to use the Eco-Logo on their products.

Certification is based on a multi-criteria approach, combining technical and environmental requirements. Since 2003, for chain saw oils a French Eco-label is available, as first and only luibricant group. The identification number is NF375: "Lubrifiants pour Chaines de Tronconneuse".

### 7.10.6.3 Japan

Since 1989, the Japanese Environmental Association (JEA) has administered the Eco Mark Program with the goal of disseminating environmental information on products and encouraging consumers to choose environmentally sound products. The symbol itself represents the desire to protect the earth with our own hands, using the phrase 'Friendly to the Earth' at the top of the symbol and the product category below it.

This goal will be accomplished by authorizing the Eco Mark to be displayed on products that reduce the environmental load caused by every-day activities, thereby contributing to the preservation of the environment.

In principle, products must meet the following criteria: impose less environmental load than similar products in their manufacture, use and disposal; and reduce the environmental load in other ways, thus contributing significantly to environmental conservation. This scheme also is applicable to lubricants.

The Eco Mark Program is intended as a means of offering a choice of products with a lower environmental impact.

### 7.10.6.4 USA

Green Seal is an American non-profit environmental labeling organization that awards a 'Green Seal of Approval' to products found to cause significantly less harm to the environment than other similar products. The Green Seal certification mark identifies those products which are environmentally preferable, empowering consumers to choose products based on their environmental impacts.

Green Seal develops environmental standards for consumer products through a public review process involving manufacturers, environmental organizations, consumer groups and government agencies. Products are certified only after rigorous testing and evaluation. Underwriters Laboratories Inc. (UL) is the primary testing contractor for Green Seal.

To date, Green Seal has awarded its seal of approval to 234 products, and certifies products in over 50 categories including apart from major household appliances also re-refined engine oil.

### 7.10.6.5 The Netherlands

The Netherlands have a special system to give support to environmental compatible products – the accelerated depreciation of environmental investments, the so called VAMIL regulation.

The VAMIL measure has been in effect since September 1, 1991. It is a tax facility offering companies the opportunity to apply accelerated depreciation on environmentally friendly operating assets. If the asset is operational and fully paid for, it even allows depreciation of the full purchase price in the year an asset is acquired. This provides an attractive liquidity and interest gain for these companies. Eligible operating assets appear on a special 'VAMIL list'. The measure is not aimed at a specific environmental problem or region, but has a very wide operating ambit. The 1993 list, for example, contained elements aimed at reducing water, soil and air pollution, noise emissions, waste production and energy use.

To be eligible for the VAMIL list, operating assets should:

- Be clearly defined for fiscal purposes;
- Have relatively good environmental impacts;
- Not yet be widely accepted in The Netherlands;
- Have no negative side effects, such as excessive energy use; and
- Have a substantial potential market.

Periodically – in principle, once every year – the VAMIL list is replaced by a new one. Adaptations include the removal of operating assets that have become widely accepted and the addition of new environmentally friendly technologies. The list is prepared by: Bureau Vervroegde Afschrijving en Milieu, Ministry of Finance and Department of Environmental Investments (VROM – Department of Environmental Investments). It is hoped that eventually the VAMIL list will correspond to approximately 30% of all investments in environmentally friendly operating assets.

All companies and persons liable to pay income or corporate taxes in the Netherlands can make use of the measure. However, the measure aims mainly at small- and medium-sized companies. The government determines a budget for the VAMIL measure once every year, setting an upper limit for tax allowances. The budget does not reflect government expenditures, as the reduced tax revenue in a given year is followed by increased tax revenue in later years. Therefore the cost to the government consists only of lost interest. Additional costs necessary to make the asset operative are also eligible for accelerated depreciation. If an asset is developed and produced within a company, own-production costs can be depreciated in an accelerated manner.

## 7.11
## Base Fluids

### 7.11.1
### Biodegradable Base Oils for Lubricants

The main 'chemistry' concerning biodegradable lubricants is different types of ester oil, e.g.:

- Vegetable oil from harvestable raw materials, for example rapeseed or sunflower oil

- Semi-saturated, transesterified ester oils with natural fatty acids, for example trimethyolepropanetrioleate
- Fully saturated, synthetic esters based on chemical modified vegetable oils or mineral oil, for example Diisotridecyladipate

Other in principle biodegradable base oils mostly are not present in the market of ‘environmentally compatible lubricants’ due to different technical or environmental reasons.

- Poly(alkylene glycol) (PAG)
- Low viscosity polyalphaolefins (PAO2)
- Some special types of synthetic hydrocarbon up to a viscosity of 6 $\mathrm{mm^2\ s^{-1}}$ at 100 °C.

The lubricants industry has invested significant sums of money in developing and marketing biodegradable lubricants.

Natural fatty oils such as castor oil, palm oil, rapeseed oil, soybean oil, sunflower oil, lard, tallow and sperm oil have been used in lubricants for years. They are so-called triglycerides of more or less unsaturated fatty esters. This type of base is biodegradable and, compared to mineral oils, will show excellent tribological qualities (low friction coefficient, good wear protection). Their range of use is limited by lower stability against thermal oxidative and hydrolytic stress and partly inferior cold flow properties. These limits can be improved gradually either with additives, or with the selection, cultivation or genetically modification of new types of plants. With new types of ‘High oleic sunflower oils’ (HOSO) with an amount of oleic acid of more than 90 % it seems possible to formulate oils for higher performance levels.

### 7.11.2
### Synthetic Esters

The wider use of natural base oils for additional, large volume lubricant technologies highlighted the dilemma that unaltered natural oils cannot satisfy a number of technical requirements while defined and highly-specialized modification in a number of manufacturing stages prices them out of competition.

The collective name ‘synthetic esters’ covers a broad range of chemicals with different qualities and prices. For the development of environmentally acceptable lubricants, esters have to be selected which fulfil the ecological requirements and have more favorable properties than natural fatty oils.

These properties are mainly thermal-oxidative resistance, better low temperature behavior and better resistance to hydrolysis. Chemistry offers a wide range of possibilities in the area of synthetic esters.

At present, polyolesters such as trimethylolpropane esters (TMP-esters) dominate. The basis of these are mainly alcohols from petrochemical and oleochemical industries and fatty acids derived from natural oils.

In regard to hydrolytic stability, ‘normal’ polyolesters differ only slightly from rapeseed oil; the difference in oxidation resistance is much greater. Both characteristics are significantly improved with complex esters. Normally an improvement in hydrolytic stability worsens the base fluid's biodegradability.

The most important chemical reactions to improve the properties of esters are transesterification, (selective) hydrogenation, ozonolysis, dimerization.

However, there are complex esters (medium chain, saturated fatty acids on trimethylolpropane or other polyols) which combine excellent thermal-oxidative characteristics with good hydrolysis resistance and good biodegradability.

### 7.11.3
### Polyglycols

Lubricants based on polyalkyleneglycols (PAG) can have very good technical properties and are well-known in long-term practical use.

Polyethylene glycols (PEG) are mostly biodegradable. They are not miscible with mineral oils or esters, but are water-soluble which, at the present time, is considered to be a disadvantage. If PEG is released through a leak or an accident, it will migrate quickly in the ground or in the water.

Polypropylene glycols (PPG) are (partially) miscible with mineral oils or esters, but in general not easily biodegradable.

The chemical industry is attempting to develop biodegradable, non-water soluble PAGs. These could be alternatives to esters.

### 7.11.4
### Polyalphaolefins

Low viscous polyalphaolefins (PAO 2) are biodegradable. These base stocks have only limited application in the formulation of lubricants. The new types of biodegradable, higher viscosity synthetic hydrocarbons may have a greater influence in the future.

### 7.11.5
### Relevant Properties of Ester Oils

#### 7.11.5.1 Evaporation Loss

The evaporation loss of esters and vegetable oils is excellent as can be seen in Fig. 7.3. The evaporation loss of various base fluids have been determined with the Noack test method according to DIN 51 581. It is apparent that vegetable oils such as rapeseed oil give the best results.

The lower evaporation loss of ester oils has great advantages for example in regard to emissions from machine tools in metalworking and for the emissions, especially the particle emissions, of internal combustion engines.

#### 7.11.5.2 Viscosity–Temperature Behavior

Using an ester instead of a mineral oil improves the viscosity–temperature behavior of a lubricant. The higher viscosity index (VI) of an ester results in a wider temperature

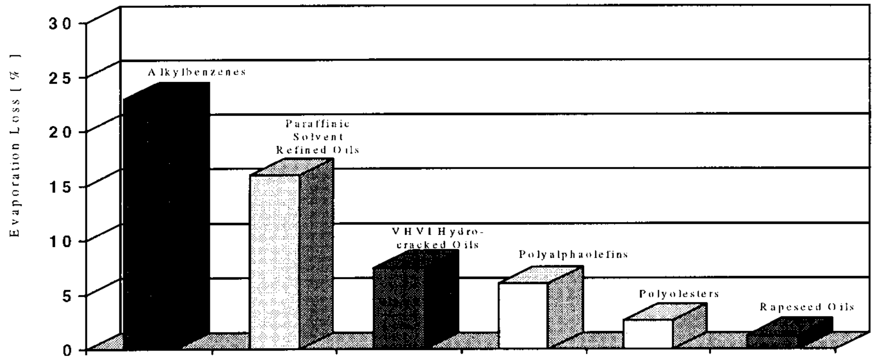


**Fig. 7.3** Dependence of evaporation loss on base-oil chemistry.

range in application but with the recommended working viscosity. Also the higher VI can lead to polymer-free multigrade lubricants with improved shear stability.

#### 7.11.5.3 **Boundary Lubrication**

Most vegetable oils, synthetic esters and glycols display excellent lubricity in boundary lubrication conditions. The high degree of polarity of these lubricants results in superiority against mineral oil based lubricants. This has been proven by a series of tests. Experimental investigations on twin disc test rigs showed that the friction coefficient of vegetable oils, synthetic esters and glycols is half that of mineral oils:

Figure 7.4 compares of a mineral oil and a TMP-ester of the same viscosity grade. The experimentally determined friction coefficients are represented depending upon the slip of the two-disk test rig. It can be stated that, for these measurements, the friction coefficient for this ester is half that of a mineral oil.

## 7.12
## Additives

This section contains a brief summary only. Further information is given in Chapter 6.

### 7.12.1
### Extreme Pressure/Antiwear Additives

Most vegetable oils and synthetic esters used in lubricants display have a high degree of polarity. This characteristic results in better lubricity than mineral oils in boundary conditions. Sulfurized fatty materials are environmentally friendly EP additives. Sulfur as an additive in esters provides an AW effect. Sulfur carriers with 15 % total sulfur and 5 % active sulfur have proven effective in rapidly biodegradable esters and vegetable oils.

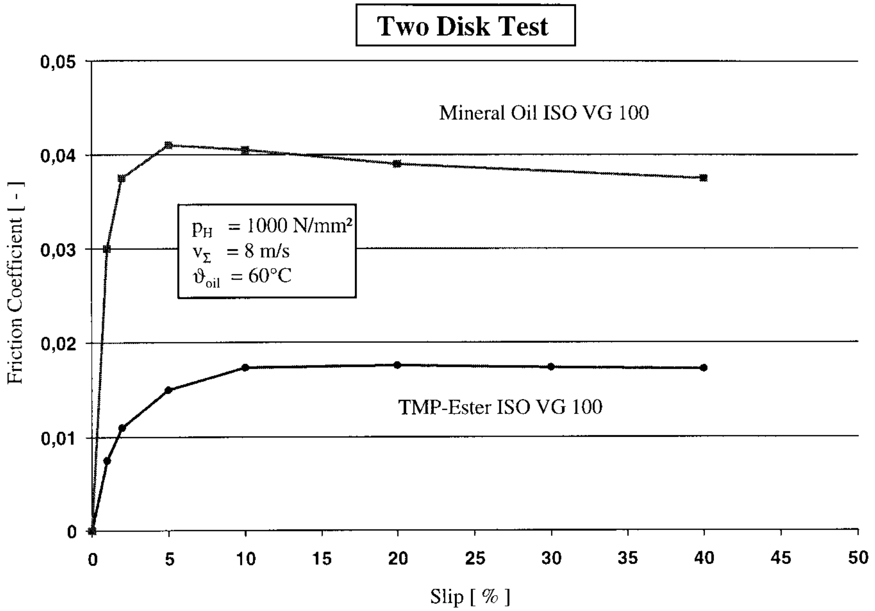


**Fig. 7.4** Friction coefficients of two base fluids as function of slip

## 7.12.2
## Corrosion Protection

Vegetable oils and synthetic esters show a high polarity. This also applies to corrosion inhibitors and can result in a competitive reaction on the metal surface.

Special calcium sulfonates, succinic acid derivatives or ashless sulfonates can be used to provide corrosion prevention.

## 7.12.3
## Antioxidants

Special phenolic and aminic materials are suitable antioxidants for the formulation of biodegradable lubricants.

## 7.13
## Products (Examples)

With some examples of environmental compatible lubricants the special advantages of ester-based products will be illustrated.

### 7.13.1
### Hydraulic Fluids

Viewed against the overall lubricants market (greases, engine oils etc.), hydraulic oils have a very large market volume. Of about 150 000 tonnes of hydraulic oils (Germany 2005), about 40 % were used in mobile and 60 % in stationary systems. In 2005, about 6 % of all hydraulic fluids were rapidly biodegradable, 88 % were mineral oil-based (HLP, HLPD etc.) and about 6 % were fire-resistant products (HFC, HFD etc.).

Especially hydraulic fluids are of great interest for biodegradable substitutes because the leakages and uncontrolled losses are well known since a long time. From a technical point of view today it should be possible to substitute 90 % of all mineral based hydraulic oils by ester based products. But there is a problem with the wide range of cost/performance ratio: Rapeseed-based, low cost hydraulic fluids only are suitable up to 70 °C tank temperature. The high performance ester oils, with four to five times higher prices, sometimes more than fulfil the requirements – the best oil for the application is to be chosen individually for each unit.

### 7.13.2
### Metal Working Oil

Metal working/cutting oils are used to reduce heat when cutting or forming metal. As well, they lubricate the cutting area, remove contaminants, and prevent corrosion. There are two broad categories of conventional metal working oils: neat and water based. Neat oils can be derived from animal, petroleum, vegetable, or synthetic sources; however, most are petroleum based. Water based or aqueous metal working oils contain water or must be mixed with water after purchase. There are three types of aqueous oils: soluble, semi-synthetic, and synthetic.

Potential consumers of metal working oils include companies involved in the manufacture of automotive parts, aerospace equipment, heavy equipment, and electronics.

Having achieved success in Europe, vegetable oil based metal working oils are now being introduced in other countries like Australia, Brazil and Canada. Because vegetable oil cutting fluids are more environmentally friendly, less of a health hazard, and superior in performance, they could become the oil of choice.

Metal working oils are sold to the end users directly from the manufacturer or through machine tool shops. Machine tool shops also sell the actual metal cutting tools. Ester based metal working oils typically sell for twice that of conventional oils.

Because much technical testing is required to determine which applications are feasible for ester based cutting oil, researching this product idea may be very time consuming and complex. In order to achieve success, the ester product would have to perform better, have a universal use, or be less expensive in an overall analysis of manufacturing cost.

### 7.13.3
### Oil-refreshing System

Because of their physical properties, refined, chemically unchanged vegetable oils can be used as alternatives to petroleum products in a large number of lubricants. Compared to mineral oils, their performance in most applications is limited by their relatively poor aging resistance. The reasons for this are the unsaturated hydrocarbon chains in the natural fatty acids and the easy hydrolysis of the ester compounds.

Vegetable oils, at present, are primarily used for total-loss applications and products with relatively low technical specifications. At face value, using rapeseed oil as a basis for engine oils does not appear very promising – the present operating conditions of engine oils cannot be satisfied by chemically unmodified vegetable oils.

Over many years the development in the lubricants market had the main aim to formulate products for 'long oil drain intervals' or for lifetime. This was not possible with vegetable oil. But today we can recognize the opposite development in some applications – the substitution of circulating oils by total-loss lubricants:

- Example 1: Minimum Quantity Lubrication in metalworking
- Example 2: Die-casting and forming instead of cutting and grinding
- Example 3: Oil-refreshing systems for lubrication of four-stroke engines.

Those ideas primarily are not motivated to use chemically unchanged vegetable oils in new applications. The main idea behind all examples is the lower overall impact of lubricants and its degradation products to environment – with total-loss lubricants.

For four stroke engines, a novel lubrication concept for diesel engines was developed which counteracts the relatively poor aging resistance of vegetable oils by continuous refreshing (Plantotronic system). This has permitted the first ever use of rapeseed and sunflower oils for engine lubrication. In this lubrication system, used oil is gradually burned along with the fuel itself without negatively influencing emission values.

The concept can be seen as a way of continuously refreshing a vegetable-based engine oil or as a way of performing a continuous oil change. Combustion in the engine was only made possible by the use of low-additive vegetable oils – burning conventional oils seems highly contentious considering the additives commonly used in such oils. The use of vegetable oils for engine lubrication depends on the progressive burning of used engine oil with the fuel. The advantage of the oil refreshing method is that chemically unmodified, low additivated vegetable oils can be used and that no waste oil has to be disposed of. Another advantage is the automated oil-change, which helps to reduce the maintenance costs especially for stationary engines (power plants).

## 7.14
## Safety Aspects of Handling Lubricants (Working Materials)

Lubricants are working materials when personnel contact with these substances via contact with the skin or clothes or by inhalation or swallowing. Toxicological, medical and industrial hygiene aspects thus have to be considered.

With regard to the protection of personnel and material goods, possible fire and explosion damage should be evaluated.

Particularly in the field of metalworking, the properties of lubricants which effect the workplace are of relevance. Metalworking lubricants have to be allocated to the group of environmental chemicals which may be released from their direct application area and spread through the environment, i.e. water, the ground or air, during or after their application. This applies in particular to the disposal of used fluids.

The Law on Working Substances significantly effects health and safety-at-work demands on the commissioning, disposal, labeling and monitoring of hazardous substances. Products which are explosive, flammable, easily ignitable, highly poisonous, slightly poisonous, corrosive or irritating are all listed.

The focal point of the Law on Working Substances is substances themselves and not mixtures such as lubricants. Mixtures are only included if they are poisonous or if they contain potentially harmful solvents in certain concentrations. Furthermore, a part of the law specifically deals with hazardous mixtures used as paints or coatings. Dangerous substances and their concentration in a hazardous working substance are defined. Certain analogies for metalworking lubricants could be drawn from this. According to this definition, mixtures with > 0.5 % pentachlorophenol or > 5 % formaldehyde (both biocides in cutting fluids) would have to be classified.

All in all, the list of hazardous substances contained in the Law on Working Substances also offers a guide for mixtures in which these substances are used.

When lubricant manufacturers develop and formulate metalworking fluids, the above-mentioned safety aspects are a high priority. However, a number of technical and economical aspects require the use of some hazardous substances. In these cases, all available measures to protect personnel must be used and every effort made to avoid personnel contact with these lubricants.

### 7.14.1
### Toxicological Terminology and Hazard Indicators

#### 7.14.1.1 Acute Toxicity

This is the toxicity of a substance after a single exposure. In animal tests, the LD50 (lethal dose) figure is given to characterize the substance. The acute LD50 is the value in grams or mg $kg^{-1}$ body weight of the animal after which 50 % of the animals die after 1 dose. In animal tests, the substance can be given orally (in the mouth), dermally (via the skin) or by inhalation. For the latter, the lethal concentration in air, LC50 is given in mg $L^{-1}$ air over a defined period of time.

#### 7.14.1.2 **Subchronic and Chronic Toxicity**

Health hazards are often only noticed after repeated exposure. Animal tests to determine these long-term effects usually last 28 days (subacute), 90 days (subchronic) and over a half-year (chronic).

The complexity of the individual tests varies greatly. Determining subacute toxicity costs about ten times more, and chronic toxicity about 100 times more than the cost of an acute toxicity test.

#### 7.14.1.3 **Poison Categories**

The German Law on Chemicals lists three poison categories for substances and mixtures; the $LD_{50}$ values for rats are listed below (Table 7.13).

**Tab. 7.13** German Law on Chemicals LD50 values for rats.

| | *Highly poisonous* | *Poisonous* | *Slightly poisonous* |
|---|---|---|---|
| Oral LD50 (mg $kg^{-1}$) | ≤ 25 | 25–200 | 200–2000 |
| Dermal LD50 (mg $kg^{-1}$) | ≤ 50 | 50–400 | 400–2000 |
| LC50, inhaled (mg $L^{-1}$ in 4 h) | ≤ 0.5 | 0.5–2 | 2–20 |

Some countries use a larger number of poison categories. At one extreme, oral LD50 can be maximum 5 mg $kg^{-1}$ to 15 g $kg^{-1}$.

The vast majority of lubricants used in metalworking do not fall into these poison categories. Most products have an acute oral $LD_{50}$ of > 10 g $kg^{-1}$ and an acute dermal $LD_{50}$ of > 2 g $kg^{-1}$. Most additives such as EP agents, polar additives and emulsifiers are also in this range. In a few cases, additive values below 2 g $kg^{-1}$ are found (some biocides). When these substances are used, their concentration in the finished product must be considered. The large dilution factor in water-miscible products is a case in point. Water-miscible concentrates must be evaluated differently to water-miscible finished products.

#### 7.14.1.4 **Corrosive, Caustic**

Substances are corrosive if 0.5 g or 0.5 mL of the substance in contact with the skin of a rabbit in defined test conditions leads to its destruction after 30 min.

Substances are caustic if they cause inflammation under the above-mentioned conditions.

#### 7.14.1.5 **Explosion and Flammability**

This applies in particular to the flammable substances or fluids with flashpoints between 21 and 55 °C. This applies to lubricants if, as an application aid, they contain a Hazard Category AII white spirit. Substances which the German Law on Chemicals defines as being explosive, flammable or easily ignitable are generally not used in metalworking fluids.

#### 7.14.1.6 **Carcinogenic**

A recurring topic in the field of metalworking lubricants is carcinogenic effect. In recent years, this was the case with some corrosion inhibitors, especially $NaNO_2$ together with amines (nitrosamines) and with polycyclic aromatic hydrocarbons.

#### 7.14.1.7 **Teratogens, Mutagens**

These are chemicals which produce malfunctions to human life. When these alter genetic structures, these are called mutagens.

### 7.14.2

### MAK (Maximum Workplace Concentration) Values

This is the maximum permissible workplace concentration for working substances in the form of gas, vapor or suspensions which are found in the air. These maximum permissible values are contained in a list of hazardous substances and this list is constantly being expanded. However, it is accepted that long-term exposure to the maximum concentrations does not pose any health risks.

As regards metalworking lubricants, it should be noted that some contain solvents. Table 7.14 shows the MAK values for the most commonly used solvents in metalworking fluids. Evaporation tendency which is characterized as vapor pressure (in mbar) or as an evaporation figure (see DIN 53170) is also of interest to the user. Solvents with low MAK values and high evaporation tendencies are particularly critical.

**Tab. 7.14** Solvent Data

| *MAK value (ppm)* | *Vapor pressure at 20 °C (mbar)* | *mg $m^{-3}$* | *Boiling point or range (°C)* | *Evaporation figure DIN 53170* |
|---:|---:|---:|---:|---:|
| (500) | 6 | (2000) | 155–185 | 60 |
| (500) | 1 | (2000) | 180–210 | 165 |
| 50 | 77 | 260 | 87 | 3.8 |
| 200 | 133 | 1080 | 74 | 3.0 |
| 100 | 19 | 670 | 121 | 9.5 |
| 200 | 453 | 720 | 40 | 1.8 |
| 1000 | 889 | 5600 | 24 | 1.0 |
| 1000 | 380 | 7600 | 47 | 1.3 |

Apart from the MAK values, substances for which no toxicological or health and safety criteria exist to determine a MAK value, are given a TRK (Technical guideline Concentration) value.

A number of carcinogenic substances are included in the TRK list. A list has also been published for the components and concentrations thereof in cutting fluids.

The subject of permissible oil mist concentration is constantly being discussed, in particular in connection with neat cutting oils (see Chapter 14). Oil mist as a dispersed phase must be treated differently to gas-like oil part. Based on the US

Threshold Limit Values (TLV) for oil mists, a MAK value of 5 mg $m^{-3}$ and total mist and vapor of 20 mg $m^{-3}$ is often proposed. The TLV value of 5 mg $m^{-3}$ is based on a toxicologically barely relevant white oil with no additives.

### 7.14.3
### Polycyclic Aromatic Hydrocarbons (PAK, PAH, PCA)

The largest proportion of raw materials in metalworking lubricants are high boiling point petroleum cuts which contain very few polycyclic aromatics (PAH = polycyclic aromatic hydrocarbons, toxicologically relevant aromatics). However, it is already known that in the area of aromatic hydrocarbons with 4 to 6 aromatic rings, some high carcinogenic substances exist. Benzo(*a*)pyrene (BaP, 1,2-benzopyrene, formerly 3,4-benzopyrene) was identified as such. This is often the key substance used to characterize the risk of environmental carcinogenicity of this group of substances.

While mineral oil hydrocarbons were evaluated for PAH content in the past by total aromatic content or UV adsorption in some areas, these days considerably analytical effort is used to quantitatively identify groups (such as isomers) or individual chemical components with known carcinogenic characteristics. In this field, special enriching processes with gas chromatographic identification have gained acceptance (e.g. Grimmer aromatics).

Neat metalworking oils are in the forefront of the PAH discussion because these can directly contact a comparatively large number of people via the inhalation of oil mists. Efforts were already made in the past to evaluate the cancer risks posed by neat cutting oils by determining their concentration of polycyclic aromatic hydrocarbons.

In recent times, precise analytical tests similar to the Grimmer method were performed on fresh and used neat cutting oils. These also determined PAH contents in air based on 5 mg oil mist per cubic meter air. Values were recorded for benzo(*a*)pyrene (BaP) which were below or at the average value measured in various locations (e.g. 0.02 mg $m^{-3}$). The increase in PAH content with cutting or grinding oil use can be dramatic but the absolute concentration still remains in a comparatively insignificant area. The conclusion drawn from this is that the cancer risks posed by polycyclic aromatic hydrocarbons in neat cutting oils is generally overestimated. And finally, new refining methods, better workplace hygiene and a reduction in oil misting will further lower potential risks.

### 7.14.4
### Nitrosamines in Cutting Fluids

Discussions on this subject were triggered by the use of alkali metal nitrates as corrosion inhibitors. The most prominent was and is sodium nitrite, a widely available and cheap substance with good inhibitor properties. In certain circumstances, the effect of amines on nitrites can create nitrosamines. It must be noted that 80 % of nitrosamines are carcinogenic. Sodium nitrite ($NaNO_2$) can be found, above all, in concentrations of low-mineral oil (semi-synthetic) or hydrocarbon-free (fully-synthetic) solutions. These concentrations are so high that water-miscible application concentrations of 0.05 to 0.2 % can be found. If other, mostly organic inhibitors are used, significantly lower concentrations can be used. Apart from the use of sodium nitrite in water-miscible cutting fluid concentrates, it can also be directly added to water-miscible cutting fluids by consumers.

A particular problem is the bonding of amines with nitrites in water-miscible cutting fluids and especially the combination of sodium nitrite and alkanolamines. Nitrosamines are the general term for *N*-nitroso compounds with the typical N–NO structures. They are created primarily by the reaction of nitric acid and its salts (nitrites) on secondary amines (e.g. diethanolamines). *N*-Nitroso compounds are created in the acidic area with primary, secondary and tertiary amines but only secondary amines are stabilized to form nitrosamines. An *N*-nitroso agent is the anhydride of nitric acid. The reaction runs as follows:

While the carcinogenic effect of some nitrosamines has been known since the mid-1950s, it was assumed that the reaction between nitrite and amines could not take place in cutting fluids because these were alkaline and the reaction requires an acidic environment. If cutting fluid mists are swallowed, nitrosamines can be formed in the acid areas of the stomach. This was proven with a grinding fluid along with sodium nitrite and diethanolamine. The reaction was also performed in a human stomach with a non-carcinogenic nitrosamine. Interestingly, the formation of nitrosamines was also proven in the alkaline area but the catalytic effects of contaminants and the biochemical reactions of bacteria may have been the cause.

High-performance liquid chromatography (HPLC) and other analytical methods were used to prove the presence of between 0.02 and 2.99 % of diethanol nitrosamine in cutting fluid concentrates of fully-synthetic products. The pH of the products was between 9 and 11.

Although nitrosodiethanolamine is the most commonly found nitrosamine in cutting fluids, the other types should not be ignored. It should be noted, for example, that the carcinogenic effect of diethanolnitrosamine is 200 times less than that of diethylnitrosamine.

The nitrosamine discussions have led a number of cutting fluid manufacturers to develop nitrite-free cutting fluids. However, it must be remembered that in the past, a much larger proportion of water-miscible cutting fluids were free of nitrite.

### 7.14.5
### Law on Flammable Fluids

The German law on the installation and operation of plants to store, fill and transport flammable fluids (Law on Flammable Fluids) does not directly effect the application of lubricants containing flammable solvents. However, the corresponding technical guidelines offer a series of practical tips concerning the handling of flammable fluids. The Law on Flammable Fluids allocated flammable fluids into two Groups, Group A (neat) and Group B (water-miscible). For the flammable solvents in metalworking lubricants, these are mainly Hazard Category A II (Flashpoint 21–55 °C) and A III (Flashpoint 55–100 °C) white spirits. Exceptions apply to lubricants with high consistencies and with low amounts of solvents.

The a.m. technical guidelines deal with the demands on containers and their labeling (from Hazard Category A II). These technical guidelines also define hazardous conditions.

As to the application of lubricants containing solvents, advice offered by professional associations should be consulted. Similar advice has been published for the application of solvent-based paints.

Regarding electrical standards, special guidelines apply when lubricants containing flammable solvents are applied. In Germany, these are VDE 0165, 0170, 0171 and 0100. Apart from the flashpoint itself, any possible heating during application must also be considered.

## 7.15
## Skin Problems Caused by Lubricants

Skin problems are second only to noise-related hearing problems according to a 1978 report by the German Association of Professional Associations. In the metalworking industry, a large proportion of the problems are caused by contact with lubricants. Chip-forming machining operations with water-miscible and neat cutting oils are in the forefront because of the large number of contact possibilities.

### 7.15.1
### Structure and Function of the Skin

With an area of about two square meters, the skin is the largest human organ. It serves as the final barrier between the body and its environment. The outer skin should protect against external effects such as radiation, heat, cold and dryness at the same time as keeping vital substances within the body. In spite of these important protective functions, it is very thin with a thickness of less than one millimeter. The outer skin is constantly being regenerated by the production of new cells. Dead skin cells, sebum and perspiration form an additional barrier, the protection layer. Its acid nature protects the skin from diseases.Its acid nature protects the skin from diseases.

The skin also offers the body protection against thermal, mechanical and chemical attack. The skin thus also plays an important role in the our working lives. The most important and most common skin damage is to the outer layers of the skin, the epidermis. Under this lies the dermis (corium) and the subcutis. The epidermis consists of three parts, the corneal layer, the basal or germinative layer and the prickle cell layer.

As long as the corneal layer is intact, i.e. elastic and smooth with no cracks, all deeper layers of the skin are protected from harmful influences. The skin's fats play an important role, at the outer surface it has a direct protection role and deeper down it keeps the corneal layer supple and stops the skin drying out by regulating the skin's moisture. Degreasing substances such as low-molecular-weight petroleum cuts and also solvents can destroy the surface oil and the skin's oil (lipids) in the walls of the cells. The result is a drying-out of the skin with cracks in the skin and the loss of the upper corneal layer cells. In this state, the ingress of harmful substances is relatively easy. Degreasing substances can also be absorbed through the tallow glands (follicles).

A further skin defense mechanism is the ability of the barrier layer to partly neutralize alkaline and acidic substances with certain amphoteric amino compounds. The skin's acidic barrier can also be seriously damaged by the ingress of alkaline substances.

### 7.15.2
### Skin Damage

For ordinary people, the nomenclature of skin damage is difficult to access because genesis terminology mixes with appearance terminology. Particularly regarding water-miscible and neat cutting fluids, apart from the influence of the fluid itself, a number of possible harmful effects can occur which interact and multiply. An example of this is the mechanical damage caused by the workpiece or the chips which are transported by the cutting fluid. The appearance of skin eczema often makes an allocation to genesis difficult and an allergic eczema cannot be differentiated from a degenerative eczema.

#### 7.15.2.1 Oil Acne (Particle Acne)

This is one of the most common skin problems caused by contact with neat cutting and grinding oils. Usually it is not the oil itself but small particles such as metal fragments which are the cause. This is why this problem is often referred to as particle acne. Oil acne can appear wherever oil directly contacts the skin and this can include oil-stained clothing. Body areas particularly at risk are the lower and upper arms, the backs of hands, the face, thighs and waist.

The drying-out of the skin is normally accompanied by the blocking of tallow glands (blackheads, spots). If these become infected, they form pussy blisters. If a number of these pussy infections join together, a large pyodermous is formed. Particularly at risk are people with Seborrhoea with greasy skin. Such people should be kept well away from wet metalworking operations where exposure to oil cannot be avoided.

Improvements in machine tools such as machine encapsulation, fume extraction, the avoidance of oil mists and general automation means that personnel have less contact with oil and the cases of oil acne have fallen significantly in recent years. The frequency of oil acne cases in factories is a measure of the hygiene standards in force and the personal cleanliness of machine room personnel.

The persistent occurrence of oil acne may also be the result of certain substances in the oil. In many cases, these are EP additive substances. A specific form of this problem is chlorine acne which can be triggered by chlorine compounds in the oil. It has been shown that some chlorinated aromatics, but not chloroparaffin, can also cause this type of acne.

#### 7.15.2.2 Oil Eczema

The term ‘oil eczema’ encompasses several skin problems. These look like scaly or wet reddened areas, sometimes with a cracked surface.

Acute toxic eczema is caused by the direct effect of the substance on the skin and causes a particular appearance.

Degenerative eczema is the most important skin problem caused by water-miscible cutting fluids. Long-term contact between the cutting fluid and the skin produces signs of degeneration and a breakdown of the skin’s defenses. Key factors are the alkalinity of the fluid, the long-term wetness and specific product ingredients and particularly boundary-active substances from emulsifiers. On the subject of alkalinity, there are a number of evaluations of acute alkalinity (pH value) or potential alkalinity (reserve alkalinity). However, there is a consensus that pH values of over 9.0 (9.5) accelerate degenerative eczemas following alkaline damage to the skin. It must be noted that it is disproportionately difficult to develop and manufacture stable emulsions with a pH of 8.5 instead of 10.0.

In microbiological eczemas, pathogenic and apathogenic germs play a part. There are considerable differences in opinion concerning the significance of microbiological eczemas in metalworking. One question is whether the large number of germs in water-miscible cutting fluids promote this type of eczema. Some experience indicates that no hygienic risk is posed by germ numbers of $10^5$ or $10^6$ $mL^{-1}$ if no particularly pathogenic germs are present.

Degenerative eczemas and degenerative dermatitis are faced by facultative, allergic eczemas. In these cases, the person is oversensitive to one or more substances. Such typical allergies must be kept away from allergens. Apart from this highly individual form of allergic eczema which effects some people, long contact with a larger group of people can also cause oversensitivity and ultimately to an allergic eczema. While degenerative dermatitis can be combated with skin protection and other measures, oversensitivity can occur wherever the allergen contacts the skin (contact dermatitis). Oversensitivity can last a number of years or even for life so that the allergen must either be removed from the process or the person in question has to be transferred to other work.

Long-term degeneration, for example, caused by exposure to alkalis in water-miscible cutting fluids can ease the ingress of allergens into the skin and thus start the oversensitivity process.

Allergic skin diseases are relatively seldom in metalworking companies. However, the increasing number of chemically active substances in cutting fluids may increase the oversensitivity of skin. Chemically active means suspiciously allergic to dermatologists. Such suspiciously allergic substances are often found in biocides, EP additives and corrosion inhibitors. Among the biocides, formaldehyde and formaldehyde-splitting substances are viewed as potential allergens. However, free formaldehyde is often overestimated as an allergen.

Apart from the contents of the cutting fluid, contaminants and particles can often act as allergens. An example is chrome from chrome workpieces and tools. Chrome suspended in the cutting fluid is less dangerous than chrome solutions with their oil-soluble, lipolytic compounds.

In general, it can be assumed that cutting fluid manufacturers together with skin specialists select product formulations which keep the risk of acute toxic or allergic eczemas to an absolute minimum. Furthermore, the increasing automation of metalworking processes means that operating personnel have less and less contact with such fluids.

### 7.15.3
### Testing Skin Compatibility

Preliminary tests on additives or finished metalworking fluids can be performed on animals. A group of tests involve the substance being applied to the shaved skin of animals at defined intervals. For example, one test involves the substance being applied 24 times to 10 mice for a period of 8 weeks. The substance can also be tested for compatibility if 0.05 mL is injected into the underbody skin of white mice (Intracutan method).

Most commonly, cutting fluids or their components are tested for skin compatibility on persons because the direct transfer of animal results to humans is not possible. In such cases, preliminary animal tests as described above are still performed.

Several skin tests are used to determine oversensitivity to potentially allergic substances. These tests include the rubbing test, prick test, scratch test, intercutan test and the epicutan test. These tests are practically painless. A positive test reaction in the case of the rubbing, prick, scratch or intracutan tests (in the case of an immediate allergic reaction) results in an itching rash similar to that caused by nettles. Such a rash takes a few minutes and up to half an hour to develop. The epicutan test serves to determine eczemic reactions. If the test is positive, the test area reddens and blisters or spots appear. In these tests, the skin reacts very slowly so that results can only be drawn after two or three days.

**Rubbing Test:** An allergen is applied to the skin but repeatedly rubbing it on the forearm skin. This test serves as a sort of preliminary test of high sensitivity.

**Prick Test:** Standard allergen solutions are applied to the upper layers of the forearm skin by lightly pricking the skin with a pricking needle. Anti-allergic medication should not be taken less than three to five days before the test because this can influence the result. This is the most common method of testing inhaled or foodstuff allergens.

**Scratch Test:** The skin is lightly scratched and the allergen is applied to the scratched area. This test is more sensitive than the previous prick test. This test is suitable for the evaluation of medicines.

**Intracutan Test:** This test is also considerably more sensitive than the prick test. The test substance is injected with a 1 mL syringe into the upper skin. This test is ideal for testing medicine allergies.

**Epicutan Test:** The allergens, mixed with Vaseline and in small receptacles, are placed on the skin and held in place with a plaster. During the test which normally lasts four days, the patient must not shower or bathe, abstain from sport and stay out of the sun. The test is ideal for determining allergic contact eczemas. When modified with light, the test serves to examine photo-allergic reactions.

In the epicutan test, cloths soaked with the test fluid are placed on attacked areas of the skin and covered with non-waterproof materials for 24 h. The concentration of the test fluid is higher than normally used to artificially induced skin irritation. The test area is them examined for skin irritations. The degree of skin irritation is evaluated by various criteria and a scale of points which are based on a comparison of test fluids and a standard reference substance.

The results are again read after 48 h, 72 h and after 96 h. In the case of water-miscible products, the normal application concentration is used but it can also be tested at a uniform 1 %. When biocides are tested, the substance is often tested at the application concentration together with water and compared to the water-miscible cutting fluid. Epicutan tests are normally performed on 40 to 80 persons. People with skin problems are more sensitive than people with health skin. This test also identifies individual allergic reactions but the method provides no information on the sensitizing effect of the test fluid.

For some critical groups of substances such as biocides, mucus tissue compatibility tests are performed in addition to the above-mentioned skin compatibility tests. This is performed by injecting the substance into the conjunctiva bag of a rabbit's eye. After 30 s the substance is flushed out and the reaction is evaluated.

**Blood Tests:** When allergens are in the body, antibodies (specific body defenses) are formed in the blood. Many other substances such as proteins are formed when atypical dermatitis or a bronchial asthma occurs. Other methods are available to isolate immune cells in the blood and to measure their sensitivity to allergens.

**Total IgE:** All immune globulin E antibodies in the blood are determined.

**Specific IgE:** A number of immunological tests can be used to determine the concentration of specific IgE antibodies which attack allergenic substances. Although specific IgE is easier to determine than total IgE, it does not always cause allergic symptoms in patients. The interpretation of the results requires a great deal of medical experience.

**Leucotriene Simulation Test:** The rate at which these are released is determined. After the white blood corpuscles have been isolated, the reaction to allergens can be measured.

**Histamine Release Test:** Similarly to the Leucotriene test, white blood corpuscles are isolated and an allergen is added. In this case, the histamine released is measured.

**Basophohile – Degranulation Test:** All the blood is exposed to an allergen and certain blood cells, the basophiles, are examined with a microscope.

### 7.15.4
### Skin Function Tests

Skin damage can be evaluated with physicochemical apparatus or chemicals alone. Skin function tests can be used to diagnose an illness as well as testing the effectiveness and compatibility of dermatological products and care products.

**Transepidermal Water Loss (TEWL):** If the upper reaches of the skin are damaged, the barrier function of the skin no longer works and the body loses water. Damage to the skin's barrier function can be measured in a simulated transepidermal water loss (TEWL) test. A sensor directly above the skin measures the amount of water which is released through the skin and evaporated (evapormetrics).

After removal of the blister roof, there is a strong increase in water loss as a result of the damaged barrier. It is generally accepted that the TEWL is well correlated with the degree of epidermal damage. The highest TEWL values are measured in fresh wounds with a continual decline until values for intact skin are reached at the end of healing. Therefore the measurement of TEWL is a suitable parameter to determine the degree of re-epithelization. The critical phases of epithelial regeneration already occur during the first days following experimental wound induction.

Normally the Duhring–Kammer test is used. The randomized, double-blind, intra-individual comparison of treatment fields (10 to 40 healthy volunteers) should include a placebo and a field treated with NaCl or Ringer's solution. The Duhring–Kammer test measurements are made over a five-day treatment period following the settling of the blisters.

The physical basis of the measurement of TEWL is the diffusion law discovered by Adolf Fick in 1855. The diffusion flow $dm/dt$ (with $m =$ water transported, $t =$ time) indicates the mass per cm$^2$ being transported in a period of time. It is proportional to the area (surface) and the change of concentration per distance $dc/dx$ (with $x =$ distance from skin surface to point of measurement). The constant factor $D$ is the diffusion coefficient of water vapor in air.

This law is only valid within a homogeneous diffusion zone which is approximately formed by a hollow cylinder. The resulting density gradient is measured indirectly by two pairs of sensors (temperature and relative humidity).

**Skin Elasticity:** The ability of the skin to regain its shape is evaluated with visual methods.

**Blood Circulation in the Skin:** The flow of blood through the skin can be measured with a laser and optical detectors.

**Skin Thickness:** The thickness of the skin can be measured ultrasonically and the special structure of the skin can be illustrated in the same way.

**Skin Color:** The color spectrum of the skin can be measured with colorimetric analysis.

**Skin Moisture:** Moisture can be determined by measuring the electrical resistance of the skin.

**Skin Oiliness:** The oiliness of the skin can be visually evaluated.

**pH of the Skin:** Upper skin pH can be measured with surface electrodes.

**Alkali Resistance Test:** Caustic soda can be used as an irritant and can various methods are available to determine damaged corneal layer.

**In vitro method according to the BUS model:** The in vitro isolated perfused bovine udder skin (BUS) model was developed for use in pharmaceutical research and is used in the cosmetics and chemical industries for testing efficacy and safety. The cow udders are obtained from slaughterhouses. After pretreatment they are cleaned carefully and shaved in the laboratory. The test substances are applied, after approximately 1 h of aerobic adaptation of the metabolism, by perfusion with oxygenated, warmed Tyrode's solution. The continuous perfusion keeps the udder, including the skin, viable for more than 8 h, during which the horny layer barrier and the skin metabolism remain active. Other advantages include the large application surface (up to 400 cm$^2$ per udder side), on which numerous product tests can be conducted comparatively (e.g. fresh and used process chemicals) and cost-efficiently. The hirsute skin on the side of the udder is histologically and functionally similar to human skin.

### 7.15.5
### Skin Care and Skin Protection

Cleaning the skin is an important aspect of hygiene practices in the metalworking industry. The cleaners used must be matched to the type of dirt involved. Under no circumstances should cutting fluids be used to clean the skin. Although water-miscible fluids or low-viscosity honing or grinding oils may clean skin effectively, the metal chips, abrasive grinding debris or other contaminants suspended in the fluid can cause considerable damage to the skin. The best cleaners are emulsions which are either slightly alkaline, neutral or slightly acidic.

Dirt which is embedded in the corneal layer can only be removed physically. These should not be so aggressive that they damage the skin (e.g. pumice, sand). Ideal are certain types of sawdust.

Cleaning the skin always means that some of the skin's oils are lost. This is the function of skin care which should allow the skin to regenerate during work breaks and above all, replace the dissolved lipophilic components. All skin care products should be capable of penetrating the corneal layer.

Skin protection products should be used to shield the skin from harmful substances. They must anchor well to the corneal layer and should not be easily dissolved by cutting fluids (they should resist washing-off for at least half a working shift). Skin protection products should not hinder the work process (by making things slippery) or by interfering with subsequent process (e.g. silicones). They should be easy to apply and easy to wash-off. As regards the principal function of skin protection emulsions, water-in-oil emulsions can best protect against water-soluble substances in water-miscible cutting fluids. On the other hand, oil-in-water emulsions offer the best protection against harmful oil-soluble substances.

# 8
# Disposal of Used Lubricating Oils

*Theo Mang*

Figure 8.1 shows what happens to the lubricants sold every year in Western Europe. Only 49 % are collectable and only 28 % are actually collected [8.1]. The chart also includes process oils which are not lubricants . It is possible to identify the following objectives from these figures: the intensive gathering of collectable oils and an improvement in the environmental compatibility of the lubricants, of which more than 50 % pollute the environment by way of total-loss applications, leaks, evaporation and other routes.

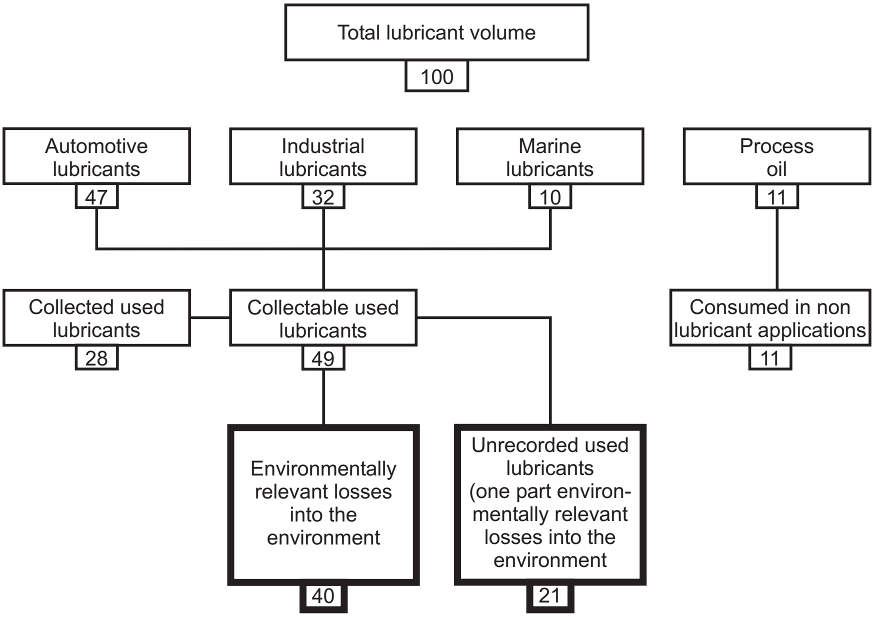


**Fig. 8.1** Lubricating oil supply, use and disposal in Western Europe (CONCAWE 1996) [8.1]

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## 8.1
## Possible Uses of Waste Oil

Used lubricants represent a problem for the environment. Their ecologically-compatible use is therefore an important environmental protection measure.

Used lubricants are created when all mechanical possibilities in a machine or at the user's premises no longer suffice to maintain the performance of the lubricant and especially when chemical additives have been used-up and aging by-products are present in the oil. The demand that new lubricants should be made of used products is based on the erroneous notion that re-refining can restore the original condition of a lubricant. In fact, lubricants lose value during use and re-refining, at best, can only restore the value of base oil. In the case of conventional mineral oils, this value is only slightly higher than fuels or heating oils. This is also the reason why re-refining is hardly economical without legislative provisions or subsidies. From a global competition point of view, other disposal options include the direct incineration of untreated waste oils, the simple pre-treatment (cleaning) and alternative uses such as flux oils for bitumen or for the manufacture of secondary feeds in sec-feed plants for catalytic crackers and as blending stock for high-sulfur fuels.

The different possibilities of regeneration, recycling, and waste-to-energy application of used lubricants has been illustrated by Krishna (2006) for an integrated steel plant as an important consumer of lubricants [8.11].

## 8.2
## Legislative Influences on Waste Oil Collection and Reconditioning

The EC Directive 87/101 contains a recommendation to all member countries concerning the regeneration of used oils insofar as economic, technical and organizational conditions allow. Emission thresholds for incineration plants (<3 MW) make the burning of untreated used oils difficult. However, in some European countries, incineration in smaller incineration plants is still possible. Legislation permits incineration in high-temperature furnaces and by the cement manufacturing industry. In some countries, fuels and heating oils reclaimed from used lubricants are not taxed and are thus subsidized.

The PCB problem which surfaced in Europe in 1983 significantly influenced European legislation on wastes. Polychlorinated biphenyls (PCB) which enter the re-refined oil chain as fire-resistant hydraulic oils or condenser oils, have changed German waste oil legislation. While the PCB problem has practically disappeared, the division of waste oils into two groups has have considerable consequences. Used oils which contain more than 0.2 % chlorine cannot be re-refined and are subject to expensive disposal procedures. This in turn has promoted the development of chlorine-free lubricants.

Re-refined used oils are subsidized in Italy. In Germany, the manufacturers of lubricants (including distributors) have transferred their legal requirement to properly dispose of waste oils to collection organizations. In 1999, these received about US$ 90 per tonne from the lubricant consumers.

In the USA, state law on this subject differs. Since 1986, used oils have been classified as hazardous wastes in California and other states have since followed. In some states, the collector is paid up to 20 cents per gallon by the oil user, in other states the collector has to pay.

Viewed globally, some extremely differing situations exist. While some countries do not regulate the collection and disposal of used oil and used oil is generally not collected, other countries can point to high collection and disposal rates (in 1996, 99 % of used oils were collected or properly incinerated in Germany; but only 60 % in the USA).

## 8.3
## Re-refining

The re-refining of used oils to lube base oils started in 1935 [8.2]. The principal reasons why re-refining was unable to find acceptance were: high process costs and therefore high selling prices compared to relatively low virgin oil prices, in inadequate removal of carcinogenic polycyclic aromatics, the negative image of such oils in most markets and the increasing complexity of base oil blends in engine and other lubricants. In Western Europe, only 7 % of base oil demand was satisfied by re-refined products in 1998.

Numerous re-refining technologies have been developed over the last 20 years. Many were patented but only few were suitable for large-scale application [8.3–8.8].

In general, the process stages shown in Table 8.1 are common to all the different methods

**Tab. 8.1** General process stages for re-refining of used oils.

| | |
|---|---|
| 1 | Separation of larger solid impurities along with most of the water. This is normally achieved by sedimentation |
| 2 | Separation of the volatile parts (fuel residues in engine oils, solvents and low boiling-point lubricant components). This normally happens by atmospheric distillation. The separated light hydrocarbons can usually be used in-house for energy creation. |
| 3 | Separation of the additives and aging by-products. This can occur by acid refining, solvent (propane) extraction, vacuum distillation or partly also by hydrogenation. |
| 4 | Finishing process to separate any remaining additives, aging by-products and refining reaction products. This normally happens by hydrofinishing, with absorbents such as bleaching clay or mild, selective solvent extraction (e.g. Furfural). |

### 8.3.1
### Sulfuric Acid Refining (Meinken)

The sulfuric acid refining process was mostly developed by Meinken. Compared to older acid-based methods, various process stages reduce the amount of acidic sludge and used bleaching clay generated as well as increasing the lube oil yield.

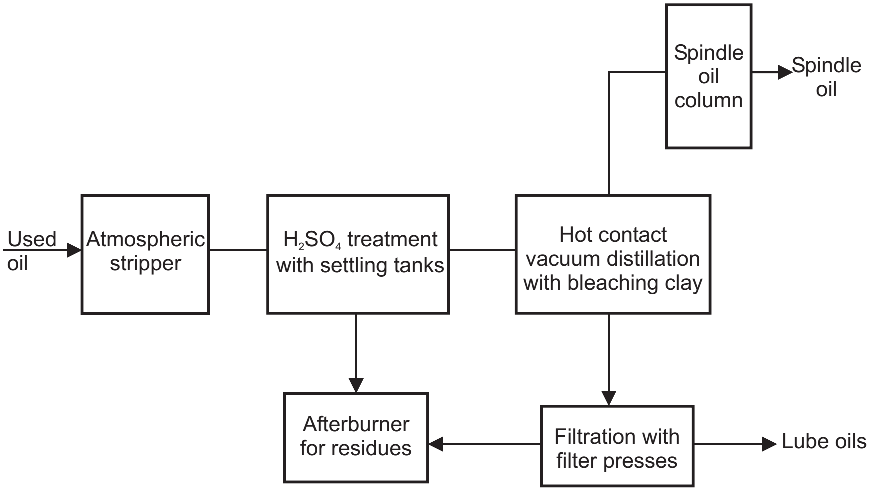

**Fig. 8.2** Sulfuric acid re-refining (flow chart of the Meinken process) [8.9]

Due to the acidic sludge problem, acid refining has largely been replaced by other methods. However, numerous such plants were still in operation in 1999. Figure 8.2 shows a Meinken flow plan.

### 8.3.2
### Propane Extraction Process (IFP, Snamprogetti)

Of the principal extractive refining processes, the IFP (Institut Français de Pétrole) technology is worth mentioning. This initially used propane extraction together with acid refining and later together with hydrofinishing. Propane extraction is also used by Snamprogetti (Italy) as the main refining step before and after vacuum distillation. Figure 8.3 shows the process with propane extraction [8.9, 8.10].

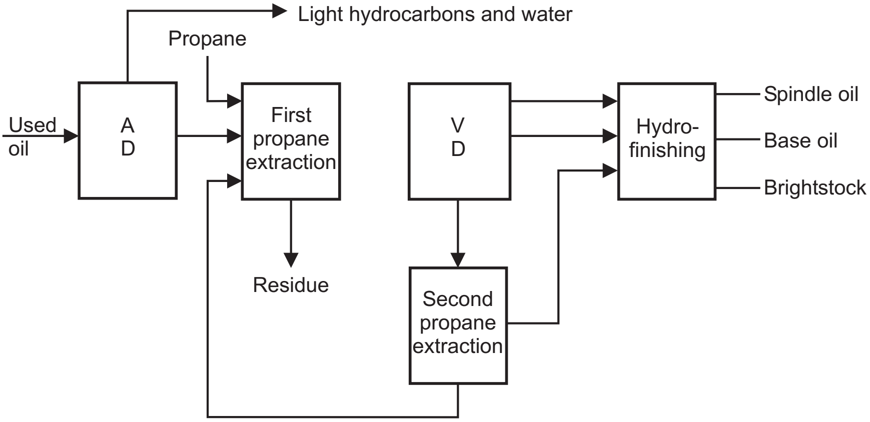

**Fig. 8.3** Re-refining by propane extraction (IFP, Snamprogetti).

### 8.3.3
### Mohawk Technology (CEP–Mohawk)

The Mohawk Process (subsequently CEP–Mohawk) using high pressure hydrogenating was introduced in the USA at the end of the eighties. The process begins with thin-film vacuum distillation (after flashing the light hydrocarbons and water). This is followed by hydrogenation of the distillate at 1000 psi over a standard catalyst. Special steps realized catalyst life of 8 to 12 months, which was essential for the economy of the process

A marked reduction in the amount of water which must be treated as effluent as well as the cheaper materials for construction (absence of corrosion) are further advantages. The Mohawk process which is based on the KTI process has been licensed for Evergreen Oil (USA and Canada).

### 8.3.4
### KTI Process

The KTI (Kinetics Technology International) process combines vacuum distillation and hydrofinishing to remove most of the contamination and additives. The key to the process is the thin-film vacuum distillation to minimize thermal stress through mild temperatures not exceeding 250 °C.

The hydrofinisher removes sulfur, nitrogen and oxygen. The yield of finished base oils is high (82 % on a dry waste oil basis). Figure 8.4 shows the flow chart of this process.

### 8.3.5
### PROP Process

PROP technology was developed by Phillips Petroleum Company. The key elements of the process are the chemical demetalization (mixing an aqueous solution of diammonium phosphate with heated base oils) and a hydrogenation process. A bed of clay is used to adsorb the remaining traces of contaminants to avoid poisoning of the Ni/Mo catalyst.

Figure 8.5 shows the PROP Process.

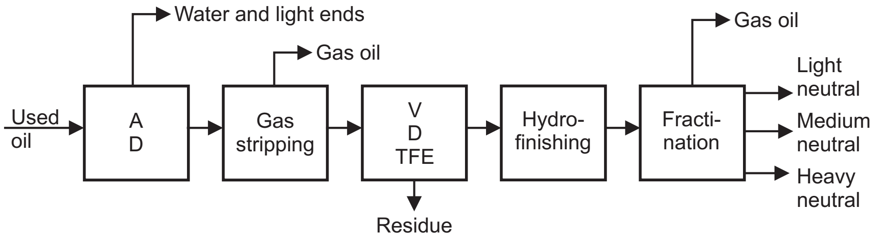

**Fig. 8.4** Flow chart of the KTI Process: thin film evaporator (TFE) with hydrotreatment.

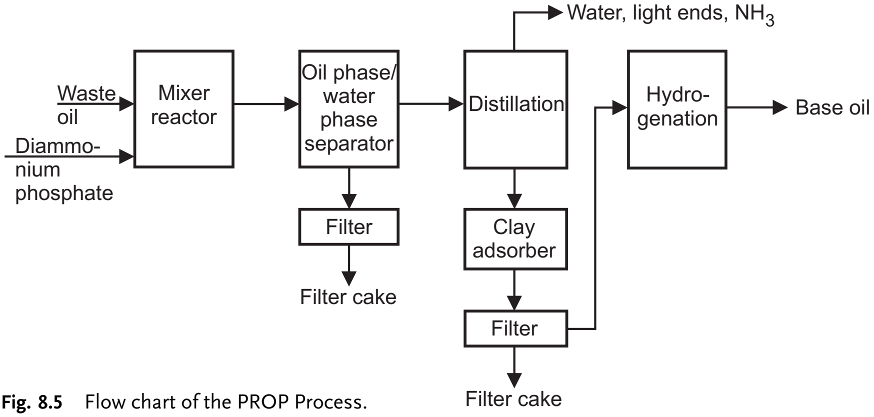

**Fig. 8.5** Flow chart of the PROP Process.

### 8.3.6
### Safety Kleen Process

This process uses atmospheric flash for removing water and solvents, a vacuum fuel stripper, vacuum distillation with two thin-film evaporators, hydrotreater with fixed bed Ni/Mo catalysts. When using high severity the hydrotreater is in the position to reduce polynuclear aromatics; it also removes higher boiling chlorinated paraffins.

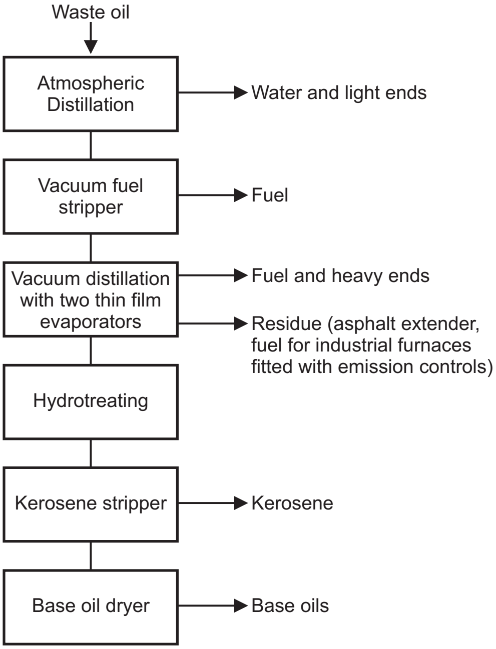

**Fig. 8.6** The Safety Kleen process.

Figure 8.6 shows a simplified block diagram. In 1998 the Safety Kleen process was used in the largest waste oil re-refinery in the world (East Chicago, Indiana, USA, plant capacity 250 000 t $y^{-1}$).

### 8.3.7
### DEA Technology

The best results with regard to the technical and environmental quality of the re-refined oil and the elimination of PAH are provided by a combination of thin film distillation followed by selective solvent extraction. In this process, the distillate from vacuum thin-film distillation towers equipment at the re-refinery (Dollbergen/ Germany) are finally treated in a lube refinery solvent extraction plant followed by hydrofinishing (DEA, Hamburg/Germany). After this extraction process, the PAH content is lower than that of virgin solvent neutrals. Figure 8.7 shows the corresponding flow-chart [8.10].

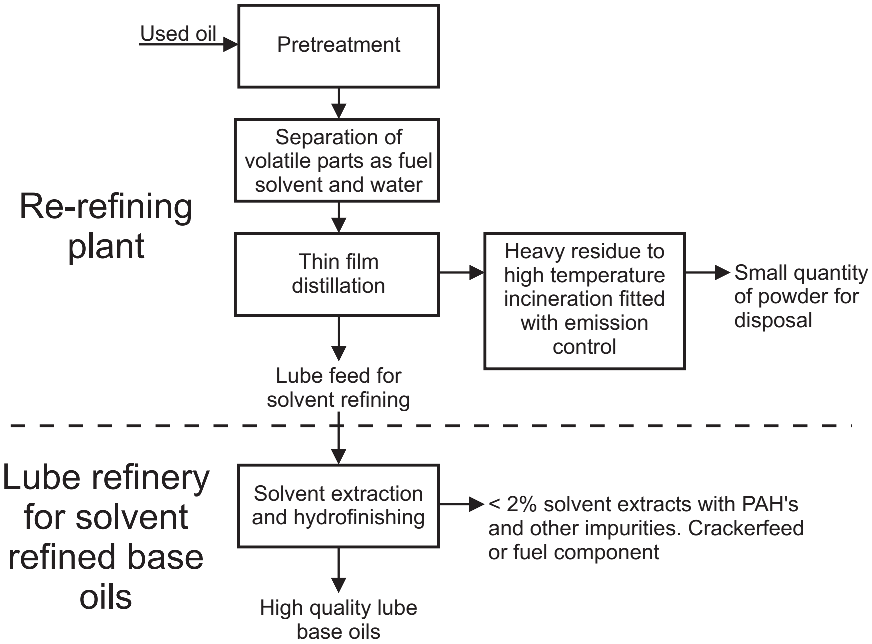

**Fig. 8.7** Introduction of selective solvent extraction in the re-refining process (DEA / Mineralöl-Raffinierie Dollbergen, Germany).

### 8.3.8
### Other Re-refining Technologies

Vaxon (Enpotec fabrication facilities in Denmark) uses three or four vacuum cyclone evaporators and finishing treatment with chemicals.

The key step in the ENTRA Technology is the special vacuum evaporation in a vacuum linear tubular reactor (single tube). After continuous evaporation by means of rapidly increasing temperature, vapor condensation is performed by fractional condensation. Complete dechlorination can be achieved with metallic sodium. Clay polishing is used as a finishing process.

The TDA Process (Thermal Deasphalting) has been developed by Agip Petroli / Viscolube on the technology of PIQSA Ulibarri in Spain. The process is based on chemical treatment to facilitate subsequent deasphalting.

The de-asphalting process is combined with high fractionating efficiency (TDA unit). Finishing can be performed by clay treatment or hydrofinishing.

# 9
# Lubricants for Internal Combustion Engines

*Manfred Harperscheid and Jürgen Omeis*

## 9.1
## Four-stroke Engine Oils

Technically and commercially, engine oils are the Number 1 among lubricants and functional fluids; on the global lubricants market, they account for more than 60 %.

While demand in Europe is more or less stagnating and will decline slightly in the foreseeable future in spite of increasing vehicle registrations, there is still considerable growth potential in Southeast Asia and third world threshold countries (China, India, Korea etc.) [9.1].

### 9.1.1
### General Overview

Historically the development of engine oils over the last 50 years has been focussed on the specifications issued by the international automobile industry. Starting with the first specifications published by the US Army (MIL specs), today there are three internationally recognized sets of minimum requirements. In Europe, these are ACEA, in the USA, API and ILSAC in Asia. Details of these specifications can be found in Section 9.1.3.

In principle, all specifications reflect the successive adaptation of oil qualities to developments in engine design. Back in the 1950s, monogrades dominated the engine oil market. As their name indicates, the viscosity of these oils was matched to the prevailing ambient temperature and therefore had to be changed between summer and winter. The 1960s saw the development of mineral oil-based multigrades, i.e. combined summer and winter oils, initially high-viscosity types (SAE 20W-50) and later the present standard 15W-40. As time passes, base oil distillation and refining processes caught-up with the new viscosity and performance requirements (see Chapter 4). This led to new qualities with comparatively low evaporation losses and optimized cold flowing properties. Semi-synthetic, and in recent years, fully-synthetic oils have dominated the premium-quality market, especially in the passenger car sector. In the recent past, a niche for environmentally friendly, biodegradable products has been created in the German-speaking market [9.2].

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#### 9.1.1.1 Fundamental Principles

Engine oils have to fulfil a wide range of functions in engines. The purely tribological task consists of guaranteeing the functional reliability of all friction points in all operating conditions (Fig. 9.1). Apart from this classical tribological task, engine oils have to perform a number of additional functions. This begins with the sealing of the cylinder and ends with the transport of sludge, soot and abraded particles to the oil filter.

Starting with the tribological functions, the three classic sections of the Stribeck graph are satisfied, from hydrodynamic full lubrication to the elasto-hydrodynamic (EHD) area in bearings to the boundary friction conditions at TDC and BDC [9.3]. All friction pairings and a whole series of parameters are covered. Sliding friction speeds from simple linear up-and-down movement of the piston in the cylinder through to extreme rotational movements in floating needle roller bearings found in advanced turbochargers rotating at speeds of up to 200 000 rpm with micron tolerances. The temperatures encountered range from ambient in the Arctic (–40 °C) to sump temperatures of 100 °C to peak values of over 300 °C under the piston crown.

During the combustion process, the engine oil helps to seal the piston and cylinder. At the same time, it should burn off the cylinder wall without leaving any residue. As for the piston itself, the engine oil dissipates heat from the piston and thus cools it. The blow-by gases formed when fuel is burned and their reaction by-products have to be neutralized and held in suspension. The same applies to the soot and sludge particles caused by incomplete combustion. The oil also transports dirt and any abraded particles to the oil filter and ensures its filterability. In addition, any water formed during the combustion process should be emulsified and even when higher concentrations are present and when the phases separate as temperature falls, the oil should protect against corrosion.

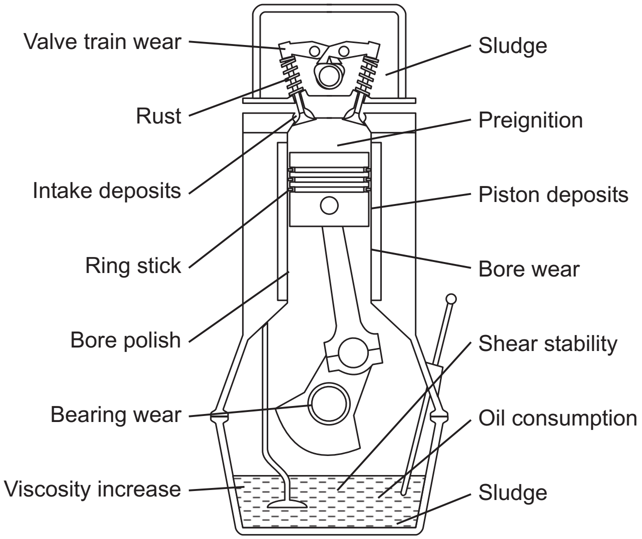

**Fig. 9.1** Scheme of lubrication points in an engine.

Engine oils should reduce friction and wear during extreme, low-temperature start-ups as well as when the lubricating film is subject to high temperatures and pressures in bearings and around the piston rings. While the oil should still flow well and be pumpable without aeration at low temperatures (down to –40 °C) to avoid metal-to-metal contact during cold start-ups, the lubricating film must perform satisfactorily in bearings and hydraulic tappets [9.4]. At low temperatures, additives must not precipitate and the oil must not gel. At the upper end of the temperature scale, the oil must offer far-reaching resistance to thermal and mechanical aging. And finally, the stability of the lubricating film should not be diminished by fuel dilution of up to 10 %.

#### 9.1.1.2 **Viscosity Grades**

The viscosity of an engine oil is an indicator of how readily a load-carrying film can be formed at all lubrication points in an engine. As viscosity is a function of temperature, this applies to all potential ambient and operating temperatures. The adequate and rapid circulation of the oil at low temperatures which is given by low viscosity [9.5] must be achieved at cold start cranking speeds. On the other hand, viscosity must not fall too much at high temperatures so that adequate lubricating film stability is given at high thermal loads.

**Tab. 9.1** Engine oil viscosity classification SAE J 300 July 01.

| *SAE viscosity grade* | *Low temperature cranking viscosity (mPa s) at temp. in °C (CCS)* | *Low temperature pumping viscosity (mPa s) at temp. in °C (MRV)* | *Low shear-rate kinematic viscosity at 100 °C ($mm^2 s^{-1}$)* | | *High shear-rate viscosity at 150 °C and $10^6 s^{-1}$ (mPa s)* |
|---|---|---|---|---|---|
| | *Maximum* | *Maximum* | *Minimum* | *Maximum* | *Minimum* |
| 0 W | 6200 at –35 | 60 000 at –40 | 3.8 | – | – |
| 5 W | 6600 at –30 | 60 000 at –35 | 3.8 | – | – |
| 10 W | 7000 at –25 | 60 000 at –30 | 4.1 | – | – |
| 15 W | 7000 at –20 | 60 000 at –25 | 5.6 | – | – |
| 20 W | 9500 at –15 | 60 000 at –20 | 5.6 | – | – |
| 25 W | 13000 at –10 | 60 000 at –15 | 9.3 | – | – |
| 20 | – | – | 5.6 | < 9.3 | 2.6 |
| 30 | – | – | 9.3 | < 12.5 | 2.9 |
| 40 | – | – | 12.5 | < 16.3 | 2.9* |
| 40 | – | – | 12.5 | < 16.3 | 3.7** |
| 50 | – | – | 16.3 | < 21.9 | 3.7 |
| 60 | – | – | 21.9 | < 26.1 | 3.7 |

* for 0 W, 5 W, 10 W

** for 15 W, 20 W, 25 W and Monogrades

As these requirements cannot be satisfactorily described with just one viscosity test method (see Section 9.1.2), corresponding threshold values were determined by the Society of Automotive Engineers (SAE) and the American Society for Testing and Materials (ASTM) as shown in Table 9.1. This table was last revised in December 1999.

According to this list, all viscosity grades can be described by their minimum kinematic viscosity at 100 °C. Additional dynamic viscosity thresholds apply to winter grades, which display the letter W. These values are determined in cold cranking

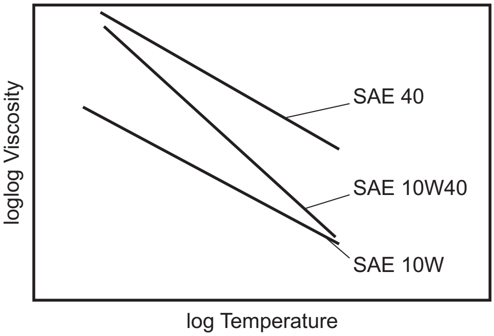

**Fig. 9.2** Comparison of monogrades and multigrades.

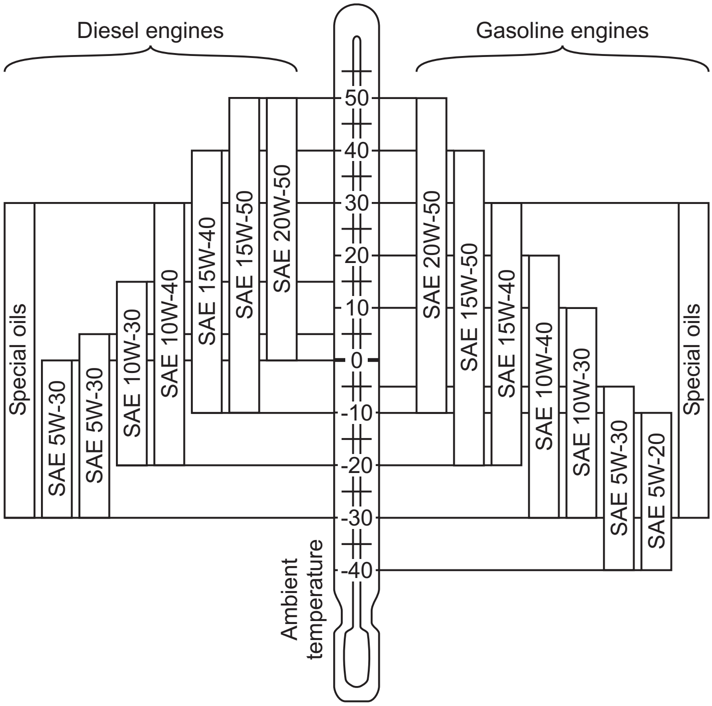

**Fig. 9.3** BMW engine oil recommendations.

simulators (CCS) or in mini-rotary viscometers (MRV). The dynamic viscosity value given by the CCS is a measure of flow properties [9.6] at low temperatures whereby the high shearing rate can mask paraffin crystallization. In the MRV, a so-called threshold viscosity of max. 60 000 cP has been determined for 10 °C lower temperatures to ensure that the oil pump does not draw air.

The high-temperature high-shear viscosity is an additional criteria for evaluating lube film stability at high shear rates and high temperatures in summer grades. In principle, mono or multigrade oils could be used, depending on the climate. As already stated, modern engine oils are multigrade oils whose low temperature characteristics are indicated by the W and the high temperature viscosity by the number following the W. In central Europe, 90 % of the engine oil market is accounted-for by multigrade oils. A graphical description for the VT Behaviour of multigrade oils is schematically sketched in Fig. 9.2. Manufacturers ensure that the correct viscosity grade for the climate considered is used in their engines by general descriptions (see Section 9.1.3) and/or by specific product approvals. In Fig. 9.3 one OEM recommendation is shown as an example.

#### 9.1.1.3 **Performance Specifications**

As a result of the continuing increase in specified oil performance, leading automobile manufacturers (OEMs) have discovered lubricants as constructional elements and have adopted their quality philosophy. The result is a qualitative shift in the engine oil market away from conventional products to semi-synthetic and synthetic formulations. Along with their higher price, the economic and ecological demands on these oils have also risen. Detailed demands, which are covered in Chapter 4, include:

- longer life in spite of higher thermal and mechanical loads [9.7]
- improved emission characteristics by a cut in fuel consumption [9.8]
- lower oil-related particulate emissions [9.9]
- improved wear protection even in severe conditions.

Put simply, engine oils will have to offer significant potential regarding reducing fuel consumption and extending oil change intervals. At present, the oil change intervals for cars are between 10 000 and 50 000 km and 30 000 and 100 000 km for trucks. In the future, these figures are expected to increase even further. A retrospective view of specific oil performance shows that this development has been in progress for the last 50 years [9.10]. Figure 9.4 shows that oil consumption per energy unit has fallen eight times during this period.

As the established CEC engine tests do not allow comprehensive testing of all required oil properties, additionally in-house methods are now being used. A series of OEM-specific, long-term trials, which have now been taken-over for developing oils, are part of these engine tests. In total, these tests represent an enormous technical advance and financial expense because the engine tests themselves have been supplemented by radionuclide techniques (RNT) [9.11]. The advantages of this technology lie in the on-line monitoring of wear in defined running conditions, from running-in to full-throttle operation as well as the selective examination of critical engine components.

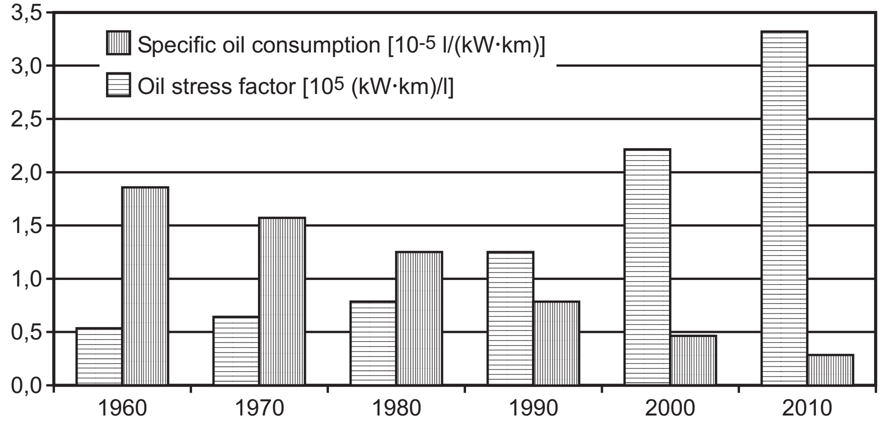

**Fig. 9.4** The history of oil stress.

### 9.1.1.4 **Formulation of Engine Oils**

Engine oils are complex mixtures which are best described as formulations containing base oils and additives. Compared to other groups of lubricants, the base oils [9.12] play an important role (see Chapters 4 and 5). Without going into the characteristics and manufacturing in detail (see Chapters 4 and 19), mixtures of base oils are selected which have the necessary viscosity and performance to correspond to a rough classification. The final products are then marketed as conventional mineral oil-based, unconventional semi-synthetic (hydrocracked) and synthetic engine oils. Precise international nomenclature divides base oils into six groups:

- Group 1: SN mineral oils with saturates < 90 %, 80 < VI < 120, S > 0.03 %
- Group 2: HC oils with saturates > 90 %, 80 < VI < 120, S < 0.03 %
- Group 3: HC oils with saturates > 90 %; VI > 120, S < 0.03 %
- Group 4: Polyalphaolefins
- Group 5: Esters and others
- Group 6: Polyinternalolefins

### 9.1.1.5 **Additives**

Depending on the base oil used and the required engine performance, engine oils can contain up to 30 different additives whose percentage content can range from 5 to 25 % in total. In the oil industry, a differentiation is made between performance additives [9.13], viscosity improvers and flow improvers. As a rule, the performance additives make up the largest group.

### 9.1.1.6 **Performance Additives**

The following species of chemical components are summarised under the general term of performance additives (Table 9.2).

**Tab. 9.2** Performance additives.

| | |
|---|---|
| Antioxidants | Phenols, amines, phosphites, sulfurized substances |
| Anti-wear agents | Metal dithiophosphate, carbamate |
| Detergents | Ca and Mg sulfonates, phenolates, salicylates |
| Dispersants | Polyisobutylene and ethylene-propylene oligomers with nitrogen and or oxygen as a functional group |
| Friction modifiers | MoS compounds, alcohols, esters, fatty acid amides, etc. |
| Anti-misting agents | Silicone and acrylate |

Chemical formulation, tribological effect and theoretical background to these additive groups are examined in detail in Chapter 6. Particularly in the case of engine oils, the substance categories listed generally perform more than one function. ZnDDPs, for example, are primary anti-wear additives but also have a secondary antioxidant character resulting from a specific decomposition mechanism [9.14]. Furthermore, complex formulations of a number of individual components typically display synergistic as well as antagonistic interactions which have to be matched to the application considered. The composition of the base oil components has an additional effect on these specific interactions. Considerable experience and know how is thus necessary to create an optimum formulation.

#### 9.1.1.7 **Viscosity Improvers**

Viscosity improvers can be divided into two groups, the non-polar, non-dispersing and the polar, dispersing group. The first group are only really needed to set the viscosity of multigrade oils. Viscosity improvers increase viscosity and the viscosity index by altering their solubility at various temperatures (see Section 6.2). In an absolute concentration of 0.2 to 1.0 %, they can generate a viscosity increase of between 50 and 200 % depending on chemical structure and base oil solubility. Due to special modification, dispersing viscosity improvers are often ashless dispersants with additional thickening effects. Furthermore, viscosity improvers and pour-point depressants have an effect on low temperature behavior of a formulation (PP, CCS, MRV) and are essential parts of the HTHS viscosity. In the USA, additional demands are made on low-temperature stability (gelation index) which cannot be achieved without the viscosity improvers and pour-point depressants being matched to the base oil.

### 9.1.2
### Characterization and Testing

To explain performance specifications and viscosity grading better, this section discusses engine oil tests in detail.

#### 9.1.2.1 **Physical and Chemical Testing**

The physicochemical properties of an engine oil can be determined in the laboratory with standard test methods (Chapter 19). This characterization mainly focuses on rheological test values and the previously shown SAE classification system.

Various viscosity tests are used to determine exact low- and high-temperature viscosities [9.15]. The viscosity thus determined is a characteristic of the engine oil at a defined engine state. At low temperatures (–10 to –40 °C), a MRV (mini rotary viscometer) with a low shear gradient is used to determine the apparent viscosity and thus the oil's flowability in the area of the oil pump. In addition, maximum viscosity as the threshold of viscosity is determined in five graduated steps. The dynamic CCS (cold cranking simulator) viscosity, which is determined at –10 to –40 °C with a high shear gradient, is also an apparent viscosity which represents the tribological conditions at the crankshaft during cold starts. The maximum values laid-down in SAE J 300 guarantee reliable oil circulation during the start-up phase. The rheological characteristics at higher thermal loads which occur during full-throttle operation are described by the dynamic viscosity at 150 °C and a shear rate of $10^6$ $s^{-1}$ or HTHS (high-temperature high-shear). The corresponding threshold values also guarantee an adequate lube film even in these conditions.

Apart from the rheological characteristics, the Noack evaporation test described in Chapter 19 to test the volatility of base oils and additives as well as foaming tendency and air release can be characterized with simple methods. Furthermore, the compatibility of high-additive oils with seal materials is tested on standard reference elastomers in static swelling and subsequent elongation tests [9.16]. The viscosity loss resulting from mechanical load is described in Section 6.2.

#### 9.1.2.2 **Engine Testing**

Because realistic engine oil tests cannot be performed only over long lasting field trials, a number of international committees have created methods of testing engine oils in defined test engines operated in reproducible and practically relevant conditions.

In Europe, the CEC (Coordinating European Council for the Development of Performance Tests for Lubricants and Fuels) is responsible for testing, approval and standardization [9.17]. Performance requirements are set-up in the form of ACEA (Association des Constructeurs Europeen d'Automobiles) oil sequences which are decided together with the additive and lubricant industries. In the USA, this task is performed by the automobile industry and the API (American Petroleum Institute). This institution lays down test procedures and limits. The Asian ILSAC has largely adopted the American specifications for automobiles.

In principle, the test procedures detailed in Sections 9.1.3 and 9.1.4 focus on the following general performance criteria:

- oxidation [9.18] and thermal stability
- dispersion of soot and sludge particles
- protection against wear [9.19] and corrosion
- foaming and shear stability [9.20]

In detail, the specification of the tests differentiate between gasoline- and diesel-powered car engines and truck engines whereby every test engine is characterized by one or a group of criteria. Tables 9.3 and 9.4 show the relevant criteria for gasoline and diesel engines.

**Tab. 9.3** Passenger car engine tests.

| *Test engine* | *Test procedure* | *Test criteria* |
|---|---|---|
| Peugeot XUD 11 | CEC L-56-T-95 | Soot handling<br>Piston cleanliness |
| Peugeot TU5 JP | CEC L-88-T-02 | Cleanliness<br>Oxidation<br>Ring sticking |
| Peugeot TU3 S | CEC L-38-A-94 | Cam and tappet wear |
| Sequence II D | ASTM STP B15 M P1 | Bearing corrosion |
| M 111 SL | CEC L-53-T-95 | Black sludge<br>Cam wear |
| Sequence IIIE | ASTM STR 315 M P2 | Oxidation<br>Wear<br>Cleanliness |
| Sequence VG | ASTM D 6593 | Sludge<br>Piston cleanliness<br>Ring sticking |
| BMW M52 | | Valve train<br>Air entrainment<br>Wear |
| VW T4 | | Oil oxidation<br>TBN depletion<br>Piston cleanliness |
| M111 FE | CEC L-54-T-96 | Fuel efficiency |
| VW-DI | P-VW 1452 | Piston cleanliness<br>Ring sticking |
| VW-TD | CEC L-46-T-93 | Piston cleanliness<br>Ring sticking |
| M271 Sludge | | Black Sludge |
| M271 Wear | | Wear<br>Cleanliness<br>Oxidation<br>Oil consumption |
| OM611 | | Wear<br>Cleanliness<br>Oxidation<br>Oil consumption |

**Tab. 9.4** Heavy duty engine tests.

| *Test engine* | *Test procedure* | *Test criteria* |
|---|---|---|
| Caterpillar 1K/1N | | Piston cleanliness<br>Wear<br>Oil consumption |
| Cummins M11 | | Valve train wear<br>Sludge |
| Mack T8 | ASTM D 4485 | Soot handling |
| Mack T10 | | Liner and ring wear |
| GM 6,2 Liter | | Valve train wear |
| OM 364LA | CEC L-42-T-99 | Piston cleanliness<br>Cylinder wear<br>Sludge<br>Oil consumption |
| OM 602A | CEC L-51-A-98 | Wear<br>Cleanliness<br>Oxidation<br>Oil consumption |
| OM 441LA | CEC L-52-T-97 | Piston cleanliness<br>Cylinder wear<br>Turbocharger deposits |

### 9.1.2.3 **Passenger Car Engine Oils**

Car engines include all gasoline and light diesel engines with direct or indirect injection. To ensure that the minimum requirements are met, the performance of the oils must be proven in the listed test engines irrespective of viscosity grade or the base oil used.

For gasoline engines, oxidation stability is tested in Seq. III E ($T$ max = 149 °C) and in a Peugeot TU5 JP engine. Apart from the oxidation-related increase in viscosity (KV 40), the effect of aging-induced deposits on the piston and ring groove cleanliness is evaluated. Another three standardized tests focus on sludge evaluation. This is the ability of an oil to efficiently disperse oil-insoluble aging residues which result from the combustion process. Insoluble and inadequately dispersed particles lead to a sticky, pasty oil sludge which can block oil passages and filters and thus lead to lubrication breakdowns. According to M 271 SL and M 111 SL, such sludge should be visually examined in the sump, in the crankcase and oil passages as well as by measuring the pressure increase created in filters. While the European M 271 SL and M 111 SL tests are performed ‘hot’, i.e. at high loads and speeds with a fuel which is sensitive to nitroxidation, sequence VG focuses on the generally lower operating temperatures in North America which lead to the formation of a socalled ‘cold’ black sludge. The Peugeot TU3 engine is used to check critical valve-train wear which can effect the timing of the engine. After a variable-load test program, cam scuffing and tappet pitting is evaluated.

The light diesel test engines, which are gaining popularity in passenger cars in Europe, are exclusively European engines. Again, oxidation stability and diesel-specific soot dispersion are in the forefront. The increase in injection pressures has led to an increase in soot formation and thus up to 500 % oil thickening and combustion temperatures have also increased. These criteria as well as their influence on exhaust gases are tested in VW 1.6 liter intercooler and a Peugeot XUD 11 (viscosity increase). Also to be avoided are secondary effects on cylinder and cam wear and bore polishing which indicates that the original honing patterns have been worn away. A socalled multipurpose OM 602 A test engine was also added to the testing program.

In 2003 the OM 611 DE 22 LA got an important additional multipurpose test in diesel engine oil development. This test has to be run with today's low-sulfur diesel fuel and shows soot concepts up to 8 % after its 300 h runtime. Such conditions need engine oils with extremely good soot handling properties to avoid large viscosity increases and wear.

Further-reaching OEM-specific tests include the severe criteria of extended oil drain intervals and fuel saving. These apparently contradictory aspects of lower viscosity and less consumption on one hand and lower viscosity and greater reliability on the other represents a great challenge to oil manufacturers.

#### 9.1.2.4 **Engine Oil for Commercial Vehicles**

Commercial vehicles include trucks, buses, tractors, harvesters, construction machines and stationary machinery powered by diesel engines. Apart from the prechamber diesels engines which have largely been superceded in Europe, the engines are usually highly turbocharged direct injection motors. Economic and ecological aspects along with high injection pressures, have improved combustion and thus reduce emissions. As an initiative of ACEA, oil change intervals have been extended up to 100 000 km for long haulage. The following highlights the fundamental differences between diesel and gasoline engines.

Long-life and reliability are the criteria for the commercial vehicle sector. The HD (Heavy Duty) oils have to match these requirements. The predominant requirements are the dispersion of large concentrations of soot particles as well as the neutralization of sulfuric acid combustion by-products. Performance is also judged by piston cleanliness, wear and bore polishing. Oxidation and soot-related deposits, mainly in the top ring groove lead to poor piston evaluations and an increase in wear. This, in turn, leads to the abrasion of the honing patterns in the cylinders, a problem better known as bore polishing. The result is increased oil consumption and poorer piston lubrication because the oil cannot be trapped by the honing rings. Inadequate soot and sludge dispersion as well as chemical corrosion can lead to premature bearing wear. And finally, advanced turbocharged diesel engines have also been evaluated. Blow-by gases always carry some oil mist into the exhaust and turbochargers are very sensitive to unstable oil components.

In total, all characteristics can be found in HD oils whereby these are allocated to the following categories with increasing performance:

- heavy duty (HD)
- severe heavy duty (SHPD), and
- extreme heavy duty (XHPD).

Despite numerous efforts to use screening tests to find the information, 4 to 6 cylinder engines are used to test the main performance criteria in runs of over 400 h and have displaced the original single-cylinder test engines (MWMB; Petter AWB).

Apart from the above-mentioned OM 602 A and OM 611 multipurpose test engines, European specifications demand a OM 364 LA or OM 441 LA Daimler–Chrysler engine. Both test procedures are only used with XHPD oils (oil change intervals up to 100 000 km), piston cleanliness, cylinder wear and bore polishing are determined and evaluated. Particularly in the OM 441 LA, deposits on the turbocharger as well as a pressure increase have been recorded. The criterion, soot-induced oil thickening is tested by the ASTM test (Mack T 8).

Independent of the viscosity grade and the base oils used, classic HD oils have a high reserve alkalinity and thus a higher content of earth alkaline salts and organic acids [9.21]. Also regarding ashless dispersants, the oils are designed for soot dispersing. Special viscosity improvers are used generally to avoid additional deposits.

Oils for vehicle fleets pose a particular challenge. As opposed to special products, these should simultaneously satisfy as many car and truck demands as possible. Possible piston cleanliness provided by high concentrations of over-based soaps is sacrificed because gasoline engines are prone to self-ignition if high proportions of metal detergents are present. As a result, other components are selected, such as the skillful use of unconventional base oils along with detergents, dispersants, VI-improvers and antioxidants.

## 9.1.3
## Classification by Specification

As already mentioned, physical and chemical properties are not enough to select the best lubricant for an engine. Complex and expensive practical and bench engine tests are performed to test and understand the performance of a lubricant. These requirements reappear in delivery conditions, in-house standards and general specifications.

### 9.1.3.1 MIL Specifications

These specifications originate from the US Forces which set the minimum requirements for their engine oils. These are based on certain physical and chemical data along with some standardized engine tests. In the past, these classifications were also used in the civilian sector to define engine oil quality. In recent years, this specification has become almost irrelevant for the German market.

**MIL-L-46152 A to MIL-L-46152 E** These military specifications have now been discarded. Engine oils which meet these specifications are suitable for use in US gasoline and diesel engines. MIL-L-46152 E (discarded in 1991) corresponds to API SG/CC.

**MIL-L-2104 C** Classifies high-additive engine oils for US gasoline and normally aspirated and turbocharged diesel engines.

**MIL-L-2104 D** Covers MIL-L-2104 C and requires an additional engine test in a highly-charged Detroit 2-stroke diesel engine. In addition, Caterpillar TO-2 and Allison C-3 specifications are fulfilled.

**MIL-L-2104 E** Similar in content to MIL-L-2104 C. The gasoline engine tests have been up-dated and include more stringent test procedures (Seq. III E / Seq. V E)

### 9.1.3.2 API and ILSAC Classification

The American Petroleum Institute (API) together with the American Society for Testing and Materials (ASTM) and the SAE (Society of Automotive Engineers Inc., New York) have created a classification in which engine oils are classified according to the demands made on them, bearing in mind the varying conditions in which they are operated and the different engine designs in use (Table 9.5). The tests are standard engine tests. The API has defined a class for gasoline engines (S = service oils) and for diesel engines (C = commercial). Diesel engines in passenger cars are still outnumbered but have increased in recent years and are finding more acceptance in the USA. In addition, a number of fuel economy stages has been determined (EC = energy conserving).

**Tab. 9.5** Engine oil classification according to API SAE J 183.

| *Gasoline engines* | *(Service classes)* |
|---|---|
| API-SA | Regular engine oils possibly containing pour-point improvers and/or foam inhibitors. |
| API-SB | Low-additive engine oils low-power gasoline engines. Include additives to combat aging, corrosion and wear. Issued in 1930. |
| API-SC | Engine oils for average operating conditions. Contain additives against coking, black sludge, aging, corrosion and wear. Fulfil the specifications issued by US automobile manufacturers for vehicles built between 1964 and 1967. |
| API-SD | Gasoline engine oils for more difficult operating conditions than API-SC. Fulfil the specifications issued by US automobile manufacturers for vehicles built between 1968 and 1971. |
| API-SE | Gasoline engine oils for very severe demands and highly-stressed operating conditions (stop and go traffic). Fulfil the specifications issued by US automobile manufacturers for vehicles built between 1971 and 1979. Covers API-SD; corresponds approximately to Ford M2C-9001-AA, GM 6136 M and MIL-L 46 152 A. |
| API-SF | Gasoline engine oils for very severe demands and highly-stressed operating conditions (stop and go traffic) and some trucks. Fulfil the specifications issued by US automobile manufacturers for vehicles built between 1980 and 1987. Surpasses API-SE with regard to oxidation stability, wear protection and sludge transportation. Corresponds to Ford SSM-2C-9011 A (M2C-153-B), GM 6048-M and MIL-L 46 152 B |
| API-SG | Engine oils for the severest of conditions. Include special oxidation stability and sludge formation tests. Fulfil the specifications issued by US automobile manufacturers for vehicles built between 1987 and 1993. Specifications similar to MIL-L 46 152 D |

**Tab. 9.5** (Continued)

| ***Gasoline engines*** | ***(Service classes)*** |
|---|---|
| API-SH | Specification for engines oils built after 1993. API-SH must be tested according to the CMA's Code of Practice. API-SH largely corresponds to API-SG with additional demands regarding HTHS, evaporation losses (ASTM and Noack tests), filterability, foaming and flashpoint. Furthermore, API-SH corresponds to ILSAC GF-1 without the Fuel Economy test but with the difference that 15W-X multigrade oils are also permissible. |
| API-SJ | Supercedes API-SH. Greater demands regarding evaporation losses. Valid since 10/96. |
| API-SL | For 2004 and older automotive engines. Designed to provide better high-temperature deposit control and lower oil consumption. May also meet the ILSAC GF-3 specification and qualify as Energy Conserving. Introduced in July 2001. |
| API-SM | For all automotive engines currently in use. Designed to provide improved oxidation resistance, improved deposit protection, better wear protection, and better low temperature performance. May also meet the ILSAC GF-4 specification and qualify as Energy Conserving. Introduced in November 2004. |

| ***Diesel engines*** | ***(Commercial classes)*** |
|---|---|
| API-CA | Engine oils for low-power gasoline and normally aspirated diesel engines run on low-sulfur fuels. Corresponds to MIL-L 2104 A. Suitable for engines built into the 1950s. |
| API-CB | Engine oils for low-to-medium power gasoline and normally aspirated diesel engines run on low-sulfur fuels. Corresponds to DEF 2101 D and MIL-L 2104 A Suppl. 1 (S1). Suitable for engines built from 1949 on. Offer protection against high-temperature deposits and bearing corrosion. |
| API-CC | Gasoline and diesel engine oils for average to difficult operating conditions. Corresponds to MIL-L 2104 C. Offer protection against black sludge, corrosion and high-temperature deposits. For engines built after 1961. |
| API-CD | Engine oils for heavy-duty, normally aspirated and turbocharged diesel engines. Covers MIL-L 45 199 B (S3) and corresponds to MIL-L 2104 C. Satisfies the requirements of Caterpillar Series 3. |
| API-CD II | Corresponds to API-CD. Additionally fulfils the requirements of US 2-stroke diesel engines. Increased protection against wear and deposits. |
| API-CE | Engine oils for heavy-duty and high-speed diesel engines with or without turbocharging subject to fluctuating loads. Greater protection against oil thickening and wear. Improved piston cleanliness. In addition to API-CD, Cummins NTC 400 and Mack EO-K/2 specifications must be fulfilled. For US engines built after 1983. |
| API-CF | Replaced API-CD for highly turbocharged diesel engines in 1994. High ash. Suitable for sulfur contents > 0.5 %. |
| API-CF-2 | Only for 2-stroke diesel engines. Replaced API-CD II in 1994. |
| API-CF-4 | Engine oil specification for high-speed, 4-stroke diesel engines since 1990. Meets the requirements of API-CE plus additional demands regarding oil consumption and piston cleanliness. Lower ash content. |

**Tab. 9.5** (Continued)

| *Diesel engines* | *(Commercial classes)* |
|---|---|
| API-CG-4 | For heavy-duty truck engines. Complies with EPA's emission thresholds introduced in 1994. Replaced API-CF-4 in June 1994. |
| API-CH-4 | Replaces API-CG-4. Suitable for sulfur contents > 0.5 %. |
| API-CI4 | For high-speed, four-stroke engines designed to meet 2004 exhaust emission standards. Formulated to sustain engine durability where exhaust gas recirculation (EGR) is used and are intended for use with diesel fuels ranging in sulfur content up to 0.5 % weight. Replaces oils with API CD, CE, CF-4, CG-4 and CH-4. |

| *All engines* | *(Energy Conserving)* |
|---|---|
| (API-EC I) | (min. 1.5 % less fuel consumption than an SAE 20W-30 reference oil in a 1982, 3.8 liter, Buick V6 gasoline engine. Sequence VI test) |
| (API-EC II) | Same as API-EC I but with minimum 2.7 % lower fuel consumption |
| API-EC | Replaces API-EC I and II. Only together with API SJ, SL, SM. Cuts in fuel consumption: 0W-20, 5W-20 > 1.4 %, 0W-XX, 5W-XX > 1.1 %, 10W-XX, others > 0.5 %, Sequence VI A test: In a 1993, 4.6 liter Ford V8 engine. Reference oil 5W-30 |

### 9.1.3.3 **CCMC Specifications**

As API and MIL specifications were only tested on large-capacity, slow-running US V8 engines, and the demands made by European engines (small capacity, high-speed) were only inadequately satisfied, the CEC (Co-ordinating European Council for the Development of Performance Tests for Lubricants and Engine Fuels) together with the CCMC (Committee of Common Market Automobile Constructors) developed a series of tests in which European engines were used to test engine oils (Table 9.6). These and the API tests formed the basis for the development of new engine oils. In 1996, CCMC was replaced by ACEA and ceased to be valid.

**Tab. 9.6** Engine oil classification according to CCMC.

| *Gasoline engines* | *(Gasoline Engines)* |
|---|---|
| CCMC G1 | Corresponds approximately to API-SE with 3 additional tests in European engines. Withdrawn on December 31, 1989. |
| CCMC G2 | Corresponds approximately to API-SF with 3 additional tests in European engines. Applies to conventional engine oils. Replaced by CCMC G4 January 1, 1990. |
| CCMC G3 | Corresponds approximately to API-SF with 3 additional tests in European engines. Makes high demands on oxidation stability and evaporation losses. Applies to low viscosity oils. Replaced by CCMC G4 January 1, 1990. |
| CCMC G4 | Conventional multigrade oils in line with API-SG, with additional black sludge and wear tests. |
| CCMC G5 | Low viscosity engine oils complying to API-SG with additional black sludge and wear tests. Greater demands than CCMC G4. |

**Tab. 9.6** (Continued)

| *Diesel engines* | *(Diesel Engines)* |
|---|---|
| CCMC D1 | Corresponds approximately to API-CC with 2 additional tests in European engines. For light trucks with normally aspirated diesel engines. Withdrawn on December 31, 1989. |
| CCMC D2 | Corresponds approximately to API-CD with 2 additional tests in European engines. For trucks with normally aspirated and turbocharged diesel engines. Replaced on January 1, 1990 by CCMC D4. |
| CCMC D3 | Corresponds approximately to API-CD/CE with 2 additional tests in European engines. For trucks with turbocharged diesel engines and extended oil change intervals (SHPD oils). Replaced on January 1, 1990 by CCMC D5. |
| CCMC D4 | Surpasses API-CD/CE. Corresponds to Mercedes–Benz Sheet 227.0/1. For trucks with normally aspirated and turbocharged diesel engines. Better protection against wear and oil thickening than CCMC D2. |
| CCMC D5 | Corresponds to Mercedes–Benz Sheet 228.2/3. For heavy-duty trucks with normally aspirated and turbocharged diesel engines and extended oil change intervals (SHPD oils). Better protection against wear and oil thickening than CCMC D3. |
| CCMC PD 1 | Corresponds to API-CD / CE. For normally aspirated and turbocharged diesel engines in cars. Replaced by CCMC PD 2 on January 1, 1990. |
| CCMC PD 2 | Defines the requirements of high-performance, multigrade oils for the present generation of diesel engines in cars. |

#### 9.1.3.4 **ACEA Specifications**

As a result of persistent internal differences, the CCMC was disbanded and succeeded by the ACEA (Association des Constructeurs Europeens d'Automobiles). CCMC specifications remained valid in the interim period. The first ACEA classifications came into force on January 1, 1996.

The ACEA specifications were revised in 1996 and replaced by 1998 versions. The 1998 specifications became valid on March 1, 1998.

Additional foaming tests were introduced for all categories and the elastomer tests were modified.

A-categories referred to gasoline, B-categories to passenger car diesel, and E-categories to heavy-duty diesel engines.

The 1998 specifications were then replaced by the 1999 version, on September 1, 1999, and remained valid until February 1, 2004. Categories E2, E3, and E4 for heavy-duty diesel oils were updated, and a new category, E5, was introduced; these were specifically aimed at the new demands for Euro 3 engines and the often higher soot content of such oils. A and B categories remained identical with the 1998 version.

On February 1, 2002 the ACEA 2002 oil sequences were issued to replace the 1999 sequences; these will be valid until November 1, 2006. Updates in cleanliness and sludge for gasoline engines (categories A1, A2, and A3) and a new category A5 with the engine performance of A3 but higher fuel economy were introduced. Tests for cleanliness, wear, and soot handling were updated for diesel passenger cars and a new category B5 with outstanding cleanliness and increased fuel economy was introduced. For category E5 oils wear performance in respect of ring, liner, and bearings was tightened.

Since November 1, 2004 the ACEA 2004 oil sequences have been in use and can be claimed by oil marketers. Oils in these categories are backwards compatible with all other issues (Table 9.8). Categories A and B are now combined and can only be claimed together. Categories C1, C2, and C3 are new and refer to engine oils for use in cars with exhaust after treatment systems such as diesel particulate filters (DPF). Such oils are characterized by especially low content of ash-forming components and reduced sulfur and phosphorus levels to minimize the impact on filter systems and catalysts.

**Tab. 9.8** Engine oil classification according to ACEA 2002 and 2004.

| *Passenger car engines category* | *Application area* |
|---|---|
| ACEA 2002: | |
| A1-02 | Low-viscosity (HTHSV max 3.5 mPa s) oils with extra high fuel economy. Preferred SAE grades are xW-20 and xW-30 |
| A2-96 issue 3 | Multigrade fuel-economy oils, HTHSV min. 3.51 mPa s, performance higher than API SH |
| A3-02/ | Multigrade fuel-economy oils, HTHSV min. 3.51 mPa s, performance higher than A2 especially with regard to high-temperature stability and evaporative loss |
| A5-02 | Low-viscosity (HTHSV max. 3.5 mPa s) oils with extra high fuel economy, engine performance similar to ACEA A3-02 |
| B1-02 | Similar to A1-02 low viscosity (HTHSV max. 3.5 mPa s) oils with extra high fuel economy. Preferred SAE grades are xW-20 and xW-30 |
| B2-98 issue 2 | Similar to A2 multigrade fuel-economy oils, HTHSV min. 3.51 mPa s, performance above API CG-4 |
| B3-98 issue 2 | Similar to A3-02 multigrade fuel-economy oils, HTHSV min. 3.51 mPa s, performance higher than B2 especially with regard to piston cleanliness, soot handling, and shear stability |
| B4-02 | Multigrade fuel-economy oils, HTHSV min. 3.51 mPa s, additionally tested in turbocharged DI-Diesel (85kW-"VW-"Pumpe-Düse" engine") with regard to piston cleanliness and ring sticking |
| B5-02 | Similar to A5-02 low viscosity oils with extra high fuel economy. Also tested in turbocharged DI-Diesel (85 kW-"VW-"Pumpe-Düse" engine"). Extra high piston cleanliness limit |
| ACEA 2004: | |
| A1/B1-04 | Combines A1-02 and B1-02. Engine performance unchanged |
| A3/B3-04 | Combines A3-02 and B3-98. Engine performance unchanged |
| A3/B4-04 | Combines A3-02 and B4-02. Engine performance unchanged |
| A5/B5-04 | Combines A5-02 and B5-02. Engine performance unchanged |

**Tab. 9.8** (Continued)

| ***Passenger car engines category*** | ***Application area*** |
|---|---|
| C1-04 | New category for multigrade oils with extra fuel economy (HTHSV max. 3.5 mPa s), but extra low ash, phosphorus, and sulfur content (0.5, 0.05, and 0.2 % *w*/*w*, respectively), in particular for use in Euro 4-type engines with advanced exhaust-treatment systems (e.g. DPF). The oils meet the performance level of A5/B5-04 |
| C2-04 | New category for multigrade oils with extra fuel economy (HTHSV max. 3.5 mPa s), but lower ash, phosphorus, and sulfur content (0.8, 0.09, and 0.3 % *w*/*w*, respectively), in particular for use in Euro 4 engines with advanced exhaust-treatment systems (e.g. DPF).The oils meet the performance level of A5/B5-04 |
| C3-04 | New category for multigrade fuel-economy oils (HTHSV min. 3.51 mPa s), but lower ash, phosphorus, and sulfur content (0.8, 0.09, and 0.3 % *w*/*w*, respectively), in particular for use in Euro 4 engines with advanced exhaust-treatment systems (e.g. DPF). The oils meet the performance level of A3/B4-04 |

| ***Heavy duty engines category*** | ***Application area*** |
|---|---|
| ACEA 2002: | |
| E2-96 issue 4 | Multigrade general-purpose oils for naturally aspirated and turbocharged heavy-duty diesel engines, medium to heavy-duty cycles and usually normal oil-drain intervals (MB 228.1 level and additional Mack T8 test). |
| E3-96 issue 4 | Multigrade oils with advanced performance of wear, piston cleanliness, bore polish and soot handling. Mostly recommended for diesel engines meeting Euro 1 and Euro 2 emission requirements and running under severe conditions, often with extended oil-drain intervals according to manufacturers recommendations. (MB 228.3 level and additional Mack T8 test) |
| E4-99 issue 2 | Multigrade oils mostly recommended for diesel engines meeting Euro 1, Euro 2, and Euro 3 emission requirements and running under severe conditions, often with extended oil-drain intervals according to manufacturers recommendations. (MB 228.5 level and additional Mack T8 and T8E test). Provides further control of piston cleanliness, wear, and soot handling compared to E3 |
| E5-02 | Multigrade oils with performance level between E3 and E4. Recommended for diesel engines meeting Euro 1, Euro 2, and Euro 3 emission requirements and running under severe conditions. Advanced soot handling compared with E4, for use in engines with high exhaust gas recirculation (EGR) |
| ACEA 2004: | |
| E2-96 issue 5 | Similar to E2-96 issue 4. Engine performance unchanged |
| E4-99 issue 3 | Similar to E4-99 issue 2. Engine performance unchanged |

Tab. 9.8 (Continued)

| *Heavy duty engines category* | *Application area* |
|---|---|
| E6-04 | New category of multigrade oils for latest-generation diesel engines with advanced exhaust-treatment systems. Lower ash, phosphorus, and sulfur content (max. 1.0, 0.08, and 0.3 % *w/w*, respectively) compared to E4. Engine performance similar to E4 plus Mack T10 test for additional control of liner, ring, and bearing wear |
| E7-04 | New category for multigrade oils with extended performance of E4 with regard to soot handling and wear (additional Cummins M11 and Mack T10 tests), including former E5 demands |

### 9.1.3.5 Manufacturers' Approval of Service Engine Oils

Apart from the specifications already listed, some manufacturers have their own specifications and usually demand tests on their own engines (Table 9.9).

Tab. 9.9 Manufacturers' Approvals

| *BMW* | *Application area* |
|---|---|
| Special oil | For BMW vehicles built before 1998, predominantly SAE 10W-40 or lower viscosity classes. BMW special oils could be used throughout the whole year whereas use of other fuel-economy oils was restricted by low outside temperatures |
| Longlife 98 | For nearly all BMW cars from 1998 onwards, suitable for the second-generation of flexible service systems, covering more than 20,000 km. This category is backwards compatible |
| Longlife 01 | For nearly all BMW cars from 2001 onwards. With introduction of a new test engine the oil performance increased significantly. The average service interval, given by the flexible service system increased. This category is backwards compatible also |
| Longlife 01 FE | BMW introduced a new generation of gasoline engines with the capability of using engine oils with reduced high temperature high shear viscosity. Therefore the Longlife 01 FE category was introduced which provided a fuel economy benefit of min. 1 % compared with an SAE 5W-30 Longlife 01 engine oil |
| Longlife 04 | This category was designed for the specific requirements of exhaust gas after treatment with, for example, particulate filters. Longlife 04 oils therefore have components resulting in especially low phosphorus, sulfur, and ash content. They are backwards compatible for vehicles operating in central Europe |

**Tab. 9.9** (Continued)

| ***DAF*** | ***Application area*** |
|---|---|
| HP-1 | Specifies engine oils of ACEA E4 and E5-type in SAE XW-30 grades for standard oil-drain intervals according to the DAF maintenance system |
| HP-2 | Specifies ACEA E5-type engine oils, independently on viscosity class and ACEA E4-type engine oils in SAE XW-30 grades to provide capability for long drain intervals according to the DAF maintenance system |
| HP-3 | A specific category for ACEA E5 engine oil if used in a XE / 390 kW engine for standard oil-drain intervals |
| HP-GAS | This category specifies the engine oils for DAF vehicles equipped with gas engines |

| ***Deutz*** | ***Application area*** |
|---|---|
| DQC I | Specifies oils meeting ACEA E2, API CF/ CF-4 for natural aspirated diesel engines under light and medium operating conditions |
| DQC II | Specifies oils meeting ACEA E3/ E5 or E7 or alternatively API CG-4 to CI-4 or DHD-1. For use in natural aspirated and turbo charged engines under medium and severe operating conditions |
| DQC III | Specifies oils meeting ACEA E4 / E6 requirements for modern engines under more severe operating conditions, for example power plant application |
| DQC IV | Specifies synthetic engine oils meeting ACEA E4/ E6 requirements for use in high-power engines with closed-crankcase ventilation systems |

| ***MAN*** | ***Application area*** |
|---|---|
| MAN 270 | Monograde oils for turbocharged and non-turbocharged diesel engines. Oil-drain intervals of 30,00045,000 km |
| MAN 271 | Multigrade oils for turbocharged and non-turbocharged diesel engines. Oil-drain intervals of 30,00045,000 km |
| MAN M 3275 | SHPD oils for all diesel engines with oil-change intervals of 45,000-60,000 km |
| MAN M3277 | UHPDO oils for all diesel engines with oil-drain intervals up to 100,000 km |
| MAN M3477 | UHPDO oils for all diesel engines with oil-drain intervals up to 100,000 km. Reduced ash, sulfur, and phosphorus content for use in trucks with advanced exhaust-treatment systems |
| M 3271 | Oils for natural gas fired engines |

| ***Mercedes- Benz*** | ***Application area*** |
|---|---|
| MB 227.0 | Monograde oils for turbocharged and non-turbocharged diesel engines |
| MB 227.1 | Multigrade oils for turbocharged and non-turbocharged diesel engines |
| MB 228.0 | Monograde oils for turbocharged and non-turbocharged diesel engines with higher performance than MB 227.0 |

**Tab. 9.9** (Continued)

| *Mercedes- Benz* | *Application area* |
|---|---|
| MB 228.1 | High-performance multigrade oils for turbocharged and non-turbocharged diesel engines. Extended oil-drain intervals up to 30,000 km in medium/heavy duty |
| MB 228.3 | Super-high-performance diesel Oil (SHPDO) for highly turbocharged diesel engines. Extended oil-drain intervals up to 45,000 km in medium/heavy duty |
| MB 228.5 | Ultra High Performance Diesel Oils (UHPDO) for highly turbocharged diesel engines. Extended oil-drain intervals up to 100,000 km in heavy duty (e.g. MB Actross) |
| MB 228.51 | UHPDO with lower ash, sulfur, and phosphorus for use in trucks with advanced exhaust gas after-treatment systems. Extended oil-drain intervals up to 100,000 km in heavy duty |
| MB 229.1 | Multigrade oils passenger car gasoline and diesel engines |
| MB 229.3 | Multigrade fuel-economy oils for passenger car gasoline and diesel engines. Extended oil-drain intervals |
| MB 229.31 | Multigrade fuel-economy oils for passenger car gasoline and diesel engines. Extended oil-drain intervals. Lower ash, sulfur and phosphorus for use in cars with advanced exhaust gas-treatment systems |
| MB 229.5 | Multigrade fuel-economy oils for passenger car gasoline and diesel engines. Extended oil-drain intervals. Fuel economy and engine performance above MB 229.3 |
| MB 229.51 | Multigrade fuel-economy oils for passenger car gasoline and diesel engines. Extended oil-drain intervals. Fuel economy and engine performance above MB 229.31. Lower ash, sulfur and phosphorus for use in cars with advanced exhaust gas-treatment systems |

| *MTU* | *Application area* |
|---|---|
| Oil type 1 | Specifies oil qualities generally corresponding to API-CF, CG-4, or ACEA, E2) for light and medium operating conditions and short oil-drain intervals |
| Oil type 2 | Specifies oils of higher quality levels, corresponding to SHPDO like ACEA E3 for medium and severe operating conditions and medium length oil-change intervals |
| Oil type 3 | Specifies oils with highest quality levels corresponding to UHPDO types like ACEA E4-99 for medium and severe operating conditions of all engines. Such oils achieve the longest oil-drain intervals in MTU engines and provide the highest cleanliness of air intake systems of super charged diesel engines |

**Tab. 9.9** (Continued)

| *Opel/Saab/GM* | *Application area* |
|---|---|
| GM -LL-A-025 | This category describes the gasoline engine oil performance for European GM engine types. The specified SAE 0W- or 5W-XX grades have significant fuel economy benefit compared with 10W-30 standard motor oil. Such oils are suitable for extended drain intervals and backwards compatible to former gasoline engines of Opel |
| GM-LL-B-025 | This category describes the diesel engine oil performance for European GM engine types and specifies also SAE 0W- or 5W-XX grades which are backwards compatible to former diesel engines of Opel |

| *Scania* | *Application area* |
|---|---|
| LDF | ACEA E5 or DHD-1 oils with special long drain field test approval. Oil-drain intervals up to 120,000 km |
| LDF-2 | ACEA E4, E6 or E7 performance level is required for this category. A field trial in an Scania engine of the Euro 3 or Euro 4 generation is required to demonstrate specific performance. These oils are required for use in Euro 4 engines with extended oil-drain interval and Scania maintenance system |

| *Volkswagen* | *Application area* |
|---|---|
| VW 505 00 | Multigrade oils for turbocharged and non-turbocharged diesel engines (indirect injected and normally aspirated). Standard oil-drain intervals |
| VW 500 00 | Multigrade, low-viscosity fuel-economy oils for gasoline and normally aspirated diesel engines. Standard oil-drain intervals |
| VW 501 01 | Multigrade oils for gasoline and normally aspirated diesel engines. Standard oil-drain intervals |
| VW 502 00 | Multigrade oils for gasoline engines, higher aging stability than VW 501 01 |
| VW 503 00 | Multigrade, low-viscosity fuel-economy oils for gasoline engines. Extended oil-drain intervals (“Long Life”) |
| VW 505 01 | Multigrade oils for gasoline and diesel engines, including “Pumpe-Düse” DI diesel engines. Standard oil-drain intervals |
| VW 506 00 | Multigrade, low-viscosity oils fuel-economy oils for DI diesel, except “Pumpe-Düse” engines. Extended oil-drain intervals (“Long Life”) |
| VW 503 01 | Multigrade fuel-economy oils for gasoline turbocharged engines (Audi). Extended oil-drain intervals (“Long Life”) |
| VW 506 01 | Multigrade fuel-economy oils for all types of diesel engine. Extended oil Drain intervals (“Long Life”) |
| VW 504 00 | Multigrade fuel-economy oils with reduced ash content for all gasoline engines. Extended oil-drain intervals (“Long Life”) |
| VW 507 00 | Multigrade fuel-economy oils with reduced ash content for all diesel engines. Extended oil-drain intervals (“Long Life”) |

**Tab. 9.9** (Continued)

| *Volvo* | *Application area* |
|---|---|
| VDS | Oils for heavy-duty diesel engines with oil-drain intervals up to 50,000 km |
| VDS-2 | Oils for heavy-duty diesel engines with oil-drain intervals up to 60,000 km. (Euro 2 engines) |
| VDS-3 | Oils for heavy-duty diesel engines with oil-drain intervals up to 100,000 km |

The European ACEA, North American EMA (Engine Manufacturers Association), and Japanese JAMA (Japanese Automobile Manufacturers Association) are working together on specifications for a worldwide classification system with consistent oil performance. The first specification of this kind DHD-1 (diesel heavy duty) was issued in early 2001. The testing includes a combination of engine and bench tests from the API CH-4, ACEA E3/E5, and Japanese DX-1 categories. In 2002 categories for light duty diesel engines (DLD) also were set up (Table 9.17).

**Tab. 9.17** Global performance classification for engine oils.

| *Category* | *Application area* |
|---|---|
| DHD is a performance specification for engine oils to be used in high-speed, four-stroke cycle heavy-duty diesel engines designed to meet 1998 and newer exhaust emission standards worldwide. Oils meeting this specification are also compatible with some older engines. Application of these oils is subject to the recommendation of individual engine manufacturers | |
| DHD-1 | Multigrade oils for engines meeting emission requirements from 1998 and later. Mack T8, Mack T9, Cummins M11, MB OM 441LA, Caterpillar 1R, Sequence III F, International 7.3 l and Mitsubishi 4D34T4 tests are necessary to qualify such oils to a level, which can be seen as comparable with an MB228.3/ACEA E5 level of the European market |
| Engine oils meeting the minimum performance requirements of Global DLD-1, DLD-2, and DLD-3 are intended to provide consistent oil performance car engines worldwide and may therefore be recommended as appropriate by individual engine manufacturers to maintain engine durability wherever their light duty diesel engine is being used | |
| DLD-1 | Standard multigrade oils for light duty diesel engines. The scope of testing includes several passenger car engine tests out of ACEA categories (VW IDI-Intercooler, Peugeot XUD11BTE, Peugeot TU5JP, MB OM602A) plus the Japanese Mitsubishi 4D34T4. The quality level of such oils can therefore be seen as comparable with B2-98 issue 2 |
| DLD-2 | Standard low-viscosity multigrade oils for light duty diesel engines with extra high fuel economy with basic engine performance like DLD-1 |
| DLD-3 | Multigrade oils for light duty diesel engines tested also in DI turbocharged diesel (VW TDI) with quality level comparable to ACEA B4-02 |

#### 9.1.3.6 **Future Trends**

New generations of engines using optimized technologies advance the concept of tailor-made, special oils.

The continuing optimization of the combustion process to increase the efficiency of gasoline engines has led to the development of direct-injection gasoline engines (GDI engines) which may offer fuel savings of about 20 %. On the diesel side, direct injection with unit pumps or common rail technology using pressure of up to 3000 bar have become the norm. These designs which originate in truck engines, offer power increases of up to 50 % at almost constant fuel consumption.

For trucks, and for passenger cars, reducing exhaust emissions has the highest priority. The thresholds of Euro 2 and Euro 3 (from 2001) could easily be surpassed by use of special exhaust recycling and catalytic converter systems. Euro 4 for passenger car vehicles (since 2005) required many light-duty diesel engines to implement diesel particulate filters (DPF), to meet the rigorously tightened threshold for particle emission (max. 0.025 g km). The introduction of Euro 4 for trucks and buses (October 2006) requires further reductions of $NO_x$ (and particulate matter (which need a further optimized burning process and/or more advanced exhaust gas-purification equipment, for example selective catalytic reduction (SCR) of nitrogen oxides or use of additional soot filter systems in most vehicles. Because these new engines and exhaust gas-purification systems require low sulfur and, most suitable, sulfur-free diesel fuels (below 10 ppm S), new demands will be made of the lubricants used.

Furthermore, the surface treatment of pistons and cylinders has improved to such an extent that topology-specific oil consumption is steadily falling. As illustrated in Fig. 9.4, the sum of these measures has permanently increased oil temperatures and specific oil loading. In addition, oil change intervals have been constantly increased. All in all, three factors will characterize engine oils of the future – fuel efficiency, long oil drain intervals, and low emissions.

#### 9.1.3.7 **Fuel Efficiency**

As a result of strict limits to fuel consumption in the USA (CAFE = Californian Act for Fuel Emissions) and the proven fuel economy effect of low-viscosity engine oils [9.22], this topic is attracting attention in Europe and Asia. As a rule, engine-based savings can reach a theoretical 8–10 % (see Fig. 9.5) [9.23]. As engine oils cannot totally eliminate frictional losses, saving potentials of 4 and 5 % present enormous challenges. However, values of between 3 and 4 % are already possible today.

According to the opinion of experts, reduction in car fuel consumption in urban conditions are achieved by lowering frictional losses during cold start-ups which simultaneously result in less wear and a lowering of viscosity in constant throttle conditions. As Fig. 9.6 shows, there is a plateau-like optimum for the correlative fuel savings in engines with HTHS values between 2.5 and 2.9 mPa s. The critical boundary to high wear conditions are seen by some to be different viscosities. Figure 9.6 shows piston ring wear in boundary to static friction conditions between 2.6 and 2.7 mPa s [9.24]. This threshold is viewed critically by different OEMs and is set individually. The European specification for fuel economy oils contains a span of 2.9 to 3.5 mPa s, whereby

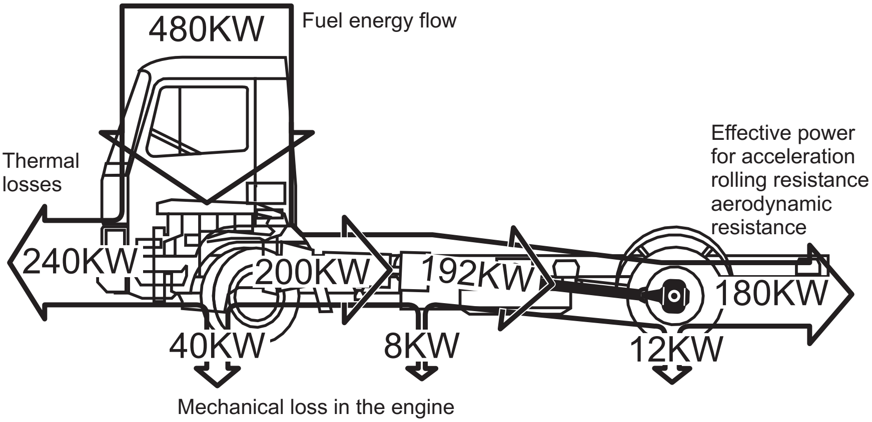

**Fig. 9.5** Total losses in commercial vehicles.

the fuel savings in the M 111 FE test must be at least 2.5 % compared to the reference oil. It has to be remembered that absolute fuel savings figures depend largely on the test method and the reference oil used. Standardized dynamometer tests, which more accurately reflect driving conditions, provide more realistic values than the established bench tests which cannot reproduce all operating conditions.

According to OEMs, HTHS values must not be lower than minimum 2.6 mPa s in all manufacturer's approvals and new engine oil developments because of possible wear between critical material pairings.

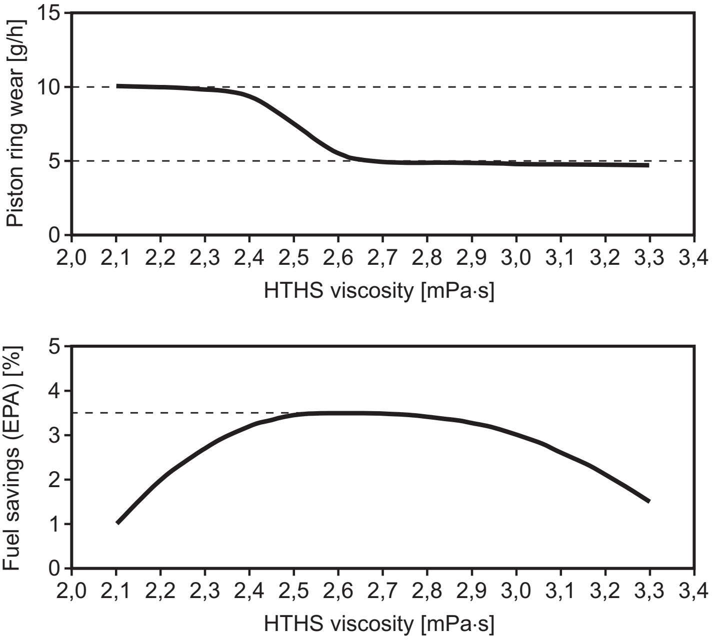

**Fig. 9.6** Fuel efficiency and wear versus HTHS [9.24].

#### 9.1.3.8 Long Drain Intervals

Concerns about higher wear and thus shorter life caused by low-viscosity oils run contrary to the trends of the new generation of engines which are designed to cope with even longer oil change intervals. As stated in manufacturer's specifications (see Section 9.1.3), oil change intervals for cars are currently 30 000 km for gasoline engines and 50 000 km for diesel engines. During the last two to three years, evolution of the oil-drain intervals has stagnated because of increased demands for lower ash, phosphorus, and sulfur content of the oil at the same time as aggravated conditions in the engines. In the future, however, further prolongations of oil-drain intervals can be again expected. As a result of these diverging requirements, future engine oil specifications might contain a whole series of new OEM-specific engine tests.

The radionuclide technique (RNT), as a proven on-line tool, is experiencing a renaissance for examining the effects on wear in various operating conditions such as during running-in or to determine long-term stability. As can be seen in Fig. 9.7 with the example of a new-generation DI turbodiesel, the rate of wear and total wear can be precisely and reproducibly selected for every critical material pairing, for example, in valve trains, in bearings or in piston-cylinder geometries in every engine.

Apart from long-term wear, very high demands are made on oxidation stability and evaporation losses. This strengthens the trend towards synthetic and unconventional oil as the basis for such high performance engine oils. This is well illustrated by Fig. 9.8 with the graphic correlation between evaporation tendency (Noack) and oil consumption. Evaporation losses, illustrated with the example of ILSAC thresholds for GF-2 and GF-3, serve as a generally recognized and reproducible value. A technically realized milestone for fully synthetic engine oils based on present synthetic base oils is a threshold of 5 to 6 %.

The suitability of extended oil change interval oils is tested in thermally stressed test engines which run hot and without oil top-up. The typical indicators of aging like viscosity and TBN are measured. Based on the standardized VW T4 test which is used for current specifications, Fig. 9.9 shows a comparison for modern ACEA oils of different viscosity grades demonstrating the influence of base stocks and evaporation loss.

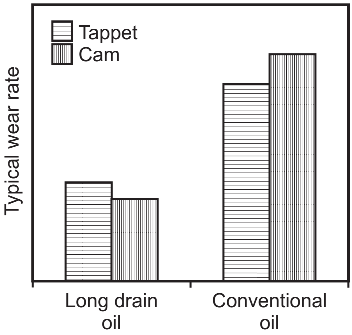

**Fig. 9.7** Wear characterization via radionuclide technique.

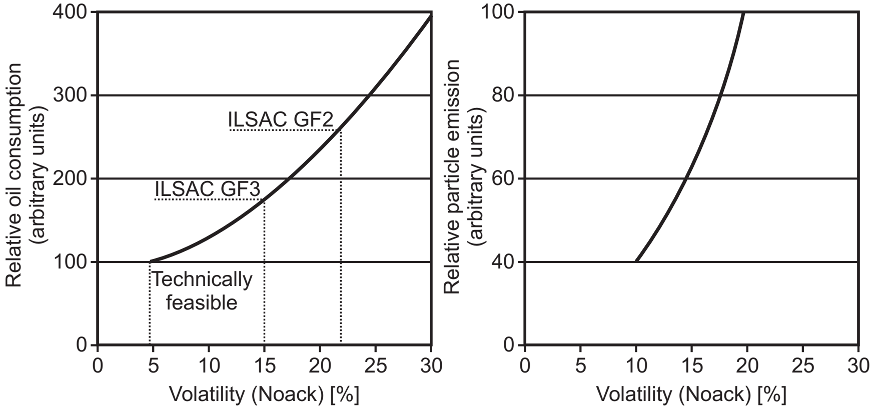

**Fig. 9.8** Oil consumption and relative oil generated particulate emissions versus evaporation loss.

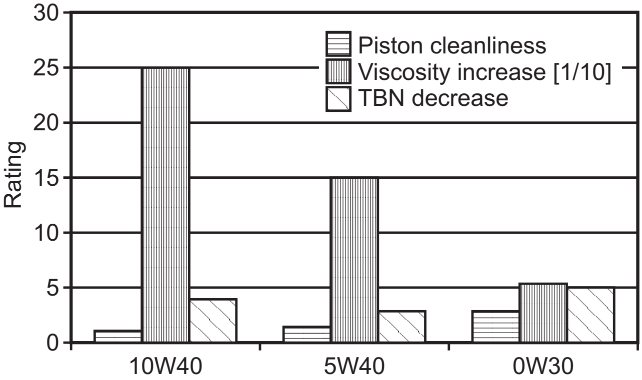

**Fig. 9.9** T4-engine tests for typical ACEA engine oils.

### 9.1.3.9 **Low Emission**

Compared with car engine oils, heavy duty engine oils already achieve drain intervals of 100 000 km. Because of the ever increasing number of trucks on roads, this is a useful contribution to improving environmental compatibility.

Apart from the CO, HC and $SO_2$ emissions which are seen to be caused by the fuel, particulate emissions play a significant role in HD engines. These particulates are a result of incomplete combustion and are a mixture of fuel- and lubricant-based components. As the oil-based particles are largely caused by highly volatile elements in the formulation, evaporation losses have a direct effect on cutting pollutants (see Fig. 9.8). Furthermore, it is assumed that sulfur compounds in diesel fuels will poison the catalytic converters of future Euro 4 and Euro 5 engines. The introduction of 10 ppm sulfur in fuels places the sulfur content of HD engine oils in a new light. Low sulfur and most probably low phosphorus not just lead to some rethinking concerning additives but also to the rejection of Solvent Neutral oils which, as a rule, contain between 0.2 and 1.0 % sulfur. As already with cars, unconventional and synthetic base oils are preferred so that the trend towards low viscosity, fuel economy oils will spread definitively to the heavy-duty sector.

As can be seen in Tables 9.8 and 9.9, during recent years engine oils have already been adapted to the new requirements of engine designs and exhaust gas-purification systems for cars and for trucks (e.g. ACEA C-categories, ACEA E6, MB 229.31, MB 229.51, MB 228.51, MAN 3477). Ash sulfur, and phosphorus content had to be reduced by up to 50 %. At the same time engine designs often needed increased oil performance with regard to engine cleanliness and oil ageing (higher temperatures), soot handling (higher soot content in the oil), and increased wear control (higher torques, lower weights, more soot). These new requirements called for much effort and innovation in the development of new formulations and additive technology. New and better anti-wear systems, more effective dispersant and detergent technology, and more active ageing-protection systems can satisfy the most challenging requirements of today's development of engine oils for low-emission vehicles.

## 9.2
## Two-stroke Oils

### 9.2.1
### Application and Characteristics of Two-stroke Oils [9.25–9.27]

Two-stroke engines are mostly used when high specific power, low weight and low price are key parameters. These engines are thus often used in motorcycles, boats (outboard engines), jet-skis, lawn mowers, chain saws and small vans, whereby the vast majority are found in motorcycles and boats.

Almost all two-stroke engines use total-loss lubrication. The oil is not circulated as in the case of four-stroke engines but added to the fuel. A large part is burnt in the combustion process but about one quarter is exhausted as unburned oil mist. Simple engines as found in older mopeds still use the premix method which involves the operator manually adding a suitable two-stroke oil to the fuel tank at a ratio of about 1:20 to 1:100. More advanced designs use an automatic oil metering system. These either add a constant amount of oil to the fuel or add oil according to engine loading. Typical ratios in such are between 1:50 and 1:400.

In the majority of simple two-stroke, the engine breathes through classic carburetors. Contrary to four-stroke engines, the fresh fuel/air mixture in a two-stroke scavenges the cylinder after combustion. This simultaneous charging and emptying causes about 30 % of the fresh mixture to be exhausted without burning.

This disadvantage, along with the only partial burning of the oil, causes many two-stroke engines to generate comparatively high emissions. In highly populated areas with a large number of small motorcycles, such as in many Asian cities, this leads to severe odour, smoke and noise pollution.

In recent years, these typical disadvantages have been countered by some advances in two-stroke technology. The development of direct or indirect fuel injection has led to significantly reduced emissions and improved fuel efficiency.

Today's engines require correspondingly high quality oils for reliable operation and long life. The principal criteria for the quality of two-stroke oils are:

- lubricity and anti-wear properties
- cleaning function (detergent/dispersant properties)
- avoidance of deposits in the exhaust system
- low smoke
- spark plug cleanliness and the avoidance of pre-ignition
- good fuel miscibility even at low temperatures
- corrosion protection
- good flowing properties.

About 85–98 % of two-stroke oils are base oils with the rest consisting of various additives, which similarly to four-stroke engine oils, provide a series of the above-mentioned characteristics. In principle, all common base oils can be used, ranging from Brightstocks, Solvent Neutral types to fully-synthetic polyalphaolefins. As most two-stroke oils need not perform particularly well at low temperatures, Brightstocks are often used to achieve the desired viscosity. Apart from hydrocarbon types, higher quality two-stroke lubricants often contain various synthetic esters and this is particularly the case for biodegradable oils which were specially developed for marine outboards.

The additives in two-stroke oils are usually matched to the requirements of the engine. As in the case of four-stroke engines, anti-wear additives are included which chemically interact with metal surfaces to protect against wear particularly in boundary friction conditions. Along with the most commonly used zinc dialkyldithiophosphates, non-ash forming types such as dithiophosphoric acid esters of alkyl and aryl esters or phosphoric acids are used.

Dispersant and detergents (DD systems) are added to the oil to keep the engine clean and to avoid deposits in the combustion chamber and around the piston rings. Alkalis or earth alkalis of sulfonates and/or phenolic compounds are often used. Dispersants are often high-molecular-weight compounds which are capable of trapping and suspending contaminants. Examples of these types of substances are polybutene succinimides whose properties result from the chemical bonding of a polar succinimide with oil-soluble polybutenes.

Furthermore, two-stroke oils contain small quantities of anti-oxidants, corrosion inhibitors, defoamers and flow improvers in addition to anti-wear and DD additives.

Low-smoke two-stroke oils contain a significant amount of polybutenes (about 10 to 50 %). These are fully synthetic fluids which are available in various viscosities. Apart from good lubricity compared to mineral oils, these fluids offer much cleaner combustion and significantly less coking [9.28, 9.29].

### 9.2.2
### Classification of Two-stroke Oils

As with four-stroke oils, two-stroke oils are allocated to certain performance groups which provide information about suitable applications. The basis for all the below-mentioned classification systems are a series of laboratory and functional tests, the latter being bench tests performed on the latest generation of two-stroke engines.

#### 9.2.2.1 API Service Groups

The API (American Petroleum Institute) currently lists three categories (Table 9.10) which cover all engines from low-power lawn mowers to high-performance motorcycles. Engine tests are no longer performed because the specified test engines are no longer manufactured. In future, it is planned to replace the API groups with Japanese JASO and global ISO classifications. There are still a number of oils on the market with API classifications because this system was widely accepted in the past.

**Tab. 9.10** API groups.

| *API* | *Application* | *Test engine* | *Test criteria* |
|---|---|---|---|
| TA | Mopeds, lawn mowers, electricity generators, pumps | Yamaha CE 50 S (50 $cm^3$) | Piston seizing, exhaust system deposits |
| TB | Scooters, small Motorcycles | Vespa 125 TS (125 $cm^3$) | Pre-ignition, power loss due to combustion chamber deposits |
| TC | High-performance Motorcycles, chain saws | Yamaha Y 350 M2 (350 $cm^3$)<br>Yamaha CE 50 S | Pre-ignition, power loss due to combustion chamber deposits<br>Piston seizing, ring sticking |

#### 9.2.2.2 JASO Classification

JASO (Japanese Automotive Standards Organization), to which all major Japanese vehicle manufacturers belong, classifies two-stroke oils into three groups, FA, FB and FC (Table 9.11).

**Tab. 9.11** Engine test criteria for JASO classifications.

| *Test engine* | *Test criteria* | *Test parameters* |
|---|---|---|
| Honda Dio AF 27 | Lubricity | Piston ring wear, ring scuffing, piston seizing |
| Honda Dio AF 27 | Detergent effect | Piston ring sticking as a result of lacquering, coking, deposits on piston and in combustion chamber |
| Suzuki SX 800 R | Exhaust smoke | Smoke particles |
| Suzuki SX 800 R | Exhaust deposits | Back pressure in exhaust system |

All three categories use the same test engines and the corresponding performance category is allocated according to pre-determined thresholds. The test results are determined in comparison to an exactly defined, high-performance reference oil (JATRE 1) and published as an Index relative to JATRE 1 (Table 9.12). The key test criteria are the lubricity and detergent effect of the oil as well as its tendency to create smoke and deposits in the exhaust system. The first specification for a low-smoke oil was created with the laying-down of JASO FC.

**Tab. 9.12** JASO performance categories (Reference oil: JATRE 1 = 100)

| *Test criteria* | *JASO FA* | *JASO FB* | *JASO FC* |
|---|---|---|---|
| Lubricity | > 90 | > 95 | > 95 |
| Detergent effect | > 80 | > 85 | > 95 |
| Exhaust smoke | > 40 | > 45 | > 85 |
| Exhaust deposits | > 30 | > 45 | > 90 |

### 9.2.2.3 **ISO Classification**

In the mid-90s, after JATRE 1 oils were tested in European engine tests, it became clear that JASO FC oils could no longer satisfy the latest demands of European two-stroke engines. An series of extended tests which satisfied all demands were thus developed in Europe. In addition to the testing of smoke, exhaust system deposits, lubricity and detergent effect according to JASO, a new category with a 3-h Honda Dio test to quantify improved piston cleanliness and detergent effect was added. The reference oil for all tests was JATRE 1. These new guidelines were created by CEC (Coordinating European Council for the Development of Performance Tests for Transportation, Fuels, Lubricants and Other Fluids) working parties in which European engine and lubricant manufacturers are represented.

The International Standards Organization (ISO) now classifies two-stroke oils into three categories, ISO-L-EGB, -EGC and -EGD. A fourth category (-EGE) is currently being drafted with strong European representation.

The categories ISO-L-EGB and -EGC mirror the requirements of the JASO categories FB and FC while requiring additional proof of piston cleanliness. ISO-L-EGC and -EGD require proof of low smoke similarly to JASO FC. Table 9.13 shows all engine-based evaluation criteria.

**Tab. 9.13** Summary of the ISO categories (Reference oil: JATRE 1 = 100)

| *Test criteria* | *ISO-L-EGB (incl. JASO FB)* | *ISO-L-EGC (incl. JASO FC)* | *ISO-L-EGD* |
|---|---|---|---|
| Lubricity | > 95 | > 95 | > 95 |
| Smoke | > 45 | > 85 | > 85 |
| Exhaust deposits | > 45 | > 90 | > 90 |
| Detergent effect | > 85 (1-h test) | > 95 (1-h test) | > 125 (3-h test)* |
| Piston cleanliness | > 85 (1-h test)* | > 90 (1-h test)* | > 95 (3-h test)* |

* New requirements in addition to JASO FC

### 9.2.3
### Oils for Two-stroke Outboard Engines

Neither API nor JASO or ISO classifications contain quality guidelines for outboard engine oils. These are usually oils whose formulation and characteristics have been matched to the engine technologies which have become established for powering boats. The main difference to other two-stroke oils lies in their additive chemistry. The additives in these oils are all non-ash-forming because these engines all display a marked coking tendency in certain parts of the combustion chamber, such as the ring grooves. If the wrong additives were used, this would lead to major functional impairment and possibly to breakdowns. In principle, the additives have the same functions as in other two-stroke oils but are chemically different. No components are used which can form deposits or ash-like residues at high thermal loads or during combustion. Metal salts such as zinc (anti-wear) or calcium/magnesium (detergents) which are often found in engine oils cannot be used. However, all such oils use typical base oils. The performance categories of two-stroke oils for outboard engines were primarily developed by the American NMMA (National Marine Manufacturers Association). All important American outboard manufacturers belong to the NMMA. Back in 1975, the minimum requirements of such oils were incorporated into the TCW specification. In 1988, a far-reaching revision was issued with the title TCW 2. During the following years, problems were encountered with technologically advanced engines which were run on TCW or TCW 2 oils. This initiated a further tightening-up of minimum requirements, which was released as TCW 3. In 1997, the standards for oil quality were again increased to keep pace with continuing developments in engine technology. The new specification, TCW 3-R

(R= recertified), now includes laboratory tests and tests on five different engines, three of which are outboards. Table 9.14 shows the tests which have to be performed on a newly developed outboard engine oil to achieve TCW 3-R classification. The costs generated by testing a TCW 3-R product development have never been so high. The engine tests alone generate costs of about $ 150 000–200 000.

**Tab. 9.14** Test criteria for NMMA TCW 3-R.

| ***Engine Tests*** | |
|---|---|
| ***Test engine*** | ***Test criteria*** |
| Yamaha CE 50 S | Lubricity (seizures) |
| Yamaha CE 50 S | Power loss due to pre-ignition |
| Mercury 15 HP (2 runs) | Compression losses<br>Ring jamming<br>Piston cleanliness |
| OMC 40 HP | Ring jamming<br>Piston cleanliness |
| OMC 70 HP | Ring jamming<br>Piston cleanliness |
| ***Laboratory Tests*** | |
| Low-temperature viscosity | Limited viscosity at –25 °C |
| Miscibility | Mixing with fuel at –25 °C |
| Corrosion protection | Standard rust tests compared to reference oil |
| Compatibility | Stability after mixing with reference oil |
| Filterability | Flow rate compared to pure fuel |

### 9.2.4
### Environmentally Friendly Two-stroke Oils [9.31]

The ever increasing severity of environmental legislation is also effecting the development of two-stroke oils, especially outboard engine oils. Such ecologically optimized oils often have regionally differing classifications which reflect local environmental legislation and their biodegradability depends on varying minimum requirements. At the international level, the ICOMIA (International Council of Marine Industry Associations) has specified harmonized requirements [9.28]. In 1997, the ICOMIA Standard 27-97 was passed for environmentally friendly outboard engine oils. From a technical point of view, products thus labeled must fulfil at least TCW 3-R as well as offering very low algae, daphnia and fish toxicity and rapid biodegradability as defined by ISO and OECD standards. These oils are based on fully synthetic components with the base oils usually being rapidly biodegradable synthetic esters. By using correspondingly high quality esters, these products are the very best two-stroke oils and can even used for lubricating chain saws. The use of ester-based lubricants combines the highest technical performance with improved environmental compatibility.

## 9.3
## Tractor Oils

Relatively newer generations of construction and agricultural machinery make differing demands on functional fluids. For reasons of simplified servicing but also because of the general wish to rationalize stocks, universal oils were developed which satisfy the various functional requirements of such machines.

These oils should guarantee long machinery life in all manner of climatic conditions as well as extending service intervals and reducing down-time. And the oil manufacturers welcome their possible use in a wide variety of machinery.

These days, two different oil technologies are used which are characterized by their application area. They are universal tractor transmission oils (UTTO) and super tractor oils universal (STOU). Table 9.15 shows where they are used in tractors.

**Tab. 9.15** Application of tractor oil types.

| *UTTO* | *STOU* |
|---|---|
| Hydraulic system | Hydraulic system |
| Gearbox | Gearbox |
| Wet brakes | Wet brakes |
| | Engine |

The demands made on tractor oils have increased sharply in line with advances in vehicle technology and ease of operation. Earlier generations of tractors had manual-shift gearboxes as well as relatively simple rear axle designs. These days, the state-of-the-art is complex hydraulic systems, hydrodynamic drive units (retarders/power splitting) or wet (oil) braking systems. In the past, simple engine oils or low-additive gear oils were used for general lubrication as well as for the hydraulic circuits.

Technical advances in tractors required a significant improvement in operating fluids as they did in the whole automotive area. Apart from big improvements in additive technology to cope with greater mechanical demands, the aging stability of the fluids has increased reflecting dramatically higher specific power outputs and lower oil volumes.

All-season use is now a standard requirement for tractor oils as it is in the automotive area. The result of this is that the viscosity grades (defined according to SAE J 300) and thus temperature ranges have been extended from SAE 15W-30 and 10W-30 to 15W-40, 10W-40 and 5W-40.

The performance of such universal oils as hydraulic fluids corresponds to, at least, HLP and HVLP levels because of the additives included to guarantee universal use.

The use of these products in vehicle gearboxes and wet brakes makes much greater demands on the fluid. Highly-stressed mechanical drive units make great demands on the wear protection and life of the lubricant. As a rule, gearbox suitability is indicated by the automotive API category, normally at least GL-4. However, in-house specifications such as ZF TE-ML 06/07, Allison C-4 or Caterpillar TO-2 may also have to be met. A special challenge to oil formulations is the use of these products in wet brakes which began back in the 1960s. Not least because of safety considerations, such oils must offer high thermal stability and balanced and stable friction characteristics in brakes. Too high or too low friction values can easily lead to excess wear on pads and discs but also to uneven braking and unpleasant brake screeching. Fine-tuning these oils with special friction modifiers is one of the most difficult aspects of developing such oils.

While UTTO oils can satisfy the above-listed applications, their use as engine oils require vastly different additives. Apart from the UTTO additive objectives of friction and wear control, low-temperature stabilizers, oxidation, corrosion and foam inhibitors, engine oils additionally require significant quantities of detergents and dispersants. These ensure engine cleanliness and adequate sludge (in particular soot) transportation. As a rule, normally aspirated engines require, at least, an API CE oil and if a turbocharger is fitted, the oil should be API CF or CF-4. In many cases, tractor engine manufacturers issue approvals for oils after they have successfully completed tests in the corresponding engines. Examples of this are Mercedes–Benz Sheet 227.0/1 and 228.0/1 or MAN 270/271.

The latest generation of tractor oils not only include products with significantly higher performance than in the past but also oils which offer better environmental compatibility. Such products often contain rapeseed or sunflower base oils or synthetic, rapidly biodegradable esters.

Tractor manufacturers now issue their own, in-house fluid specifications which satisfy all specific requirements of the corresponding machinery. Table 9.16 lists several manufacturer specifications.

**Tab. 9.16** Manufacturer specifications for tractor oils.

| *Manufacturer* | *Oil type* | *Specification* |
|---|---|---|
| AGCO Massey Ferguson | STOU | M 1139, M 1144 |
| AGCO Massey Ferguson | UTTO | M 1135, M 1141, M 1143 |
| Ford | STOU | M2C159-B/C |
| Ford | UTTO | M2C86B / M2C134D |
| J I. Case | UTTO | MS 1207, MS 1209, MS 1210 |
| John Deere | STOU | J27 |
| John Deere | UTTO | J20C, J20D |
| New Holland | UTTO | FNHA-2-C 201.00 |

## 9.4
## Gas Engine Oils [9.32–9.34]

The running of combustion engines on gas instead of liquid fuels is nothing new. Gas has long been used to power vehicles and stationary engines. Such engines require a variety of lubricating oils, depending on the type of engine and the operating conditions.

### 9.4.1
### Use of Gas Engines – Gas as a Fuel

Natural gas engines generate significantly less emissions than gasoline powered units. This has led to increasing use, especially in the mobile sector. However, as gas is not as universally available as gasoline or diesel fuel at filling stations, gas power tends to be used for fleets which can be filled centrally, e.g. urban buses, school buses or other short-haul transport vehicles. Many car and truck engines can be adapted to run on gas at no great expense.

Gas engines for stationary applications is particularly interesting in areas where gas is cheaply available. For example, they are often used to power electricity generators or pipeline transmission compressors. Apart from natural gas, landfill gas is being increasingly used these days – rubbish tips and sewage treatment plants often use the gas to drive power generators. In the stationary field, both two- and four-stroke engines are used. Similarly to mobile gas engines, these are based on conventionally-fuelled designs but are tailor-made for their particular application. The operating conditions of vehicle and stationary engines differ greatly. While vehicle engines last for about 5000 h at speeds of up to 6000 rpm, stationary engines can run for decades but at significantly lower speeds. This influences the selection of materials and operating fluids.

Several gases are used in gas engines. Most commonly used, and especially for cars, is natural gas, either under pressure as CNG (Compressed Natural Gas, mostly methane) or as LNG (Liquefied Natural Gas, mostly propane-butane). CNG is by far the most common. As to the use of gas as a fuel, a number of quality criteria have to be considered. Hydrocarbon structure, calorific value, the presence of water and above all, contamination with other gases such as hydrogen sulfide or halogen compounds all have a decisive impact. As opposed to the gas used for mobile applications, stationary engines often have to run on varying gas qualities which depend on local conditions. The design and lubricants for engines burning landfill gases have to be chosen carefully because these gases can contain a number of contaminants and can often be corrosive.

### 9.4.2
### Lubricants for Gas Engines

There is currently no universal, harmonized specification for passenger car gas engine oils. The large variations in operating conditions between mobile and stationary engines generally require oils with different additive packages. A general differentiation is made between high-, medium-, and low-ash types which are recommended by the manufacturers in line with the designed use of the engine. As a rule, gas engine oils are subject to high oxidation and nitration which can accelerate the aging of the oil. Gas-powered cars normally use the same conventional engine oils as are used in gasoline-powered engines using gas are ACEA A3/B4, A5/B5 or C2/C3 qualities as well as APi SH/SJ/SL performance types. Similarly to the automotive sector, multigrade oils are used to cope with variable operating conditions and to guarantee reliable lubrication at low ambient temperatures. As the number of CNG-powered cars increases, there is increasing pressure to develop oils which are especially formulated for these applications. This could mark the beginning of a future, uniform specification.

Special multigrade oils have already been developed for use in heavy diesel engines, with CNG-powered buses being the major application. These have been tested and approved by various engine manufacturers. Examples of these approvals are Mercedes–Benz Sheet 226.9 or MAN M 3271. These oils were tested in bench tests as well as in realistic field trials.

Stationary gas engines can make significantly more complex demands on the oil and this has an effect on their development. While the common ACEA or API bench tests suffice for CNG-powered car engines, laboratory tests on gas engine oils are limited to initial oil screening. The real development takes place in field trials in which engines often have to run for years before they are evaluated. While particular attention is paid to sludge formation, valve train wear and low temperature flowing in car engines, other effects are important in stationary engines. Particularly important is controlling oil aging caused by oxidation and nitration but also pre-ignition which is caused by high levels of ash-forming additives. This problem is most prevalent in two-stroke engines which generally need low-ash oils. Simply because of their long service lives, gas engine oils should protect against valve seat recession and spark plug fouling. These problems sometimes go unnoticed for thousands of hours in stationary engines. As regards running on corrosive gases (caused by sulfur or halogen contamination), special attention must be paid to adequate corrosion protection. Oils for stationary gas engines thus require significantly more development work and have to be better matched to the engine and operating conditions than normal automotive engine oils. The development and application of gas engine oils thus normally takes place in close cooperation between the oil and engine manufacturers who normally issue an approval after successful completion of trials.

## 9.5
## Marine Diesel Engine Oils [9.35]

These lubricants are heavily influenced by the type of fuel used and the design of the engines themselves. A number of similar engines are also used in stationary applications to generate electricity with conventional fuels or with steam power.

### 9.5.1
### Low-speed Crosshead Engines

Low speed diesel engines generating up to 1000 kW per cylinder at 50 to 120 rpm use the crosshead principle (large engines with over 900 mm bores and 3000 mm strokes and 20 000 kW outputs). In crosshead designs, the cylinder block and the crankcase are separate units. Sealing is provided by stuffing boxes and the liners are lubricated with cylinder oils by means of dosing devices. Depending on the bore and stroke of the cylinder, up to 16 dosing devices may be fitted.

The crankcase bearings are lubricated with crankcase oils which are sometimes called System Oils. These oils also lubricate the crosshead bearings and guides. As opposed to cylinder oiling which is a form of total-loss lubrication, the crankcase oil is recirculated.

### 9.5.2
### Medium-speed Engines

Medium-speed engines run at around 200 to 100 rpm. The overall design of these engines is roughly similar to that of vehicle internal combustion engines, they do not use the crosshead design and the crankshaft is connected to the pistons and thus the cylinders with connecting rods. In such designs, the same oil is used to lubricate the cylinder walls and the crankshaft bearings and forms part of a circulation system. These engines are sometimes called trunk piston engines. Figure 9.10 shows a simplified diagram of a low-speed crosshead engine.

These days, marine diesel engines are fuelled by the worst and heaviest crude oil fuel cuts. Residues are gathered for all areas of refining to make low cost fuels. These include vacuum residues, propane de-asphalting residues, heavy solvent extracts from lube refining and other by-products. Refining residues as well as the

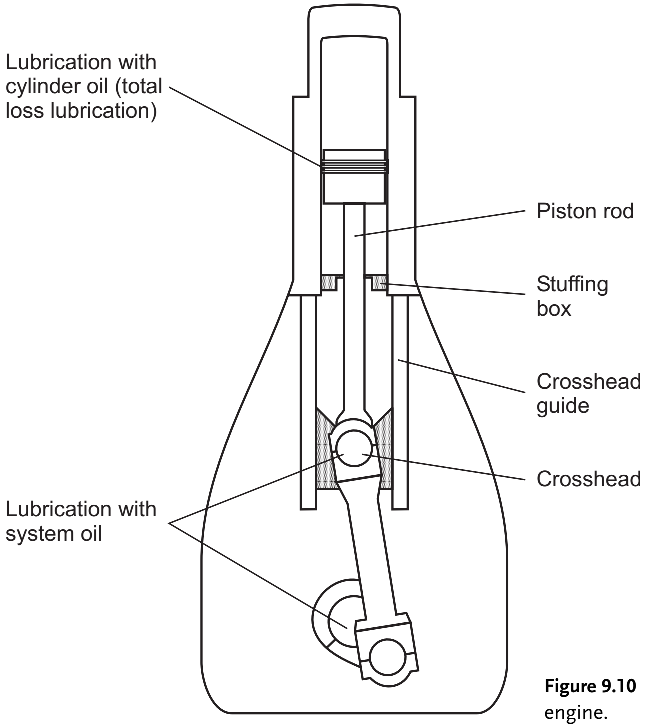

**Figure 9.10** A low-speed crosshead engine.

large share of high-molecular-weight substances provide poor combustion characteristics and create large amounts of deposits. High sulfur contents up to 3 % lead to oil acidification. The formulation of such marine diesel lubricants is considerably influenced by these fuel characteristics. Although the fuel is filtered in centrifuges, high ash and asphalt levels in the fuels lead to significant quantities of solid impurities being formed during combustion.

### 9.5.3
### Lubricants

Cylinder oils in crosshead engines are total-loss products which lubricate the sliding motion of the piston rings in the cylinder liner. To avoid the deposit of combustion residues, these oils must have good dispersant properties. In addition, they must be capable of neutralizing the corrosive acids which result from the high sulfur levels in the fuels. To satisfy these requirements, cylinder oils have a large proportion of over-based components (up to 30 % over-based calcium sulfonates or other over-based components). The alkalinity required for neutralization leads to total base numbers (TBNs) of up to 100. When additives for such oils are being selected, special attention must be given to the good colloidal solubility of the additives in the base oil to avoid precipitation. The large surface areas of cylinders in crosshead engines requires the applied oil to disperse rapidly and reliably and this is achieved by good spreadability.

The stuffing boxes fitted to crosshead engines ensure that the crankcase oils are hardly contaminated by combustion chamber residues and these oils thus contain relatively few additives. Additives to combat thermal oxidation are essential and the neutralizing capacity can be relatively low with a TBN of 5. The contamination of crankcase oil with cylinder oil leads to increased emulsifying capacity which is undesirable. Water separation is therefore another vital characteristic of crankcase oils.

An increase in dispersant and detergent properties caused by the ingress of cylinder oil can also lead to wear problems as the effect of typical anti-wear and EP additives such as zinc dialkyldithiophosphate is thereby diminished.

The type of oil used to lubricate trunk piston engines (combined crankcase and piston lubrication) is also largely determined by the sulfur content of the fuel. Oils with Total Base Numbers of 12 to 40 are common and the neutralizing capacity of the oils is very soon exhausted in some cases so that the TBN of used oils can be much lower.

During the development of such oils, tests are performed on 1- or 3-cylinder Bolnes engines but final results are only available from very time-consuming on-board trials lasting at least one year.

It is certain that significant restrictions on the sulfur content of marine diesel fuels will be imposed in the coming years. This will result in a lowering of the high proportion of over-based components necessary for such oils at present.

# 10

# Gear Lubrication Oils

*Thorsten Bartels*

## 10.1

## Introduction

The gear lubrication oil is a machine component of particular significance for gear and transmission. During operation, the lubricant comes into contact with most of the other inbuild machinary components. Apart from the important function of lubricating the sliding rolling contacts, the oil also fulfils the task of cooling and removing the friction heat generated in the sliding rolling contacts.

A comparison of the expenses and costs relating to the production or manufacture of the machine elements of a transmission, such as the roller bearings, toothed wheels, shafts, seals or gearboxes, shows that, in general, the lubricant is a transmission component which can be relatively simply produced at low cost. However, in order to ensure the reliable and long-term service life, the selection of the suited lubricant, in comparison to the selection of the other machine components, is of decisive significance during the construction and desining phase. For example, a new unlubricated ball-bearing used in a vehicle's altanator reached a service life of four minutes. A ball-bearing of the same type used under the same operating conditions reached a service life of several hours after the application of approx. of 2 mL oil on to the bearing's races.

Various types of lubricant are used in the lubrication of gear and transmissions, whereby a lubrication oil mainly consists of a base-oil and an additive adjusted to the base-oil and the application. The following base-oils are not only used for the lubrication of transmissions but also many other applications:

- mineral oils
- synthetic hydrocarbons (polyalphaolefins)
- poly(alkylene glycols) (homopolymers)
- esters (environmental-friendly oils, mainly on synthetic basis)
- naphthenic oils (rapeseed oil, castor oil)

Apart from the various base-oils, the type and quantity of the additive, which depends heavily on the base-oil, has a significant influence on the function and service life of gear and transmission.

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In principle, any type of lubricant, available on the market, including engine oils, can be used in any gear and transmission, thus ensuring for the moment the their functionality.

However, at this point we would like to warn about the risks involved in this scenario, when some next best lubricant is used without knowing the operating and environmental conditions of the application. The operating conditions include the switching periods and the forces to be transferred, the so-called specific loads of a each single machine component which result from operational speed and the transferred torque. The operation of a transmission causes friction losses in all sliding rolling contacts which, as a result of these friction losses, generating a heating-up. In this case, insufficient or inappropriate lubrication, often combined with inadequate cooling of the friction contact points, leads to a short term failure of the system. The most frequent failure criteria for gears and transmissions are:

- extreme abrasive wear
- early endurance failure, fatigue of components
- scuffing and scoring of the friction contacts

The selection of a lubricant which is not adapted to the respective construction components and the operating and environmental conditions of gears and transmissions can, given an early failure of the machine, lead to maximum consecutive damages up to the breakdown of complete systems. The resulting repair and standstill times of the system lead to unforeseen costs. In most cases, this is the result of trying to save on the relatively low lubricant costs. This example highlights the particular significance of the ubricant as a machine element in gear and transmissions today.

## 10.2
## Requirements of Gear Lubrication Oils

In many areas of machine designing, the torque transfer plays an decisive role. Figs. 10.1 and 10.2 give a general survey over the various transmission types used today.

Each of the above-mentioned types for torque transfer makes specific requirements on the lubricant which must be met to ensure the reliable function of the machines and plants. Thus, the heavily loaded lubricants in hypoid gears require high oxidation stability, together with a very good scuffing and scoring and wear load capacity, due to the high load of the tooth contacts. At the same time, the for-

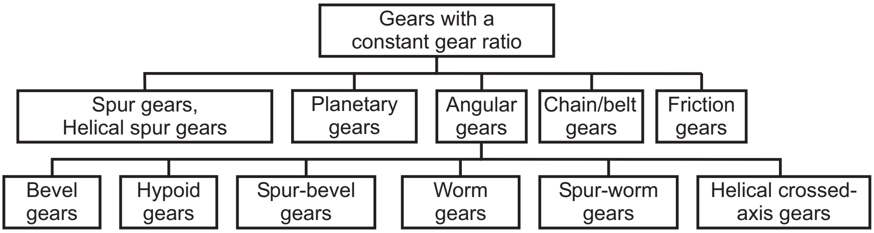


**Fig. 10.1** Gears with a constant gear ratio.

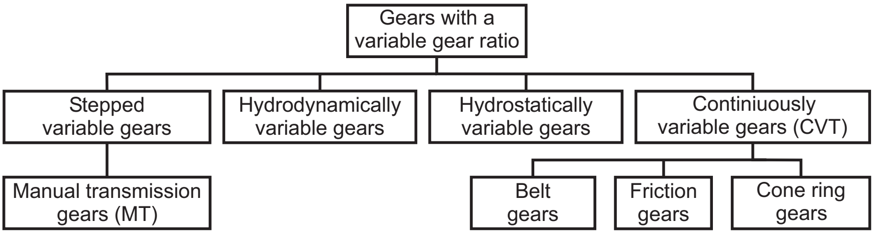


**Fig. 10.2** Gears with a variable gear ratio.

mation of a load-capable and separating film thickness for sufficient lubrication and cooling of the sliding rolling contacts in hypoid gears requires a lubricant with an adequately high viscosity at operating temperature.

On the other hand, lubricants used in hydrodynamic gears, such as torque converters, hydrodynamic wet clutches or retarders, don't need to have a good scuffing and scoring load capacity. However, they must have a high oxidation stability. Due to the viscosity-dependent losses, lubricants which are used in hydrodynamic gears therefore have a clearly lower viscosity at operating temperature in comparison to lubricants used in hypoid gears.

The above-mentioned gear types are used in machine and plant construction with variable exposure time. The requirements made on these gears, closely connected to the requirement made on the gear lubrication oils, should be viewed in an industry-wide manner where the service life and the oil drain intervals are concerned. Figure 10.3 presents the service lives currently required of machines according to the most important industries using gears for torque transfer makes obvious the large differences in the required service lives, from 100 to 500 operating hours for gears in household machines to more than 100 000 hours service life for gears used in paper machines.

The heat development in a gear generates heat and raises the temperature of the oil sump and the temperature in the oil tank. This is of a major significance for the lubricant's service life since it accelerates the oil's aging process and, therefore, can cause a reduction of the oil's service life. The heat development and, thus, the oil temperature, is determined by the type of gear, the transferred torque, the specific load, as well as from the switching periods – permanent or intermittent operation – and from the environmental conditions – use of the gear in warm or cold climates, or in a mobile or stationary application.

Gears that require extended service lives, such as paper machines or presses, the lubricant has to be exchanged according to the mechanical and thermal claim, in compliance with the oil producers' recommendations. Assuming that the average oil sump temperature in such transmissions is approx. 90 °C, the oil should be exchanged in intervals of 2500 hours of operation. An increase of the oil sump temperature of 10 K leads to a 50 % reduction of the service life, reducing the temperature by the same value doubles the oil's service life in general.

Today, gears for which short or medium service lives are required, as for example in passenger cars or mobile hydraulic systems, having life-time fluids. The oil sump temperatures in these transmissions often rise up to 130 °C. Therefore, the use of

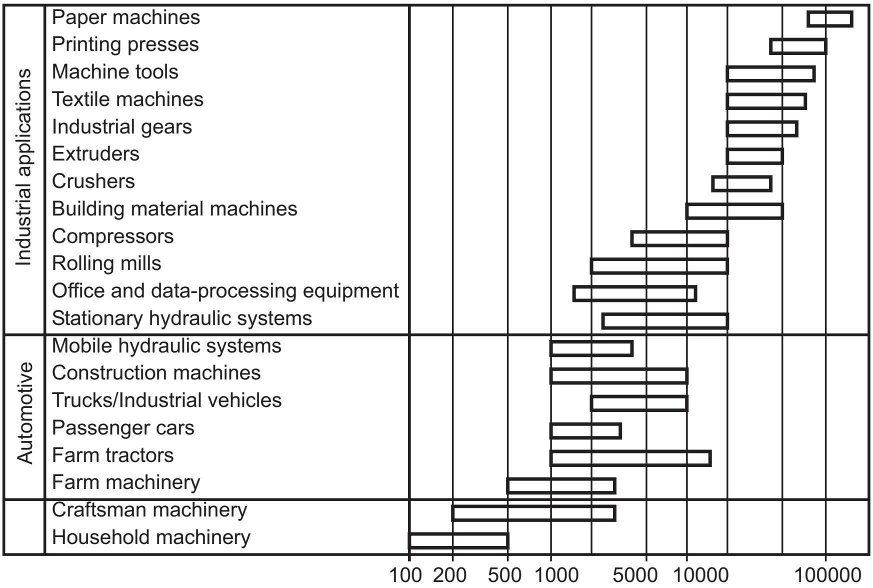


**Fig. 10.3** Service live requirements of gears.

synthetic lubricants is recommended in these applications. Suitable synthetic lubricants, such as poly(alkylene glycols) glycols or synthetic hydrocarbons, reduce the friction losses and, thus, the temperature of the transmissions. In general, the service life and, therefore, the oil drain interval of synthetic lubricants are three times longer than the service life of mineral oils. However, the current prices for synthetic lubricants are also three times higher than those for mineral oils.

According to the market share of mobile and stationary gears, one differentiates, as shown in Fig. 10.3, between ‘gears for industrial application’ and ‘gears for automotive application’. With respect to the gear lubricant, household and craftsman machinery only play a minor role, since only relatively short service lives are required here. In most cases, these machines are not lubricated with oil but with grease.

## 10.3
## Tribology of Gears

There are significant differences between the tribology of gear drives [10.1] on the one hand and the tribology of journal and roller bearings on the other hand, since the lubrication conditions characterizing the sliding rolling contacts in toothed wheels differ from those in journal or roller bearings. As shown in Fig. 10.4, the lubrication conditions in the tooth contacts are the most difficult. Therefore, very often the toothed wheels in a gear have the main requirements upon a lubricant, compare Section 10.3.2. Thus, the gear type is an element of particular concern in

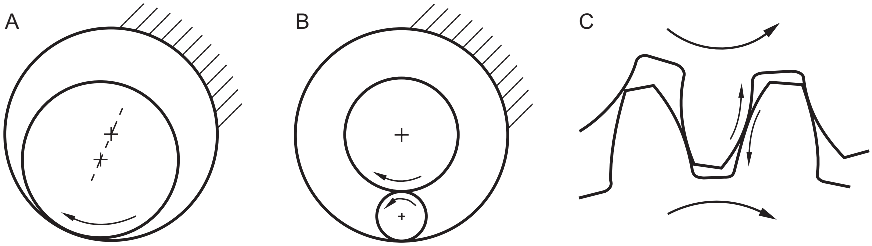


**Fig. 10.4** Schematic overview of narrowing of the gap. (A) Hydrodynamic journal bearings, constant sliding speed at good conformity and good lubrication conditions. (B) Cylindrical roller bearing, relatively high rolling speed at low sliding speed, poor conformity and medium lubrication conditions. (C) Tooth contact lubrication, poor conformity along the tooth engagement line at different sliding velocities in tooth-tip direction and under lubrication conditions inferior to A and B.

the following chapters, whereas the special lubrication requirements with respect to friction and roller bearings [10.2] are not dealt with here in detail [10.3–10.5].

### 10.3.1
### Friction Conditions of Gear Types

#### 10.3.1.1 Toothed Wheels

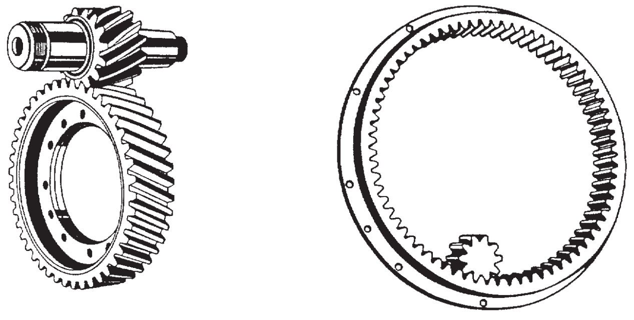
Toothed wheels differ in their gear types [10.6] which appear in gears very often in mixed form. In general, the various gear types or arrangements of the toothed wheels and axles in a gear are decisive for the classification of the gears – compare Figs. 10.1 and 10.2. Figure 10.5 gives a schematic overview of the gear types currently used [10.7, 10.8].

#### 10.3.1.2 Load and Speed Conditions during Tooth Engagement

The different gear types shown in Fig. 10.5 comply with the toothing law. According to its specifically different geometric ratios and the operating conditions, each gear type is limited to a maximum transferable torque. This maximum transferable torque results from the maximum admissible tooth flank load providing the basis for the maximum transferable torque on the one hand and from the rolling and sliding speed ratio within the tooth contact, which leads to the maximum transferable rotational speed of the gear, on the other hand. The product of the maximum transferable torque and rotational speed equals the maximum transferable power of a gear.

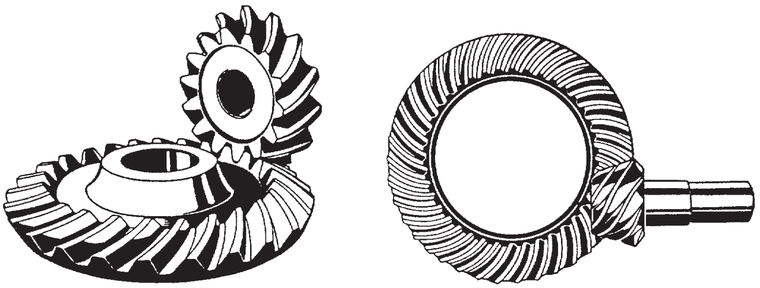
Thus, the main key factors are the sliding rolling contact pressure and the specific load in the tooth contact and the gear's rotational speed, as well as the currently effective rolling and sliding speed ratio in the tooth contact. The distribution of these factors during a tooth engagement is shown in Fig. 10.6 on a non-profile corrected spur gear – the test gear type GF-C used in the Micro-Pitting test (see Chapter 19.3.1.4 [19.31]). In principle, Fig. 10.6 can be transferred to any other gear type in accordance with Fig. 10.5.

The load and surface velocity of the tooth flanks are characterized by continuously changing conditions alongside the entire tooth engagement line. The sum of the



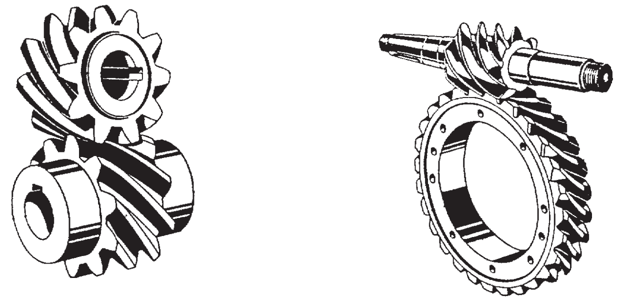
External helical spur gear

Internal spur gear



Bevel gear

Hypoid gear



Helical crossed axis gear

Cylindrical worm gear

**Fig. 10.5** Different gear types.

normal velocity of the tooth flanks equals the effective hydrodynamic velocity, $v_\Sigma$. The difference of the sliding velocity of the tooth flanks equals the sliding velocity $v_g$. Together with the locally effective contact pressure or so-called sliding rolling contact pressure, this sliding speed causes friction losses tangentially to the tooth flank and, thus, an increase in temperature in the sliding rolling contact. Only in the pitch point contact C, as shown in Fig. 10.6, a pure rolling movement can be observed without sliding speed in the whole tooth engagement at that point.

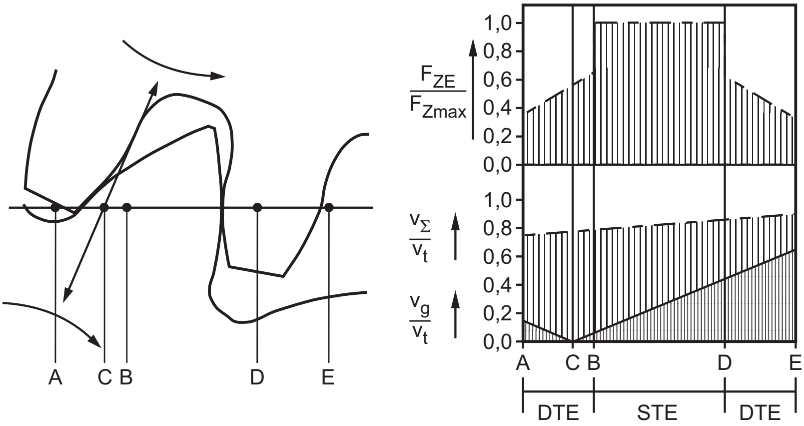


**Fig. 10.6** Load and speed distribution in tooth contact according: A, beginning of tooth engagement; B, beginning of single tooth engagement; C, pitch point, no sliding velocity; D, end of single tooth engagement; E, end of tooth engagement; DTE, double tooth engagement; STE, single tooth engagement; $F_{ZE}$, tooth force within tooth engagement line; $F_{Zmax}$, maximum tooth force within tooth engagement line; $v_\Sigma$, hydrodynamic effective velocity; $v_g$, sliding speed; $v_t$, circumferential speed.

#### 10.3.1.3 **Static and Dynamic Load Distribution within Tooth Engagement**

For the tooth contact shown in Fig. 10.6, a static load distribution alongside the tooth engagement distance can be assumed. In the single-tooth engagement area, the entire load is transferred via one pair of toothed wheels, whereas in a double-tooth engagement area this is ensured by two pairs. Due to the elasticity of the teeth, the transition from a double to a single-tooth engagement and back to a double-tooth engagement does not take place abruptly but nearly linearly.

The tooth engagement is a vibrating system which, according to the geometry and the operational area, shows a dynamic load distribution which differs and sometimes clearly exceeds the static load distribution, as shown in Fig. 10.7. In this vibrating system, the lubricant and, especially its viscosity of the lubricant have an absorbing effect. In this context, it must be mentioned that a larger absorption in the tooth contact which, for example, is achieved by using a lubricant with a higher viscosity, leads to a clearly measurable reduction in the operational noise level, while converting the absorbed energy into heat and, thus, increasing the surface contact and gear temperature.

#### 10.3.1.4 **Lubrication Film Generation within Tooth Contact**

Given a known local toothing geometry, load and speed in the tooth contact alongside the tooth flanks in the tooth engagement area, one obtains the characteristic of the Hertzian pressure [10.10] as the decisive mechanical normal force for a non-lubricated sliding rolling contact in each tooth engagement point, compare Fig. 10.6. If a separating elasto–hydrodynamic lubrication film has been generated

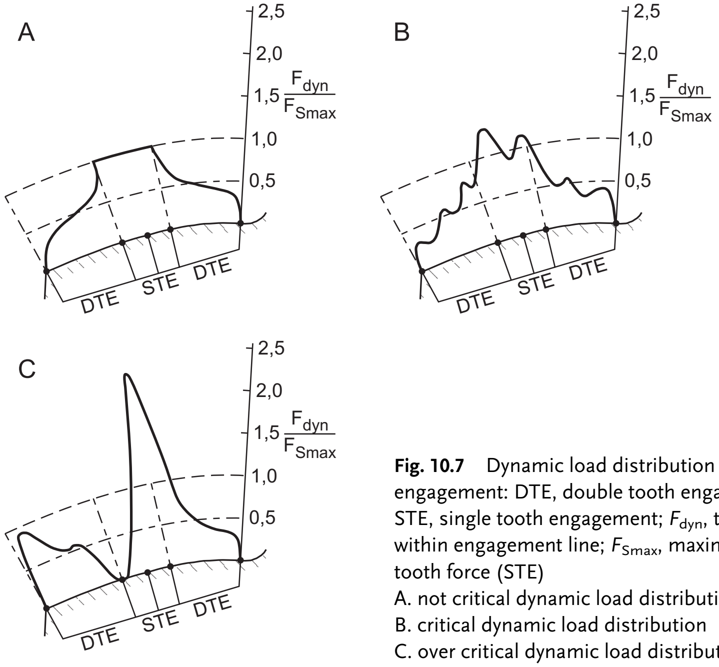


**Fig. 10.7** Dynamic load distribution during tooth engagement: DTE, double tooth engagement; STE, single tooth engagement; $F_{\text{dyn}}$, tooth force within engagement line; $F_{\text{Smax}}$, maximum static tooth force (STE)
A. not critical dynamic load distribution
B. critical dynamic load distribution
C. over critical dynamic load distribution

in the highly loaded tooth contact, see [10.11, 10.12], a pressure, temperature and lubricant distribution according to Fig. 10.8 can be assumed.

A significant key factor for the assessment of the right lubrication and condition is the minimum lubricant film thickness. A sufficient minimum lubricant film thickness is necessary to avoid the metal surface contact of the tooth flanks and to lead the generated heat out of the contact zone by means of friction and absorption. With the minimum lubricant film thickness determined according to [10.12, 10.14], the current lubrication condition in the tooth contact can be assessed. The non-stationary sliding rolling contact conditions are the reasons for the different lubrication conditions in each contact point of the tooth engagement line.

In the pitch point C of a tooth engagement where – as in a roller bearing – a pure roll-off or rolling movement without sliding speed is generated, relatively optimal lubrication conditions can be assessed. The lubrication conditions during the tooth engagement and disengagement, however, are more unfavorable due to relatively high slide speed proportions.

Another factor with negative impact on the generation of a lubrication film in a tooth contact becomes effective during a new tooth engagement of each tooth when stripping off of the lubrication film by the tooth tip of the engaging toothed wheel. The geometry of a toothing's tooth tip (compare Section 10.3.2, Fig. 10.12) is of particular interest. The lubrication film must be re-generated on this tooth tip with each new tooth engagement and breaks with each tooth disengagement. Thus, the tooth contact is characterized by strongly discontinuous operational conditions which, in general, aggravate the generation of lubricating films.

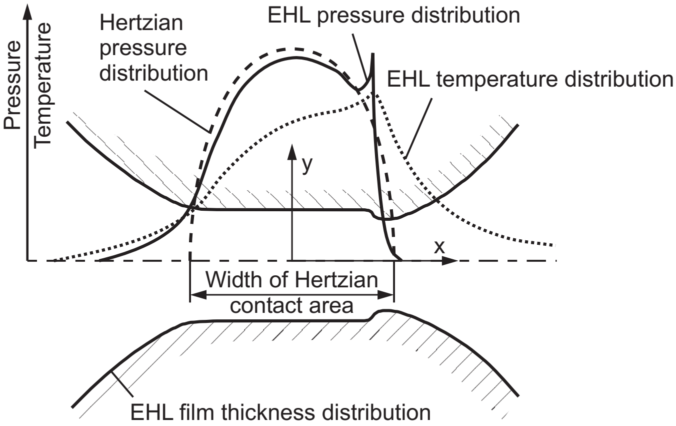


**Fig. 10.8** Distribution of pressure and temperature and lubrication film thickness in the elasto-hydrodynamic contact according to [10.13].

When a gear is characterized by a more lubrication-unfavorable toothing geometry due to the relatively large slide–speed proportions along the tooth engagement line in this type of gear, for example in a hypoid or cylindrical worm gear, these gears are subject to significantly larger friction losses and higher sliding rolling contact temperatures than a simple spur gear with a comparable or equivalent transfer performance with relatively low slide–speed proportions.

During operation, hypoid and cylindrical worm gears, in comparison to spur gears, show a relatively faster increase in temperature, correlated with higher oil sump temperatures. This leads to a lower gear efficiency level and, with respect to the lubricant for these gear types, the development of special requirements.

For example, in hypoid gears one requires an increased scuffing load capacity, whereas in worm gears low-friction lubricants have a particularly positive effect on the toothing's wear and the gear's service life.

#### 10.3.1.5 **Lubrication Conditions**

To avoid tooth damage, thus ensuring the long-term smooth and functional operation of a gear, a detailed observation and assessment of the lubrication conditions in the tooth contacts, already during the designing of gears, is particularly important.

Depending on the local force, contact pressure, temperature and based on the material values of the toothed wheels and the lubrication values of the lubricant, [10.15] calculates a film thickness over the tooth engagement line in a gear. This calculation integrates the lubricant film thickness calculation according to [10.11] and the dynamic lubricant viscosity and the pressure coefficient, $\alpha$ (2000 bar) at the mass temperature of the toothed wheels surface. According to [10.16], the above-mentioned procedure provides good information about the effective lubricant film thickness in the tooth contact. Today, using this method one generally concentrates on the observation of the pitch point C. Due to the fast development and with the help of the current possibilities of electronic data processing, a more complex evaluation of the tooth engagement is only a matter of time and is already established as a standard method today.

The determined lubrication condition in the tooth contact is assessed with the knowledge of the effective surface roughness of the tooth flanks with the value according to [10.17], the specific film thickness is assessed according to the Eq. 10.1.

$$\lambda = h_{\mathrm{min}}/R_{\mathrm{a}} = 2h_{\mathrm{min}}/(R_{\mathrm{a1}} + R_{\mathrm{a2}}) \tag{10.1}$$

where $\lambda$ is the specific film thickness according to [10.17], $h_{\mathrm{min}}$ is the minimum film thickness according to [10.11], and $R_{\mathrm{a}}$ is the arithmetic average mean roughness of the tooth flanks surface of pinion ($R_{\mathrm{a1}}$) and wheel ($R_{\mathrm{a2}}$) according to [10.18].

Many examinations carried out by [10.17] has used the specific film thickness as a guide value for the qualitative assessment of the damage behavior of the tooth flanks. According to the evaluation of numerous observations, [10.17] differentiates between two lubrication regimes.

#### Specific Film Thickness $\lambda > 2$

In general, the tooth flank surfaces are separated by a sufficiently hydro or elasto–hydrodynamic lubricant film thickness. The viscosity of the lubricant is the decisive property of the lubricant. Only little wear occurs in the long term. Surface damage is not to be expected.

#### Specific Film Thickness $\lambda < 0.7$ (Borderline)

Numerous industrial gears are operated in this regime which is also called boundary lubrication. Falling below the critical value leads to an increased risk of surface damage as well as increased wear. In this range, the right lubricant in connection with a suitable lubricant additive system is particularly important.

Figure 10.9 gives a schematic overview of the mentioned lubrication conditions in the tooth contact. If boundary lubrication has reached in the tooth contacts, the tooth flanks surface have to be protected against direct metal-to-metal contact in order to avoid tooth damage.

With sufficiently large film thickness rates ($\lambda > 2$), the effect of the lubricant viscosity is sufficient. In low speed gears connected with low oil sump temperatures and low circumferrential velocities, the lubrication conditions during boundary lubrication ($\lambda < 0.7$) is generally improved by using polar oil compounds, fatty acid or solid matter particles (molybdenum disulfide, graphite etc.). Due to the low speeds, the mentioned compounds can form physical layers on the surfaces and generate a permanent wear-protection.

In high speed gears, with relatively high oil sump temperatures and high circumferrential speed, EP additives improve the lubrication effect during boundary conditions ($\lambda < 0.7$). Depending on the surface contact temperature, the EP additives start a chemical reaction with the surface of the tooth flanks. This reaction leads to the generation of metal soaps which serve as a slide layer, thus preventing metal contact and reduces the heating-up.

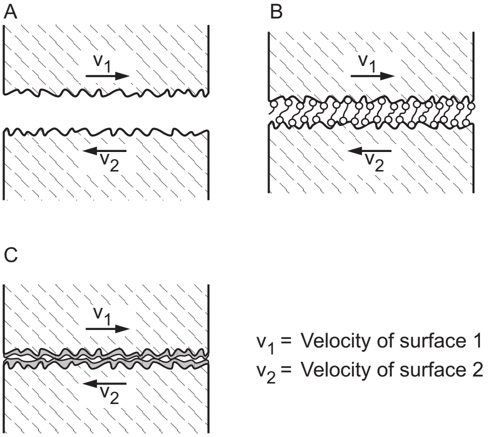


**Fig. 10.9** Lubrication regimes of oils and additives.
A. $\lambda > 2$, hydro or elasto-hydrodynamic lubrication.
B. $\lambda < 0.7$, lubrication by means of physically adsorbed layers.
C. $\lambda < 0.7$, lubrication by means of chemical reaction layers.

### 10.3.2
### Specific Gear and Transmission Failure

During the designing of gears, it must, first of all, be ensured that all sliding rolling contact points, especially the toothings, are supplied with sufficient volumes of lubricant in all operation modes. If this is the case, significant failures in the gears, with the exception of corrosion or the like, will normally not occur in low-loaded modes. Failures in toothed wheels are always the result of excessive load, i.e. a too high local load of the respective material–lubricant combination. When setting up a gear, firstly the material limits of all materials used, including those of the toothed wheels, have to be considered. In the short-therm, the right lubricant can help to avoid damage in the toothings and other gear components, if appropriately coordinated with the materials and operational conditions.

Toothed wheel damage influenced by the lubricant is divided into three categories (Table 10.1).

**Tab. 10.1** Gear damage characteristics.

| ***Damage*** | ***Characteristics*** |
|---|---|
| Wear | Continuous material wear |
| Scuffing and scoring | Sudden metal-on-metal contacts |
| Fatigue | Micro-pitting and pitting |

These types of damage are explained in detail in [10.19].

#### 10.3.2.1 **Wear**

Continuous wear is predominantly observed at low circumferrential speeds and during mixed or boundary lubrication, see [10.20]. A standardized wear calculation does not exist up to now, a rough-calculation approach is given in [10.6]. Figure 10.10 shows an example of a tooth flank with typical wear characteristics. The involute

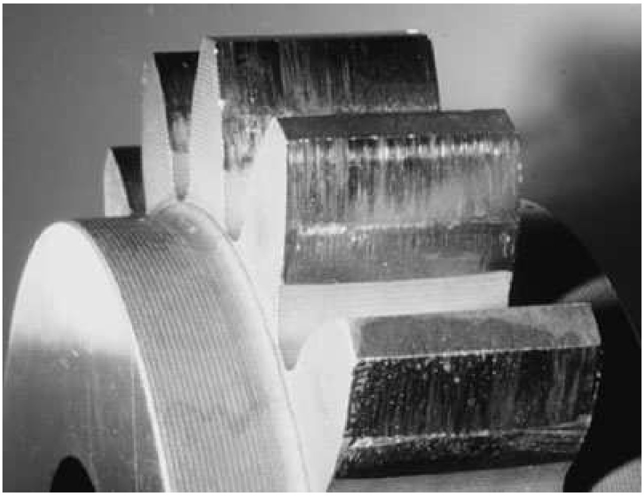


**Fig. 10.10** Tooth flank with typical wear (PT-C-type, pitting test).

flank form shows micro-pitting at the tooth tip and tooth root areas. Continued wear will cause the damage to extend to the middle sector of the tooth flank.

The wear of a toothing can be significantly influenced by hardening the toothed wheels and selecting a suitable lubricant. In this way, nitrated toothed wheels show the lowest wear, whereas pairs consisting of case-hardened pinions and quenched and tempered wheels are characterized by very high wear in comparable operational conditions.

With respect to the wear load capacity, suitable lubricants have a positive effect, when they have a high viscosity and contain wear-preventive or so-called AW (anti-wear) additives. Suitable mechanical–dynamic test methods to assess the wear load capacity are described in Sections 19.3.1.1 to 19.3.1.5.

#### 10.3.2.2 **Scuffing and Scoring**

Scuffing and scoring of tooth flanks occurs predominantly at medium to high circumferential speeds and in case-hardened toothings. According to [10.21, 10.22] the contacting surfaces weld together for a short time. The high sliding velocity of the contact surfaces towards each other breaks this welded joints immediately, thus causing the typical scuffing and scoring phenomenon, see Fig. 10.11.

Accordingly, this damage always occurs in correspondingly engaging flank areas, often at the tooth tip and tooth root, where very high sliding velocities are observed, compare Fig. 10.6. Scuffing is a very sudden damage which can already be caused by one single overload. Sudden scuffing can lead to the total destruction of tooth flanks.

Freshly produced toothed wheel surfaces without run-in are extremely subject to scuffing. Experience has shown that the scuffing load capacity of toothed wheels without run-in reaches only 20 % of that of toothed wheels with well run-in surfaces. By adding to the lubricant suitable so-called EP (extreme pressure) additives, the scuffing load capacity can be improved by a factor of 5 and more. The calculation of the scuffing load capacity is standardized according to [10.23, 19.24].

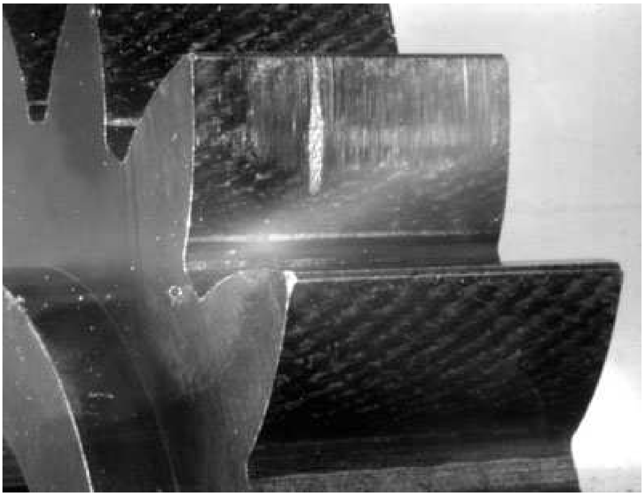


**Fig. 10.11** Tooth flank with typical scuffing damage (A-type, scuffing test).

As shown in Fig. 10.12, a damage mix of wear and scuffing can be caused by the so-called tooth tip during the tooth engagement. This phenomenon can mostly be observed in too highly loaded, non-profile corrected toothings [19.25]. Suitable mechanical dynamic test methods to assess the scuffing load capacity are described in Sections 19.3.1.1 and 19.3.1.2.

#### 10.3.2.3 **Micro-Pitting**

Micro-pitting on tooth flanks can be observed in case-hardened toothings in practically all speed ranges. Tooth flanks with rough surfaces are especially subject to micro-pitting. Micro-pitting develop predominantly in the area of negative sliding velocities or in slip areas below the pitch circle as damage due to endurance failure according to [10.25]. In this case, this leads to the formation of microscopically small fatigue fractures. Given continued damage, these first small endurance fractures, lead to profile form deviations on the tooth flanks. This leads to an increased dynamic and further follow-up damage (pitting, wear, tooth fracture). Figure 10.13 shows a tooth flank damaged by micro-pitting. Today, the influence of the grinding method (Maag, Höfler, Reißauer) on the micro-pitting load capacity examined in [10.26].

A suitable lubricant with a positive influence on the micro-pitting must have a sufficiently high viscosity as well as a suitable lubricant additive system. When selecting a suitable additive system, the operational viscosity or temperature of a gear is of superior significance.

Experience has shown that some lubricant additive systems, for example, at an gear oil sump temperature of 90 °C, have a very high micro-pitting load capacity, whereas it is very low at an oil sump temperature of 60 °C or 120 °C.

With respect to the selection of a suitable lubricant, this must be taken into consideration. Test methods, compare Section 19.3.1.4, help when taking an oil selection in this matter. The determination of the lubricants' Micro-Pitting load capacity

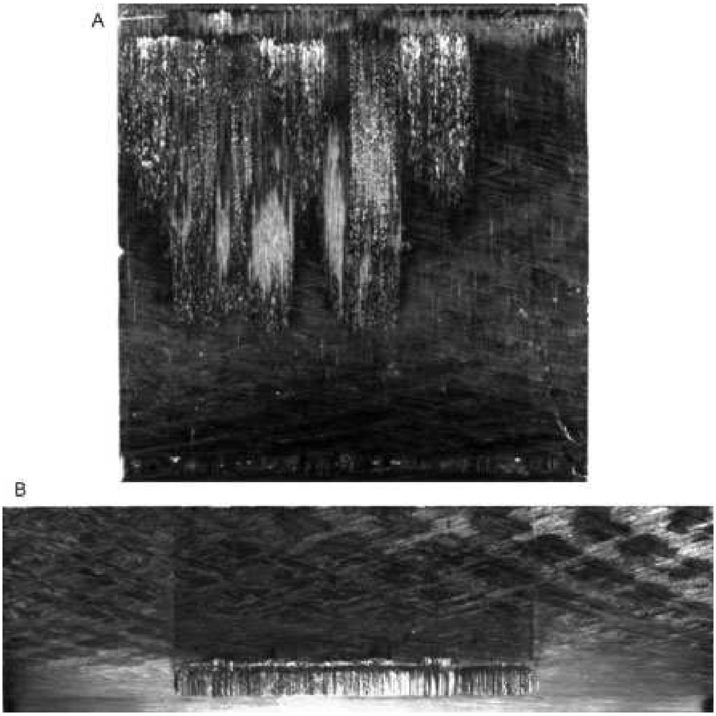


**Fig. 10.12** Pinion and wheel with shave edge at tooth tip (A10-type, high-EP scuffing test). A. Pinion flank. B. Corresponding wheel flank.

takes place using the test methods described in this chapter. A calculation method facilitating the determination of the toothings' Micro-Pitting load capacity is given in [19.33].

**Fig. 10.13** Toothed wheel flank damaged by Micro-Pitting (PT-C-type, Micro-Pitting Test).

### 10.3.2.4 **Pitting**

The damage of pitting is an endurance failure which is observed on both quenched and tempered and case-hardened toothings for all types of gears and at all circumferential speed ranges [10.28]. In case-hardened toothings, it is few or often only one damaged tooth flank which is subject to pitting, compare Fig. 10.14. The remaining tooth flanks do not show any sign of damage. As opposed to case-hardened toothings, quenched and tempered toothings very often show a pitting damage evenly distributed over the entire tooth width as well as over all tooth flanks on the perimeter, compare Fig. 10.15.

Pitting is observed predominantly on the tooth flank centers in the height of the pitch line since this is where the highest load occurs (single-tooth engagement) or the largest stress-cycle amplitudes. Here, the material shows the quickest fatigue.

The hardness of the toothing material and the stress cycles to be tolerated by the material, long-term and permanent, serve as the basis for the long-term and permanent toothing set-up. Examinations in [19.27] from which a calculation method has been developed that has been integrated into [19.24] have shown that, in principle, an influence of the lubricant on the pitting load capacity is possible.

In general, an increase in the lubricant's viscosity has a service-life extending effect. However, it is not always possible and reasonable to increase the lubricant's viscosity in a gear (higher churning losses). Currently, lubricants with a lower viscosity are used more and more often. This trend can be observed especially in gear lubricants in cars and trucks.

The results of [19.27] show that there are suitable lubricant additive systems – above all for lubricants with a low viscosity (ATF technologies) – which have a positive impact on the pitting load capacity of the toothings. Mechanical dynamic test methods to assess the pitting load capacity are described in detail in Sections 19.3.1.3.

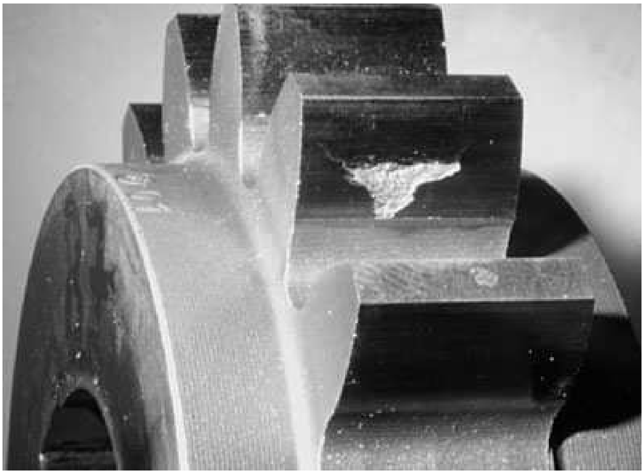


**Fig. 10.14** Pitting on a case-hardened toothed wheel (PT-C-type, Pitting Test).

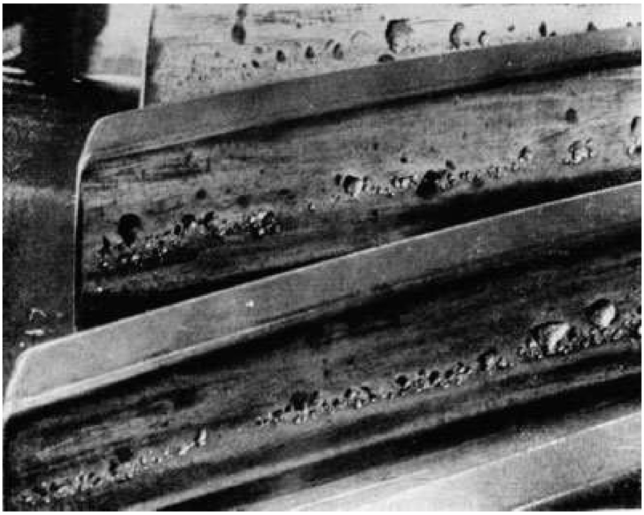


**Fig. 10.15** Pitting on a quenched and tempered toothed wheel.

### 10.3.2.5 **Tooth Fracture**

Up today, tooth fracture has been exclusively based on fracture mechanics which are dependant on the material (steel) of a toothing. The tribology does not have a direct influence on tooth fracture. Tooth fracture is mentioned here only for reasons of completeness. Indirectly, however, the lubricant can facilitate tooth fracture. If, for example, an overheating of the gear or lubricant (accelerated oil aging) leads to lubrication failure or something similar, changes in the metal structure of the toothing due to the overheating will sooner or later cause tooth fracture. Tooth fracture can also occur with teeth which have been pre-damaged by micro-pitting.

## 10.4

## Gear Lubrication Oils for Motor Vehicles

Gear lubrication oils for motor vehicles have to meet the specific requirements of the gear drives, compare Section 10.2. These gear drives transfer the torque from the engine to the drive gears and consist of a gearbox and the drive axle which use differential gears to transmit the power to the wheels. In Europe, mostly, the gearboxes which serve to change the gear ratio are manual transmissions with synchronization. In America and in Asia Pacific, they are mostly automatic or semiautomatic transmissions or so-called constantly variable transmissions. With respect to these Automatics or CVTs, the lubricant is not only responsible for the gear drive lubrication but also for the function-related operation of wet clutches, wet brakes as well as torque converters, retarders, and dual clutches.

Today, it is not possible to meet all requirements of the mentioned types of transmissions with only one fluid. Due to the increasing technological developments together with an increasingly specific component designing with new gear components, e.g. sensorics, the development trend is more and more oriented towards an individualization. Currently, more and more tailor-made individual lubricant solutions are being developed for specific applications, special transmissions and transmission family-types. Like the development of new engine oils, the current development of gear lubrication oils for motor vehicles, as well as, has not yet come to an end and is mainly driven by several factors:

- Vehicle operators welcome an improving reliability of the vehicles and increasing oil-drain intervals to reduce the operative costs incurred.
- The requirements and specifications issued by the motor, transmission and vehicle manufacturers intend to considerably extend the oil-drain intervals. Today's trend is to supply all transmissions with so-called fill-for-life fluids. Today, most of the manual transmissions and axle gears in passenger cars are operated with fill-for-life fluids (> 300 000 km). With respect to automatic transmissions in passenger cars, these long oil-drain intervals will soon be achieved. As far as commercial vehicles are concerned, the required oil-drain intervals of the manufacturers are currently experiencing an extension. It is planned to achieve transmission service lives of 500 000 km, 750 000 km up to 1 000 000 km without drain.
- The legislation in the industrialized countries has an influence on the lubricant formulation mainly due to reasons of environmental friendly fluids and disposal. This means that the use of mineral-based oils is decreasing. Today, environmentally friendly, synthetic and ester-based base oils are often used for reasons of environmental protection. Due to the better thermal oxidation stability, the ester-based oils are used together with PAO and hydro cracked base oils.

Attempts are, furthermore, made to reduce lubricant toxicity, especially in conventional components of commonly used additives. In this respect, the reduction of the chlorine content is of a great importance due to the disposal of the used lubricants. In order to reduce the particles emission, the discharge of oil into the environment and, thus, the oil consumption, future gear lubricants for vehicles are to be tested very exactly with respect to elastomers used in the seal production.

- Driver of motor vehicles expect modern car to ensure an improved performance, a reduced noise emission and a reduced fuel consumption. This trend is expressed in the increasing use of lubricants with a low viscosity together with a reduced fill volume. Furthermore, multigrade oils are currently used on a large scale to considerably reduce the fuel consumption appriciable.

### 10.4.1
### Driveline Lubricants for Commercial Vehicles

Commercial vehicles are divided into light trucks with a maximum capacity of 3 to 6 tons and trucks with a maximum capacity of more than 6 tons. In 2005, the worldwide annual demand for gear lubrication oils for commercial vehicles amounted to approximately 800 000 tons. In the same year, some 2.1 million commercial vehicles were produced in Western and Eastern Europe, which corresponds to drive-line lubricant volume demand of 170 000 tons of gear oils for the commercial vehicles' gearboxes and axle gears for the European market. In 2005, the number of manufactured light trucks amounted to some 1.7 million vehicles. According to their market share, the most important manufacturers are PSA (22 %), Ford (15 %), VW (12 %), Renault (12 %), Daimler–Chrysler (8 %), Fiat (8 %), Daewoo (3 %), Skoda (3 %), Hyundai (2 %), Mitsubishi (1 %), Iveco (1 %) and others (13 %).

Because the fill volume per transmission unit is two to six times larger and amounts to 10 to 30 l, trucks are far more interesting for the oil industry. At the same time, the oil development also confronts a bigger challenge in meeting the requirements made on the fluids used in the commercial vehicles' transmissions. In 2005, the number of manufactured trucks amounted to approximately 0.45 million in all European countries. According to their market share, the most important manufacturers are Daimler–Chrysler (23 %), Volvo (16 %), Iveco (15 %), Scania (13 %), MAN (12 %), Renault (10 %), DAF (9 %) and others (2 %).

Companies such as ZF and Getrag should also be mentioned since they play an important role as transmission manufacturers for a large number of worldwide automotive companies and produce transmissions for both commercial vehicles and passenger cars and deliver these transmissions to the mentioned automotive companies. The special oil requirements of these companies, above all of ZF, have a major influence on the current trend in the development of new fluids for the transmissions of commercial vehicles.

Tables 10.2 and 10.3 list the most important current (2005) specifications issued by the transmission manufacturers and automotive companies, split into manual transmission and rear axle gears. With respect to the specifications, the modified new SAE classes are to be taken into account. Above all, the new classification SAE J2360 is to be mentioned which will replace the old MIL-PRF-2105E. In addition to this, the new SAE 'Automotive Gear Lubricant Viscosity Classification' J306 is also to be taken into account. In this specification the shear loss of the fluids after shear (stay-in-grade) attached much importance.

**Tab. 10.2** Gear oil viscosity classification and gear oil performance level.

| | |
|---|---|
| ISO | ISO 3448 |
| SAE | SAE J300 |
| SAE | SAE J306 |
| MIL | MIL-L-2105 |
| AGMA | |
| MIL | MIL-L-2105E |
| SAE | J2360 |

**Tab. 10.3** Heavy-duty axle applications (commercial vehicles, trucks, buses).

| | |
|---|---|
| Chrysler | MS-9020 |
| DaimlerCrysler | DC 235.8 |
| API | GL-5 |
| GM | 8863370 |
| Ford | SQM-2C9002-AA |
| Volvo | STD 1273.12 |
| Scania | STO 1:0 |
| Renault | RVI TDL |
| MAN | MAN 342 |
| MAN | M 3343 |
| VW | TL 727 |
| ZF | ZF Ecofluid X |

**Tab. 10.4** Light duty axle applications (passenger cars).

| | |
|---|---|
| Chrysler | MS-9763 |
| API | GL4 |
| Ford | M2C-119A |
| GM | 9985476 |
| Clak | MS-8 Rev. 1 |
| Volvo | STD 1273.10 |
| Volvo | STD 1273.13 |

**Tab. 10.5** Heavy-duty synchronized transmissions and transaxle applications (commercial vehicles, trucks, buses).

| | |
|---|---|
| DaimlerCrysler | DC 235.11 |
| API | GL-1 |
| API | GL-3 |
| MACK | GO-J |
| MACK | GO-J PLUS |
| MAN | MAN 341 |
| Volvo | STD 1273.07 |
| ZF | ZF Ecofluid M |

**Tab. 10.6** Heavy-duty non-synchronized manual transmissions (commercial vehicles, trucks).

| | |
|---|---|
| API | MT-1 |
| Eaton | PS-164 |
| Eaton Bulletin | 2053 |

This trend in the development of lubricants for commercial vehicles leads to a significant change in the current ratio between service fill on the one hand and the factory fill on the other hand. The share in service fill and drain of currently 75 % – with a factory fill stake of 25 % – will be reduced to 20 % for the service fill – with a factory fill stake of 80 % during the next ten years.

From today's point of view it can be said that all mentioned specifications constitute a challenge for the development of gear lubrication oils for commercial vehicles. Apart from the very high chemical and physical requirements included in these specifications concerning the oxidation stability (test at 150–160 °C), corrosion, filterability, foam etc., compare Section 10.6, the mentioned specifications include a large number of mechanical–dynamic tests and require very high safety levels with respect to the scuffing and scoring, pitting and wear resistance of the toothings using different, standardized, non-standardized and company-internal test procedures. This decelerates the development of new oils and makes it increasingly complicated and expensive. A particular obstacle in the development of oils for commercial vehicles is the improvement and optimization of the synchronization behavior during shifting operations.

Although many commercial vehicles of American manufacturers still do not use synchronized transmissions, almost all commercial vehicles in Europe are equipped with these transmission systems. The SSP 180 test bench has proven very successful in the oil development for a sufficient synchronization behavior, see also Section 19.5.

One of the benefits for the oil development on the FZG SSP180 is the fact that the development is carried out with regular components at real operating conditions. However, a disadvantage is that almost all automotive companies use different friction material pairs, e.g. steel–molybdenum, steel–brass, and also often coatings such as carbon, sinter or paper. This is aggravated by the use of different component geometries with different conical angles, single or biconical, as well as different operational parameters. According to the type of synchronization, real operating conditions with axial forces, load-increase speeds, surface pressures and inertia are created for the oil development. Often, automotive companies use two or more friction material pairs in the synchronization of a gear family at the same time and expect the fluid developer to fully meet the given operating conditions with the same lubricant or lubricant technology apart from all the other requirements.

The main problem in the fluid development is to adjust the same friction or friction constancy during a required gear service life of approximately 100 000 gearshift operations with different material–lubricant combination without significantly changing the other chemical–physical and dynamic–mechanical properties.

The following example, shown in Figs 10.16 to 10.18, gives an overview of a lubricant development which complies with most of the required chemical–physical and dynamic–mechanical limits, including an excellent gear-shifting behavior in a molybdenum–steel synchronization. This is clarified by the shown individual gearshift operations which show a constant friction coefficient during the entire synchronization time (shift time). The trend shown in Fig. 10.16A, in which the average friction coefficient has been applied during the required service life of 100 000 gearshift operations, remains almost constant and sufficiently high during all gearshift operations. The gear-shifting behavior of the oil with this molybdenum–steel synchronization is to be considered excellent.

Different material combinations such as sinter–steel which is used by the manufacturer in the same gear family displays an inferior synchronization behavior in the bench test. The individual gearshift operation at the beginning of the test in Fig. 10.17 (gearshift 10 000) still shows the desired stable friction coefficient during the entire synchronization time (shift time). However, this behavior changes after a while and experiences a sharp increase in the friction towards the end of the gearshift operations. The big difference between the static friction coefficient and sliding friction coefficient leads to a jolting during the gear-shifting operation which is perceived as an undesirable phenomenon. This effect will increase during the following gearshift operations and results in a clashing of the synchronization, as shown in Fig. 10.18 (gearshift 48 500). Thus, as shows the trend figure in comparison to the molybdenum–steel synchronization, the required number of gearshift operations of 100 000 is not achieved. The test has to be stopped after 48 500 gearshift operations. A second test under the same operating conditions confirms the obtained results. The lubricant technology is rejected by the gear manufacturer as unsatisfactory.

### 10.4.2
### Driveline Lubricants for Passenger Cars

The development of passenger cars is mainly influenced by the worldwide fierce competition of numerous producers. Especially against this background, the competitiveness urges many automotive companies to enter co-operation and mergers according to which identical components and drivelines are manufactured at different locations and for different vehicles. For the oil industry, this means to supply approved lubricants which have to be available in the same quality worldwide or at least in the production locations for gear-filling purposes.

As far as the customers are concerned, the competition's main criteria are the passenger car's driving comfort, design, economy and sportiness which serve as trendsetters to ensure high sales figures. In particular with respect to passenger cars, the mentioned vehicle trends have a certain influence on the engine and total drive-line and, thus, on the fluids used in these transmissions.

A passenger car's sportiness requires a high performance level and a high torque transfer in identical or even smaller spaces for gearboxes. Due to the economy required at the same time, the lubricants are expected to optimize the total efficiency

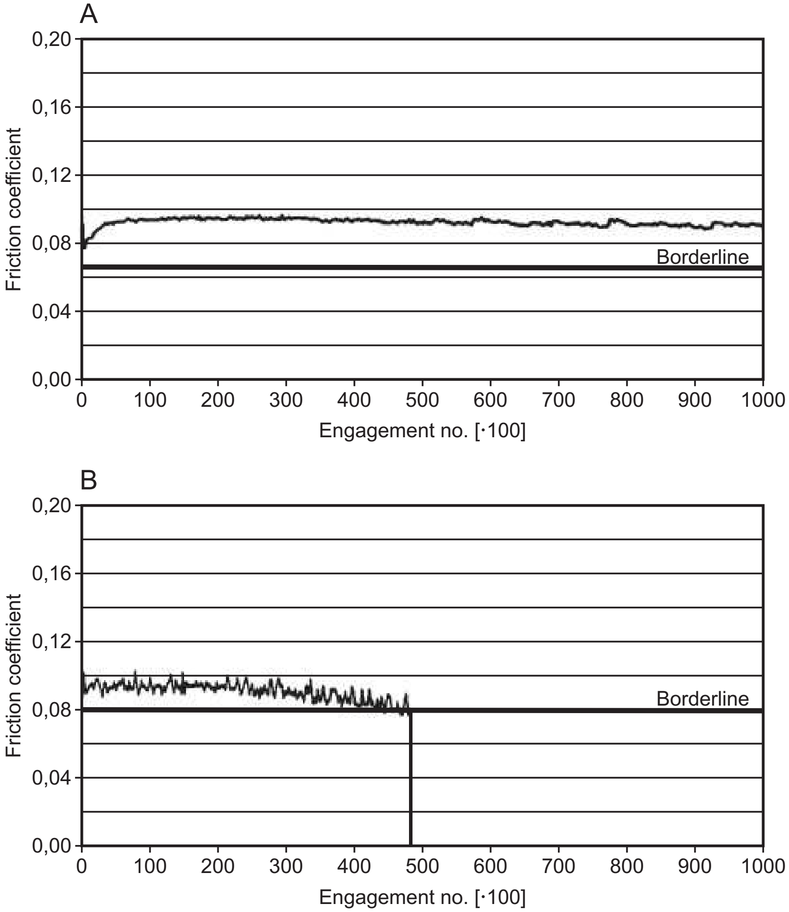


**Fig. 10.16** ZF SSP 180 test run—same fluid with different synchronization materials. A. Upshift, Mo-Synchronizer. B. Upshift, Sinter-Synchronizer.

of the engine and total drive-line without having to be changed during the entire service life of more than 300 000 km.

The best performance costs an increased heat generation in the transmissions which accelerates the aging of the gear lubrication oils. The design of new vehicles leads to the development of carefully selected chassis, often with a low air resistance. This is often connected with an inferior air circulation and cooling of the transmissions. Additional capsules facilitate the reduction of driving-related noise to advance the driving comfort. At the same time, the poor heat dissipation leads to a heat stowing in the capsular spaces. Especially these factors increase the temperature in the vehicle's transmission and enhance the operational conditions, thus accelerates the aging of the fluid.

New passenger cars include transmissions with a higher number of gear stages so that today passenger cars using gear drives with six stages are not unusual. Here, the constructors are more and more required to exploit all technical possibilities of

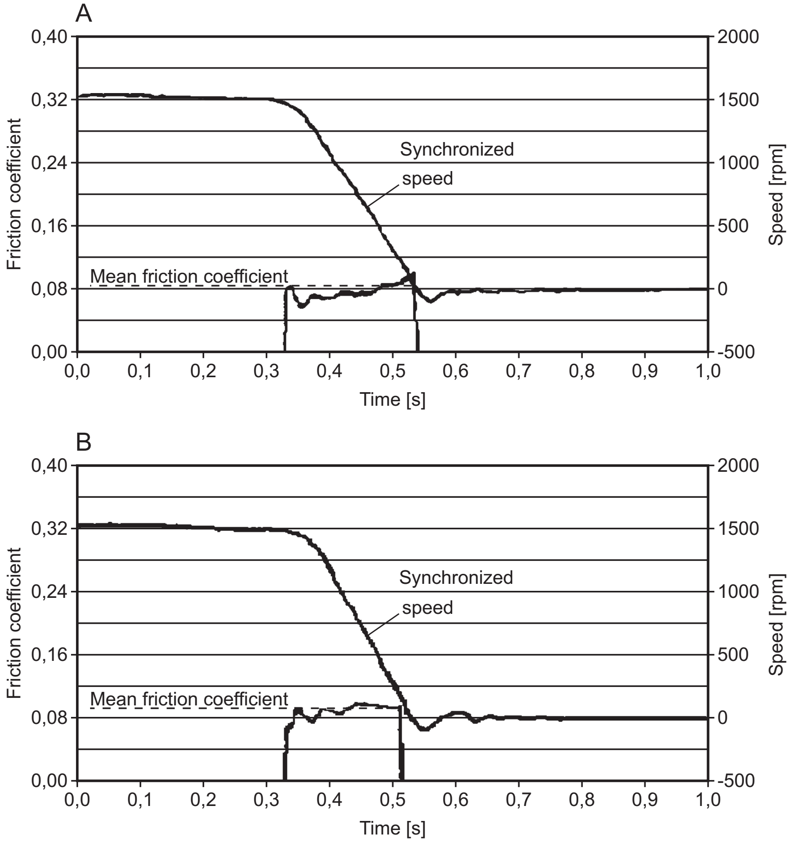


**Fig. 10.17** ZF SSP 180 test run, engagement No. 10 000–same gear oil with different synchronization materials. A. Upshift, Mo-Synchronizer. B. Upshift, Sinter-Synchronizer.

dimensioning and material, always with an effect on the corresponding fluid when new lighter or more solid materials have to be used in the transmission as new friction partners with a friction and wear characteristics unknown so far.

An increasing number of vehicles utilizes automatic transmissions and all wheel drive systems. Today, the necessary control elements and sensor systems consisting of electric components such as meters and sensors etc., are installed in the vehicles' drivelines. Here, the compatibility of these sensible components with the fluid, e.g. with regard to copper corrosion or at high oil sump temperatures leads to enhanced requirements made on the fluid and its development.

Today, transmissions for passenger cars with a synchronized manual transmission has a market share in Europe of almost 90 %. As already shown in Section 10.4.1 for commercial vehicles, all manufacturers of passenger cars make very high comfort-related requirements on the synchronization. The variety of the friction material partners used in the synchronization of passenger cars is constantly

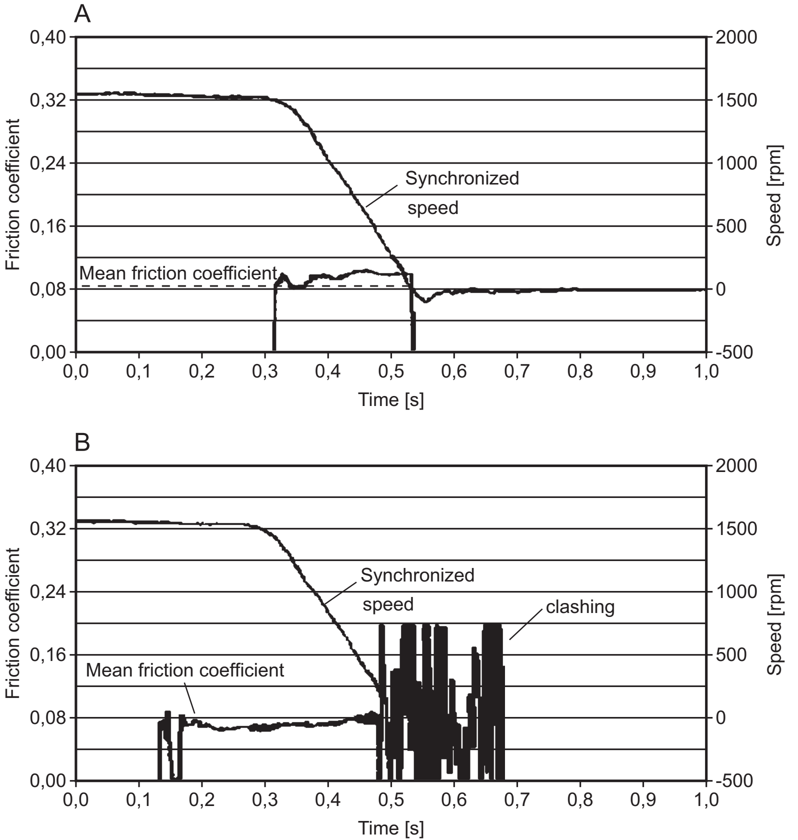


**Fig. 10.18** ZF SSP 180 test run, last engagement same fluid with different synchronization materials. A. Upshift, Mo-Synchronizer, engagement No. 100 000. B. Upshift, Sinter-Synchronizer, engagement No. 48 500.

increasing so that here more and more individual solutions are also required with respect to the fluid. Another requirement on the gear lubrication oils for manual transmission systems, as well as for rear axle gears, is the very high scuffing capacity to ensure the protection of the spur gears' which have significantly a higher sliding ratio than the planetary gears' toothings normally found in automatic transmission, or countineously variable transmissions.

All mentioned development trends, together with the requirements on the gear lubrication oils for passenger cars connected with these, are reflected in the specifications issued by leading automotive companies which continuously determine an increasing number of requirements for the screening, and approval tests. Today, some vehicle and gear manufacturers have even decided to synthetically age or oxidize fresh oils in order to additionally test them afterwards in a synthetically quickly aged condition using the conventional test methods (see Section 19.3.1.7).

Several important specifications for fluids for manual transmissions and axle fluids for gears of passenger cars are listed in Table 10.7 and 10.8.

**Tab. 10.7** Synchronized manual transmission and transaxles (passenger cars).

| | |
|---|---|
| Volvo | STD 1273.08 |
| DaimlerCrysler | DC 235.10 |
| VW | TL 726 |

Worldwide, there are further specifications for off-highway construction machines and commercial vehicles with importance for the fluid development (Table 10.8).

**Tab. 10.8** Light duty synchronized manual transmission (light trucks, passenger cars).

| | |
|---|---|
| API | PM-1 |
| Ford | SM-2C-1011 A |
| Ford | M2C 200C |
| VW | TL 52512 |
| VW | TL 52171 |
| VW | TL 52178 |
| BMW | 602.00.0 |

### 10.4.3
### Lubricants for Automatic Transmissions and CVTs

The most important consumers of fluids for automatic transmission and drive systems, the so-called automatic transmission fluids (ATFs), are off-highway vehicles and machines as well as commercial vehicles such as mainly buses (city buses, intercity buses, coaches and mini/midi buses). In addition to this, ATFs are also used to fill power steering systems in trucks, commercial vehicles and passenger cars.

90 % of all passenger cars on the North American and Asian vehicle market utilize automatic transmission systems. The total estimated ATF volume worldwide for the year 2006 amounts to approximately 1.2 million tons. The total volume breaks down as follows: North America (61 %), Asia Pacific (15.5 %), Europe (12.5 %), Latin America (8.8 %) and Middle East (2.2 %).

Most manufacturers of automatic transmission systems require fluids for the application which meet the listed specifications (Table 10.9).

**Tab. 10.9** Heavy duty automatic transmissions (commercial vehicles, trucks, buses).

| | |
|---|---|
| Allison | C-4 |
| Allison | TES-295 |
| Chrysler | MS-9602 |
| ZF | TE-ML 02 |
| ZF | ZFN 13015 |

These specifications refer to the Asian, North American and European markets and to the business for service fill and re-lubrication of vehicles. Currently, the factory fill is subject to enhanced specifications (Table 10.10).

**Tab. 10.10** Light duty automatic transmissions (light trucks, passenger cars).

| | |
|---|---|
| SAE | SAE J311 |
| TASA | TASA ATF |
| Chrysler | MS-7176 |
| DaimlerCrysler | DC 236.12 |
| DaimlerCrysler | DC 236.20 |
| DaimlerCrysler | Mopar 4+ |
| Ford | M2C 202 B |
| Ford | Mercon V |
| Ford | Mercon SP |
| GM | GM 6418-M |
| GM | Dexron III H |
| GM | Dexron VI |
| GM Opel | B 040 1068 |
| GM Opel | B 040 1073 |
| GM Opel | B 040 2030 |
| VW | TL 52162 |
| VW | TL 52182 |
| Porsche | 040204 |
| ZF | ZFN 13026 |
| ZF | ZFN 13014 |
| ZF | ZFN 904 |

European manufacturers of vehicles and, especially, transmission systems have issued particular specifications mainly for off-highway construction machines and commercial vehicles (Table 10.11).

Automatic transmissions (wet clutches) require hydrodynamic clutches, torque converters and wet brakes. Furthermore, a series of wet friction-clutches and brakes as well as their shifting and friction characteristics play a major role during the automatic gearshift operation. This fact is taken into account in the mentioned specifications. With respect to the friction characteristics, the lubricant, apart from the toothing requirements not dealt with here in detail, is nearly the most significant element.

#### 10.4.3.1 Fluid Requirements for Hydrodynamic Transmissions

The hydrodynamic clutches facilitate an improved and smooth driving and operating behavior, especially with respect to heavy vehicles with a high inertia mass. Here, so-called Föttinger hydrodynamic clutches are predominantly used. A centrifugal pump wheel (rotor) as the working machine and a turbine wheel (stator) as the engine are arranged opposite each other in a confined space in a conical housing. Thus, the exchange of energy between rotor and stator takes place over a short distance via the filled fluid. A hydrodynamic transmission displays a constantly variable operation, whereby the resulting torque independently adapts to the respective load status by changing the rotational speed. When the driving speed remains constant, the rotational speed's slip will increase as the load or output torque increase. The more slip between rotor and stator is generated, the more inefficient operates the torque converter and the more loss and heat will be produced.

Apart from the hydrodynamic start-up clutches, hydrodynamic brakes, the so-called retarders and intarders are used, especially in commercial vehicles. They serve to limit the output speed when driving down long hills in order to reduce the load on the vehicle's. Thus, the retarders protect the vehicle's brakes against overheating and failure. A retarder will also convert the fluid losses generated due to the slip between rotor and stator for the application of the braking torque into heat.

The fluid machines ensure the uncoupling of the drive-line and output torque and, thus, a constantly smooth and comfortable force transfer in the drive shaft. They will, however, only work adequately with slip and are, thus, characterized by significant fluid losses. Apart from the larger weight, this constitutes another reason for the generally higher fuel consumption of vehicles with automatic transmissions in comparison to vehicles with manual transmissions.

During start-up procedures and permanent operation as well as during braking, the lubricant as a force transfer medium in the hydrodynamic transmissions is subject to an extremely quick generation of heat despite installed radiator systems in comparison to gear drives. Due to the function-related fluid losses, hydrodynamic clutches, torque converters and brakes always require low-viscosity, mildly additivated gear lubrication oils with a high oxidation stability. Short-term oil temperatures of more than 160 °C during the operation of a vehicle are not unusual. Especially for these fluid machines, a good viscosity–temperature behavior, corrosion protection, optimal foaming behavior and air release properties are a must. With respect to the viscosity and viscosity–temperature behavior fluids for automatic transmissions are very similar to engine oils.

Therefore, engine oils are frequently used for light transmission applications which often becomes a problem if the transmission is also utilizing wet clutches. On the one hand, the high-additivated engine oils have a low viscosity and extreme oxidation resistance. On the other hand, they cause problems in the wet clutches and brakes with respect to their friction characteristics. To facilitate the distinction between ATFs and the engine oils, ATFs are always dyed with red color.

### 10.4.3.2 Fluid Requirements for Wet Clutches and Brakes

Like in the synchronization, the friction characteristics are of great importance for a comfortable and regular gearshift behavior during the transmission's entire service life in wet clutches and brakes as well. A wet clutch or brake consists of steel disks and separator discs. Such a separator disc or friction partner consists of a disk with an organic or sintered coating, paired with a steel disc. The minimum amount required is a single disc but wet clutches with six and more disc-plates are not unusual. The clutches and brakes are opened and closed hydraulically. Apart from the desired friction characteristics, the oil predominantly serves to ensure the cooling of the friction partners. Often the oil is injected from the interior into the disc-plate via the rotating shaft. Apart from the oil, the coating's compression, the spline and the material are also of great importance in the friction contact, which will not be dealt with in detail.

To adjust a fluid to a required friction characteristic, a test in the wet clutch or, at least, a test of the disc-plates used in the wet clutch is required. Therefore, test benches have been developed especially for this purpose which enable a more precise adjustment of the gearshift behavior to the select the best combination of material and fluid.

A tester which demonstrates the different sliding–friction behavior of oils is the low-velocity friction apparatus (LVFA). Fig. 10.19 gives an example of measured friction behavior for some fluids as a function of the sliding speed.

Tests of original disk-plates can also be done using an SAE 2 rig or a DKA friction test bench. These machines work with given centrifugal masses which reduce the speed of the disk-plate blocks under defined test conditions. The determination of static and dynamic friction coefficient and friction losses is able using a high-resolution measuring technology (see Sections 19.6.3.1 and 19.6.3.2).

According to the definition, the static friction is measured during the tests at low rotational speed differences (< 2 $\text{min}^{-1}$) or towards the end of the gear-shifting operation. In this case, a load-collective-capable variation of the DKA-1B test bench

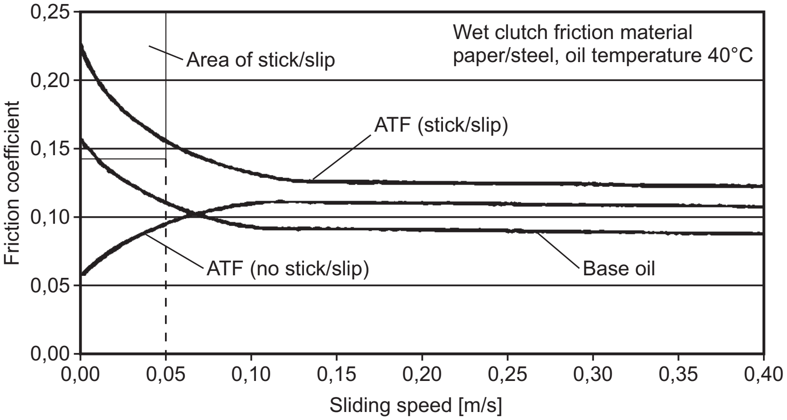


**Fig. 10.19** LVFA (Low Velocity Friction Apparatus, see Section 19.2.6). Determination of the dependence of friction on sliding speed to avoid stick-slip behavior.

is used which allows to determine the load dependence of the static and dynamic friction and friction stability during a certain period of time. Figure 10.20 shows typical friction coefficients of a four-stage load-collective test.

Figure 10.20 compares the friction charcteristics at the 1st and at the 50th cycle of two different ATF technologies. In order to achieve a smooth and soft gear-shifting, it is tried to reach a relatively high dynamic friction coefficient at the beginning of the gear-shifting operation which will decrease during braking and then fade to an even slightly lower static friction coefficient. During the test, oil A at first shows such a desired friction curve in the 1st cycle. It is, however, not able to constantly maintain this behavior, as shows the 50th cycle. The long-term friction coefficient of oil A towards to the 50th cycle sinks as well. In this

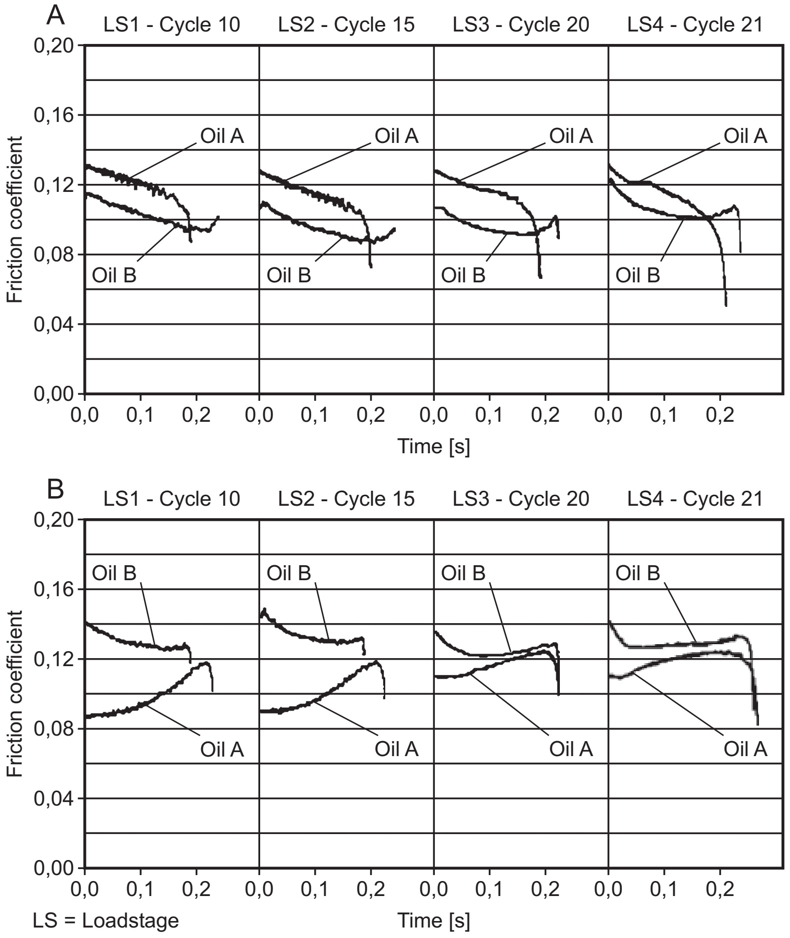


**Fig. 10.20** DKA 1B test run, different ATF technologies. Wet clutch–loadstage test; A. Run-through No. 1 at 4 Loadstages (LS 1 to LS 4 = 66 cycles) analogous to 1st run; B. Run-through No. 50, at 4 loadstages (LS 1 to LS 4 = 3300 cycles) analogous to 50st run multiplied with 66 cycles each.

case, the user will prefer oil B which even after the 50th cycle does not display such a high increase in the static friction and the long-term friction of which shows a relatively constant behavior over all.

To test the gearshift and friction behavior, especially in case of permanent slip of the friction partners, the gear manufacturer ZF has developed another test bench. The so-called GK test bench or CSTCC (continuously slipping torque converter clutch) enables the simulation of almost all real operating conditions. Even slip-controlled clutches of hydrodynamic clutches and torque converters can be simulated using the GK test bench (see also Section 19.6.3.3).

From the lubricant manufacturers' point of view, only the use of the described, very expensive rigs, especially the DKA and GK test benches, will enable a target-oriented ATF development for the adjustment and improvement of the friction properties of wet clutches, slip-controlled torque-converters brakes for a certain lubricant–material pair. Here, the additives as well as the selection and adjustment of the right base oils have a decisive influence. The use of these highly technical testers increases the development expense significantly, thus making the fluid development more expensive.

#### 10.4.3.3 **Fluid Requirements for CVT Applications**

Constantly variable transmissions in motor vehicles enable the operation of a combustion engine along certain preferred characteristic curves in the engine operating map (ignition map). In contrast to all other vehicle transmissions, the CVT enables an ideal alignment of the supply torque of a combustion engine with the request torque of the vehicle. The resulting benefits of the CVT in comparison to all other vehicle transmission system include:

- the exploitation of the engine's at any speed, through the operation along the engine's characteristic curve of the maximum torque (sporty driving); and
- achieving an economical performance through the operation along the engine's characteristic curve of minimum fuel consumption (economic driving).

Figure 10.21 shows the ignition map of a passenger car's 101-kW Otto engine. It displays the two characteristic curves 1 as the graph to describe the curve of maximum torque available and 2 as the curve along the minim fuel consumption.

Only a correspondingly adjusted and optimally controlled CVT enables the driving operation along the shown characteristic curves 1 or 2. Such transmissions require the highest values possible with respect to the operating range of the final control element and the efficiency. Up until today, mainly three constantly variable transmission concepts have proven to be successful to meet these requirements.

#### 10.4.3.4 **B-CVT Push Belt and Link Chain Drives**

For passenger cars with a low or medium engine power of up to 150 kW, the belt drive, mostly a 'Van Doorne' push belt, has been proven successful. Figure 10.22 shows the work principle. The belt wrapes the cone pulleys which have been hydrostatically and axially applied to the drive and output shafts. Thus, the radii of the belt drive's course as well as the gear ratio can be varied.

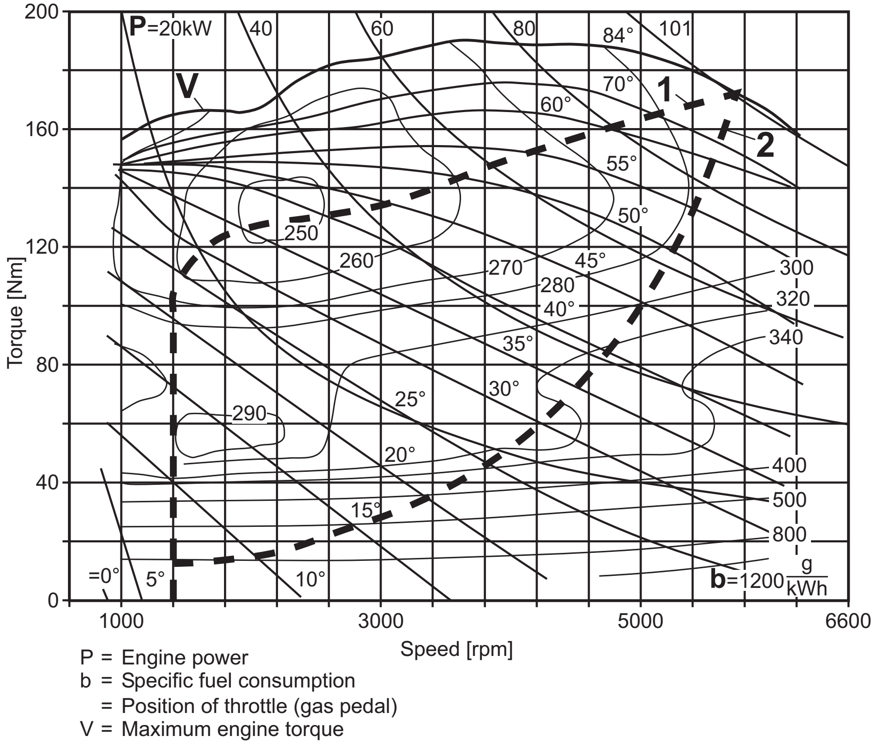


**Fig. 10.21** Ignition map of a passenger car's 101 kW Otto engine.

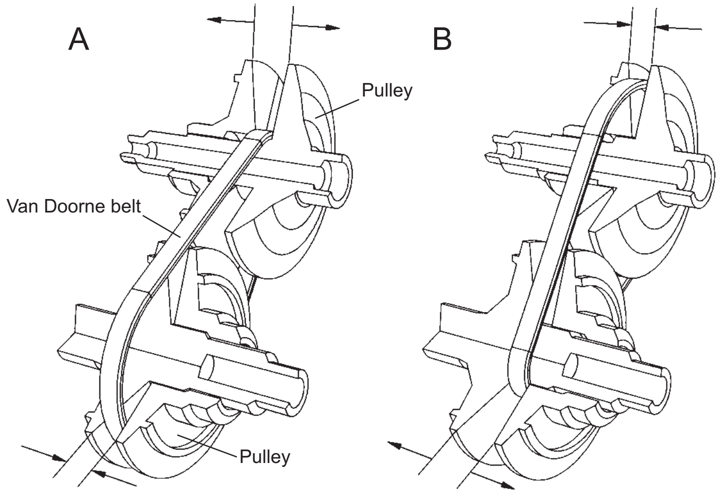


**Fig. 10.22** CVT Variator, principle of a B-CVT (Van Doorne Push Belt). A. Gear ratio in position LOW. B. Gear ratio in position TOP.

For the instationary slide–roll contacts between the belt drive and the cone's pulley surface, the fluid is an extremely important component. The contact points are often subject to very high contact temperatures and mixed-lubrication regime. Here, a very good wear protection of the oil against the extremely hardened surface of the cone pulleys is required. In addition, the pulleys grinded surfaces require from the fluid a very high pitting capacity. On the one hand, a slip of the belt drive due to insufficient contact pressure forces should be avoided by means of control engineering. On the other hand, however, it cannot be avoided totally. Accordingly, the fluid also has to ensure a sufficient scuffing load capacity for these operating conditions.

Nearly all belt drives of vehicles have a toothed wheel gear stage as well as a hydrodynamic start-up clutch or torque converter. In order to increase the driving comfort and reduce the losses, slip-controlled clutches are used. Thus, the same requirements apply for both CVT fluids and ATFs. Currently, CVTs are filled with ATF oils which are slightly modified lubricant variations or have been individually adjusted to the respective CVT. The viscosity, additives and base oils are very similar to ATFs, however, the so-called friction modifiers have great importance.

#### 10.4.3.5 **T-CVT Traction Drives**

As of a certain vehicle performance, the belt drive system's mechanics has reached its limits in power transmission. This limit is approximately reached at an input power of slightly more than 150 kW [420 Nm], i.e. in mid-range passenger cars or limousines. Currently, contineously variable transmission concepts are tested in this respect which are based on so-called traction drives. Figure 10.23 gives a schematic overview of the operation of a traction drive.

The contineously variable adjustment of a traction drive takes place by tipping the transmission or idle wheels which, axially pressed together, roll on the half or full-toroids' races in a force-conclusive set-up. In traction drives as well, the lubricant is to be considered an important construction element and is as significant as the material, the surface treatment and the hardening of the rollers and toroids. Of all transmission types, half and full-toroid gears stand out due to the highest surface pressure and circumferential speeds in the slide–roll point contacts. Surface pressures of 4500 MPa and circumferential speeds of approximately 50 m $s^{-1}$ in traction drives are not unusual.

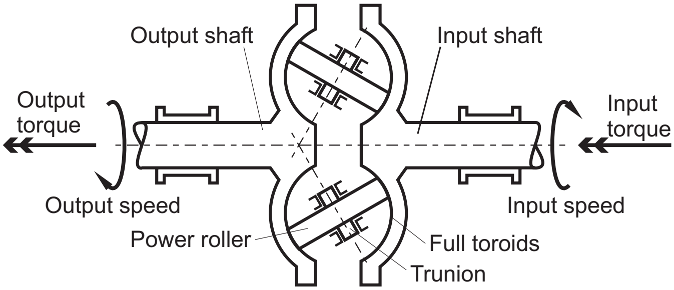


**Fig. 10.23** CVT Variator, principle of a T-CVT (traction drive).

The transferable torque is a function of the normal force in the contact point and the friction coefficient in the slide–roll contact point. In traction drives, this friction depends heavily on the fluid, the material, and the slip. According to the material partners, mainly those fluids are used which enable a high torque transfer performance at the lowest slip possible, thus having a high friction coefficient. In addition to this, the wear and corrosion protection must be ensured.

Adequately added, naphthene-based hydraulic fluids have proven very successful for these applications. However, synthetic oils of the cycloaliphatic hydrocarbon type with a particularly high friction are even more suitable for these purposes. Such oils are also called traction fluids. When contacting traction fluids in transmissions, e.g. in roller bearings and toothed wheels, the higher friction coefficient however, can lead to excessive and undesired overheating.

Another disadvantage of the traction fluids is a relatively low flash point of 130 to 150 °C. Therefore, the very high contact temperatures in the slide–roll contacts cause undesired evaporation losses. For this reason, with the lubricant technologies known today the demand for a fill for life for traction fluids can hardly be met.

#### 10.4.3.6 **H-CVT Hydrostatic Dynamic Powershift Drives**

Hydrostatic dynamic powershift drives are used in agriculture and in tracked vehicles with a usually very high drive power of more than 300 kW. In these transmission systems, planetary gear stages branch the drive power into a closed hydrostatic circuit consisting of a controllable adjustment pump, mostly an axial piston pump, and a hydrostatic constant engine, mostly of bent axis design. The branching of the power and the control of the output speed depending on the volume flow rate takes place through the adjustment of the axial piston pump.

The requirements made on the fluids for this application are limited to the gears, roller bearings and hydraulic systems. For reasons of pumpability, the very good viscosity–temperature behavior of the low-viscosity hydraulic fluids used is of great importance. In addition, these transmissions are to be protected against wear and corrosion using a suitable additive technologies. ATFs and engine oils are also often used in these applications.

## 10.5
## Multifunctional Fluids in Vehicle Gears

Special gear lubricants, so-called multipurpose fluids, are used in agricultural and working machines such as tractors, harvesters, etc.

In these vehicles, the long-term and perfect function of the wet, clutches and wet brakes is to be ensured. The scuffing load capacity of the hypoid gears is to guaranteed using a suitable fluids. Friction, hydraulic and wear requirements have to be met. In order to ensure the driving and working operation even at low temperatures, torque converters have not least to work adequately and safely, even under conditions of permanent slip of the wet clutches. Therefore, multipurpose oils have almost always a low viscosity and stand out due to a very good viscosity–temperature behavior. The presence of water and dirt has a significant impact on these oils, especially in respect to foaming and air release properties (Tab. 10.11).

This is aggravated by the fact that the mentioned requirements often have to manage within only one system. Apart from these complex requirements made on the multipurpose oils which are called UTTOs (universal tractor transmission oils – not for tractor engines), another engine-related performance is usually required in addition. In this case, these oils can also be used as engine oils and are then called STOUs (super tractor oils universal) (Tab 10.12).

Against the background of these requirements it is easy to understand that major manufacturers of tractors and agricultural machines, such as Ford, John Deere and Massey Ferguson, have developed their own specifications for UTTOs and STOUs. The most important of these specifications are listed below (Table 10.13).

**Tab. 10.11** Off-highway vehicles and construction machines (railway, excavators, cranes).

| | |
|---|---|
| Caterpillar | TO-4 |
| Terex | EMS 19003 |
| Komatsu Dresser | B22-0003 |
| Komatsu Dresser | B22-0005 |
| Voith | G607 |
| Voith | G1363 |
| ALLISON | TES-353 |
| ZF | ZF Powerfluid |
| ZF | ZFN 130031 |

**Tab. 10.12** UTTO multi-functional farm and tractor, agricultural machines (hypoid gears, synchronizers, wet clutches, hydraulics).

| | |
|---|---|
| John Deere | JDM J11 D |
| John Deere | JDM J11 E |
| John Deere | JDM J20 C |
| John Deere | JDM J20 D |
| Massey Ferguson | CMS M1127 |
| Massey Ferguson | CMS M1135 |
| Massey Ferguson | CMS M1143 |
| Ford | ESN-M2C-86-C |
| Case | JI Case 1316 |
| New Holland | STD 200 HYD OIL |
| New Holland | NHA-2-C-200 |

| | |
|---|---|
| New Holland | NHA-2-C-201 |
| New Holland | M2C134-D |

**Tab. 10.13** STOU multi-functional farm and tractor, agricultural machines (engine, hypoid gears, sychronizers, wet clutches, hydraulics).

| | |
|---|---|
| Massey Ferguson | CMS M1139 |
| Massey Ferguson | CMS M1143 |
| Ford | ESN-M2C-159-C |
| John Deere | JDM J27 |
| Renk | 530 BW |
| ZF | TE-ML 06 |

## 10.6
## Gear Lubricants for Industrial Gears

From the lubricant producers' point of view, the industrial gear applications differ from vehicle transmissions mainly due to their larger variety and higher number of combinations of toothing types and sizes used, compare Fig. 10.5. Above all, worm gears, planetary gears and helical spur gears with crossed axis are to be mentioned. Gears for industrial application are also different due to a larger variety of possible operating and ambient conditions. They stand out due to much higher torque and performances, connected with clearly larger housing dimensions. At the same time, the gears size also requires larger volumes of lubricant. According to the conditions of the industrial gears use, the service life requirements are clearly higher than those made on vehicle transmissions, compare Fig. 10.3.

With few exceptions, today's gear lubricants for industrial applications are of low aditive treat, and no high performance lifetime lubricants. In comparison to the vehicle gear lubricants, these fluids meet less requirements at one and the same time. With respect to industrial applications, the user has mainly to regularly and punctually drain in accordance with the instructions of the gear manufacturer's recommendation. In this respect, the environmental compatibility of the lubricants used should especially be taken into account.

In opposite to vehicle gears, the type of lubrication in industrial gears can also be very different. Usually, vehicle gears are equipped with an oil immersion or injection lubrication system. According to their operating conditions, industrial gears can, however, be lubricated manually by dropping or pouring, lubricated in an oil sump, oil mist or through an oil injection system. Often one finds larger oil lubrication systems, e.g. in printing presses or paper machines, which stand out due to fills of several hundreds of liters of lubricant. Figure 10.24 gives a schematic overview of a lubrication system commonly used today.

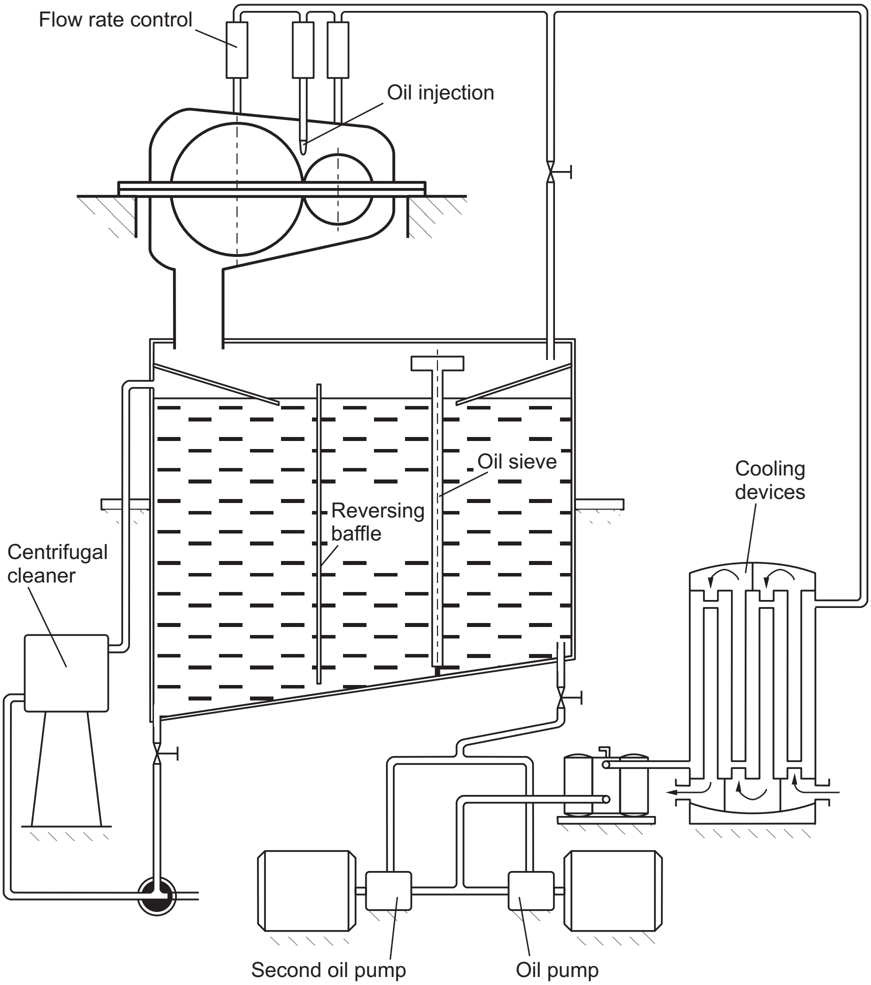


**Fig. 10.24** Schematic of a lubrication system for large industrial gears.

During the lubrication with an oil lubrication system, the total volume of the oil must not be selected in a too low volume so that the air release in the oil can be realized. In this respect, the oil's air separation and foaming properties play a very important role since air is a bad lubricant. The purity of the lubricant during the operation of these systems is another central factor with respect to the gears service life and, thus, the oil's filtration and filterability.

The viscosity-dependent pumpability of the oil, especially at cold temperatures as well as during the start-up of such systems, is also to be taken into account precisely. The wrong viscosity selection of a lubricant can lead to the standstill of the entire system. A guideline for the adequate viscosity selection as a function of load and speed for spur gears and worm gears in industrial applications is given in [10.30].

Of course, the different gear lubricants for industrial applications should meet as many technical requirements as possible. At the same time it is necessary to meet the demand of the system operators concerning longer oil drain intervals. In comparison to the lubrication oils for vehicles transmissions, the worldwide number of specifications concerning the properties of the lubricants for industrial applications is quite small. Important specifications issued by the gear manufacturers and end consumers are listed in Table 10.14.

These specifications cover both simple mechanical–dynamic test procedures and common component testers and test standards with roller bearings and toothed wheels, compare Section 19.2. Apart from these specifications used worldwide, many gear and system manufacturers are currently issuing their own, more sophisticated specifications for their industrial gears with increasing requirements.

The majority of these specifications includes stricter limits in chemical–physical and mechanical–dynamic test standards. Here, detailed information about the lubricant's following properties are required in most of the cases.

**Tab. 10.14** Industrial gear oil specifications.

| | |
|---|---|
| API | GL-2 |
| AMAA | 520 Part 6, 7 |
| AMAA | 520 Part 9 |
| AGMA | 9005-E02 |
| AGMA | 250.04,251.02 |
| David Brown | S1.53101 |
| US Steel | 224 |
| Cincinnati Milicron | P-47,50,53,63,74 |
| GM | LS2 Part 1,2,3,4 |
| Rockwell International | 0-76 |
| DIN | 51517 |
| Flender | Sheet A |
| Winergy | 02.05.2003 |

### 10.6.1
### Viscosity-Temperature Characteristics

According to the ambient and operating conditions, the required viscosity–temperature behavior of gear lubrication oils constitutes a very important requirement during the application. Here, the base oil's properties according to the viscosity range is of great importance. Worldwide, the lubrication oils for industrial gears are subject to the ISO viscosity grade conversions. In the American region as well as in a large part of the automotive industry, the SAE gear viscosity numbers apply. In the American and, mainly, in the Asian Pacific regions, the viscosity ranges for AGMA lubricant are used most often.

The viscosity index (VI) is a dimensionless number that is a measure of how much the viscosity of a fluid changes with temperature. The larger the number, the less the viscosity changes with temperature. Although all fluids become less viscous (thinner) with increasing temperature, the viscosity of a fluid with a higher VI will not change as much as that of a fluid with a lower VI. This resistance of viscosity to temperature changes has important real-life consequences. A fluid with a high VI can be used all year, eliminating seasonal fluid changes. A high-VI fluid will enable cooler operating temperatures, avoiding unscheduled shut-downs because of overheating; it will also enable efficient and smooth operation at higher temperatures and enable start-up at lower temperatures. This increases the temperature operating range of gear equipment. High-VI fluids have better low-temperature properties than standard gear or hydraulic fluids. This means that at low temperatures a fluid of a given grade (ISO VG) will have a viscosity similar to that of a lower (thinner) grade fluid. One can still identify a fluid that meets high-VI (multigrade oil) performance levels by matching the characteristics.

### 10.6.2
### Fluid Shear Stability

Another important aspect of fluid performance is viscosity and VI stability under the operating conditions used. Fluids can be made with high-VI oils (for example expensive synthetic oils) and/or by adding polymers called viscosity index improvers (VII) to the formulation. VIIs are a common and well-tested technology first used to make multigrade engine oils in the 1940s. They are still used for this purpose and to make high-VI oils for a wide range of other applications, for example transmission fluids, gear oils, and hydraulic fluids. Modern gears and hydraulic systems apply great force to their fluid. The base oil and most other additives will not be affected by this force, but in some circumstances the VII might be. In the worst case, the forces will break (shear) the VII into smaller pieces, resulting in reduced fluid viscosity and VI. Thus the benefits of a high-VI fluid could be lost in operation. Modern VIIs which are resistant to breaking (shearing) because they are of low molecular weight are now commercially available, thus eliminating this problem.

### 10.6.3
### Corrosion and Rust Protection

Especially corrosion and corrosion protection play a very important role with respect to lubrication oils for industrial gears. Due to the required extended drain of industrial gears, severe corrosion can facilitate an unexpectedly quick failure of bearings, toothed wheels and other important gear components. This has to be avoided by using a protecting lubricant combined with a suitable additive. Therefore, today's specifications include various tests, compare Chapter 18 and Section 19.9, to determine the corrosion protection properties of the lubricant for iron–steel, copper and other yellow metals. Some industrial applications, i.e. off-shore applications, are aggravated by the presence of salt water in the gear, thus requiring special test methods.

### 10.6.4
### Oxidation Stability

Oxidation stability reduces the ageing of an oil. The ageing behavior of a lubricant is connected with a change in viscosity and with an increase of the acid number. Using certain test methods it is possible to determine the aging behavior of the oil by changing the acid number. The acid number serves to determine how many milligrams of the alkali KOH are necessary to neutralize acids in the oil. Today, sensitive gears are often tested on a regular basis. Should the acid number change noticeably, the oil producers recommend an oil drain.

### 10.6.5
### Flash Point and Pour Point

The flash point is the lowest temperature at which volumes of vapor evaporate from the oil sample to be tested under defined conditions to such an extent that together with the air over the fluid level they will form an inflammable mixture. A low flash point of an oil causes high evaporation losses insofar that high local oil temperatures may occur. The pour point is identical to the temperature which is 3 K above the temperature at which the fluid is no longer flowable under the test conditions. According to the application, the specifications almost define flash and pour points.

### 10.6.6
### Demulsibility and Water Separation

Water is a poor lubricant and facilitates corrosion and should, therefore, be kept away from the lubricant. Often, however, the water cannot be prevented from penetrating the lubricant. Once the water proportions in the oil have exceeded a certain limit, the only possibility left is to drain the oil or to separate oil and water. In large lubrication systems, the water will sink to the bottom of the oil tank due to the higher specific weight and can be released through a drain cock. This, however, requires a good water separation behavior of the lubricant.

### 10.6.7
### Air Release

Naturally, air is mixed into the oil in each gear. Since air is also a poor heat conductor, the air should be separated from the oil as quickly as possible in order to minimize the air's proportions in the oil.

### 10.6.8
### Paint Compatibility

Because of the improved protection and extended service life, the interiors of large industrial gearboxes are also coated with paint which can be attacked, softened and stripped off by oils. This applies especially for one-component paints whereas two-components paints are generally resistant against polyglycols.

### 10.6.9
### Seal Compatibility

Like paints, seals often have organic components which can be attacked by lubricants. The risk of an attack rises considerably with an increase in the oil temperature. The elastomer compatibility tests of important specifications for the combination of lubricant and seal include both static and dynamic seal tests which, however, will not be dealt with here in detail.

### 10.6.10
### Foaming

Especially contamination cause the development of partly large quantities of foam in oils. This foam development can reach dimensions so that the foam escapes the gear through ventilation, thus resulting in a contamination of the environment. In order to avoid this, only those oils should be used which in their fresh condition stand out due to a very good foaming behavior.

### 10.6.11
### Miscibility with Mineral Oils

If possible, different base oils used in industrial gears should not be mixed since it must be expected that the mix will always assume the properties of the inferior product. However, the fact that mixing two base oils leads to unexpected reactions is even more critical. For example, it might happen that mixing mineral oils with polyglycols results in a significant increase in viscosity – as a result of polymerization – which makes it more difficult to pump the mixture through narrow tubes and might lead to the failure of the gears affected due to starved lubrication.

### 10.6.12
### Environmental and Skin Compatibility

Today, especially the environmental and skin compatibility is considered very important by a growing number of gear manufacturers and system operators due to the effect on health and environment. Numerous specifications require compatibility tests the design of which can, from case to case, be expensive and cost-intensive due to the health and environmental impact, see chapter 7.12.

### 10.6.13
### Open gear drives

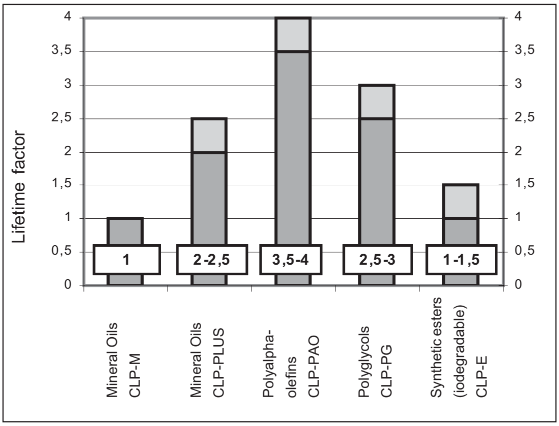
Open gear drives can often be found in the cement industry, the so-called milling gears, in rotary kilns in the iron and steel industry, in coal-burning plants or in open-cut mines. This open large gears are often lubricated with sprayable adhesion lubricants. Apart from the requirements made on the load capacity and wear protection, priority is also given to the adhesion properties, sprayability, pumpability and corrosion protection. In the mentioned systems, solid-matter so-called compounded lubricants have proven to be successful as additives to such adhesion lubricants. Today, the health and environmental impact which can occur in oil mist and spray lubrication systems as well as the direct losses due to oil-containing waste air minimize careful oil separation processes. Open gears can also be lubricated manually or with the help of dosing pumps in regular intervals with high-viscosity oils.

## 10.7
## Cost-to-benefit Ratio of Gear Lubrication Oils

From the very beginning, design and component selection focused on cost, and on cost-effective engineering. The objective of a study reported elsewhere [10.31] and the evaluation of the results obtained [10.32] was to compare the performance of synthesis-based industrial gear oils with conventional, mineral oil-based gear lubricants of CLP standard (DIN 51517) with regard to oxidation stability (oil ageing stability) and friction characteristics. This could ultimately lead to a significant increase in the service life of the lubricant because of extended drain intervals correlating with a much higher mechanical–dynamic performance level.

**Tab. 10.15** Industrial gear oils, a comparison of base fluid data.

| ***Fluid type*** | ***CLP-M*** | ***CLP-PLUS*** | ***CLP-PAO*** | ***CLP-PG*** | ***CLP-E*** |
|---|---|---|---|---|---|
| Type of fluid | Mineral | Mineral | Synthetic | Synthetic | Synthetic biodegradable |
| Base oil of the fluid | Mineral oil | Mineral oil | Polyalpha-olefins | Polyglycols | Synthetic ester |
| Viscosity at 40 °C $[\mathrm{mm}^2\,\mathrm{s}^1]$ | 220 | 220 | 220 | 220 | 220 |
| Viscosity at 100 °C $[\mathrm{mm}^2\,\mathrm{s}^1]$ | 18.4 | 18.4 | 25.7 | 34.7 | 28.0 |
| Viscosity index | 95 | 95 | 150 | 200 | 160 |
| Flash point [°C] | 230 | 230 | 260 | 230 | 280 |
| Pour point [°C] | 18 | 18 | 54 | 33 | 48 |
| Water solubility | No | No | No | Partly | No |



Rotary Bomb Oxidation Test
RBOT – ASTM 2272 (2112)

Test parameters:
50 g oil
5 ml $H_2O$
3 m Cu-wire
$O_2$ – 620 kPa
Temp. 150°C

The period of time is measured in which the oxygen in the autovclave has been used up.

**Fig. 10.25** Lifetime of industrial gear oils based on oxidation stability [10.32].

Table 10.15 gives examples of industrial gear oils blended from different types of base oil. The expected lifetimes of gear oils, with regard to oxidation stability in particular, can be compared by use of rotary bomb oxidation tests [10.33]. This test measures the time in which the oxygen in gear oil is used-up in a pressurized autoclave. Synthetic long-life oil (long-drain in accordance with lifetime factor) has good oxidation stability. Comparative tests have shown that some synthetic industrial gear oils have much greater oxidation stability than mineral oil-based products (Fig. 10.25). The performance of synthetic gear oils in application-related mechanical–dynamic tests with regard to micro-pitting, frictional torque, and persistent oil sump temperatures in gears and roller bearings are summarized in Figs 10.26 to 10.28.

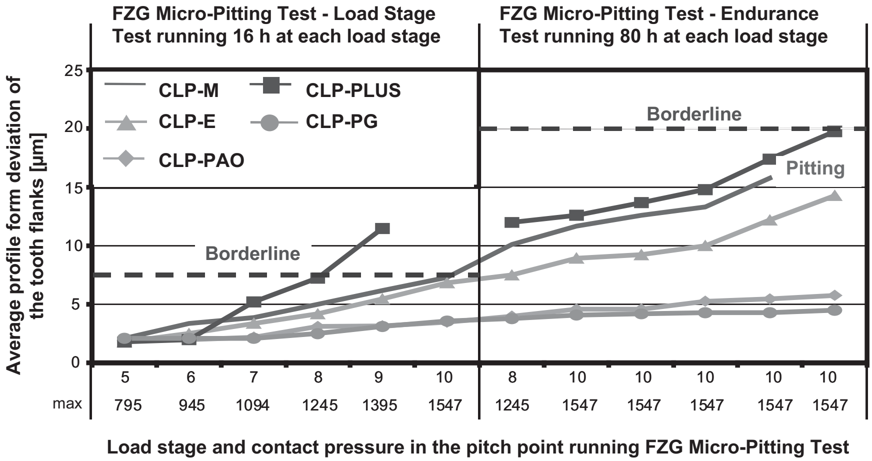


**Fig. 10.26** Industrial gear oils, a comparison of micro-pitting test results from the FZG Gear Test Rig.

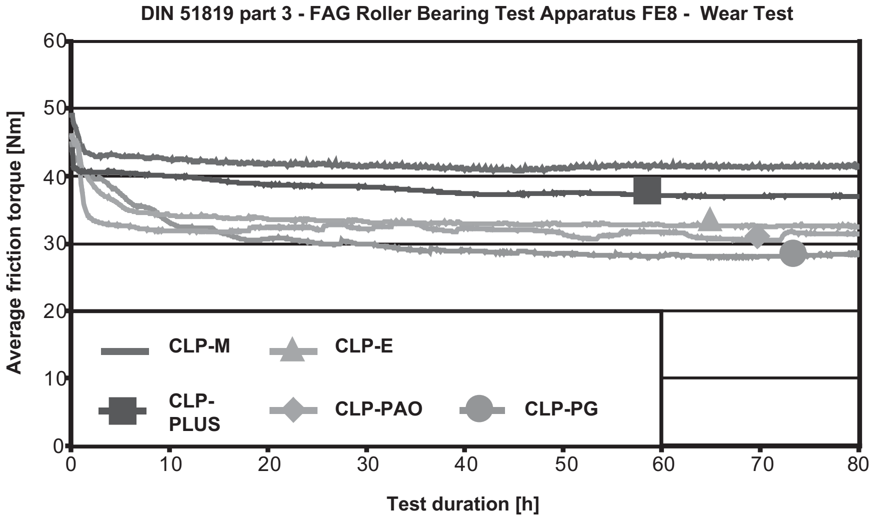


**Fig. 10.27** Industrial gear oils, a comparison of measured friction torque in the FAG Roller Bearing Test Apparatur FE8.

The micro-pitting test (Sect. 19.3.1.4) evaluates the quality of protection offered by synthetic lubricants against micro-pitting of tooth flank surfaces (Fig. 10.26). This test simulates practical conditions at moderate sliding speeds in the meshing zone, transferable to all types of gear (compare Fig. 10.5). Synthetic gear oils have wear-protection properties in gearboxes and roller bearings. Gear oil friction torque mea-

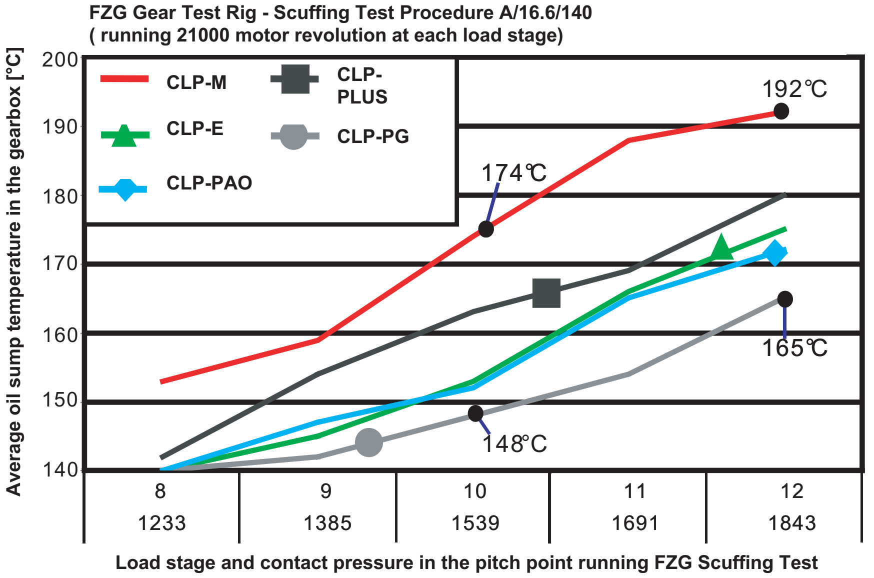


**Fig. 10.28** Industrial gear oils, a comparison of scuffing load stage and oil sump temperature on the FZG Gear Test Rig.

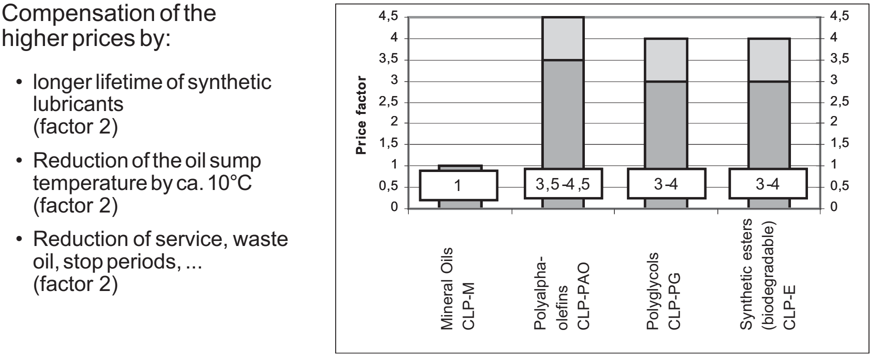


**Fig. 10.29** Cost-benefit ration of gear lubrication oils.

sured in the FAG roller bearing test apparatus FE8 (Sect. 19.4.1) is compared in Fig. 10.27. As a result of their base fluids, synthetic oils with optimized additives have enormous potential to avoid scuffing and to reduce friction, and thus reduce oil sump temperatures by up to 30 °C. This was measured in the FZG gear test rig by use of enhanced A/16.6/140 scuffing tests (Sect. 19.3.1.1); the test results are given in Fig. 10.28.

The test results of the lubricants including those from the „newer" tests provide vital information about the oil-related friction reduction in gearboxes and roller bearings and emphasize their overall performance. This level of performance from synthetic fluids results in increased power and improved productivity. Compared with standard mineral oils of types CLP-M and CLP-PLUS, the prices of synthetic base fluids of types CLP-PAO, CLP-E, and CLP-PG are approximately 3 to 5 times higher. The longer lifetime of the synthetic base oil compensates for the higher product price by a factor of two and reduces sump temperatures by 10 to 30 °C, a factor of 2 to 8 (further reducing drain intervals). All these aspects must be considered when the overall cost-benefit of a product is evaluated. The higher cost of synthetic gear oils must therefore be set against the savings generated by longer life (compared with mineral oils) and reduction in service cost. If all these aspects are considered, use of synthetic base fluids can substantially reduce total lubricant-related system costs; examples are listed in Fig. 10.29. In real-life applications it is worth performing cost–benefit analysis while considering the excellent tribological characteristics of synthetic gear oils. The higher initial price for synthetic products can be compensated by savings generated by significantly longer drain intervals compared with mineral oils and overall reduction of total operating costs.

# 11
# Hydraulic Oils

*Wolfgang Bock*

## 11.1
## Introduction

Hydraulics describe the transfer of energy and signals through fluids; power is transferred to drive, control and move. Hydraulic fluids based on mineral oils, synthetic fluids and fire-resistant fluids are used in all types of machinery and equipment. Hydraulics are a part of everyday life. There is hardly a machine or aircraft which operates without hydraulics. Hydraulic-component manufacturers supply nearly all industries including the agriculture and construction machinery sectors, conveyor technology, the foodstuffs and packaging industries, woodworking and machine tools, ship-building, mining and steel industries, aviation and aerospace, medicine, environmental technology, and chemicals. Many of these industries are leading players in the global market. Fluid technology makes a significant contribution to the competitiveness of these industries. The innovative development of hydraulic components and systems using the very latest materials, lubricants, and electronics gives technical developments new impulses [11.1]:

- fluid technology, is an essential technology: many applications are economical only if fluid technology is used;
- fluid technology is omnipresent – whether in stationary or mobile applications – throughout the world;
- fluid technology benefits the environment: as an environmentally harmless technology and used in environmentally-sensitive plants, fluid technology contributes to our quality of life;
- fluid technology promotes an orderly future – wherever something happens, forces and torque are needed, as are hydraulics; and
- fluid technology in general use is oriented to the needs of a variety of end-users.

The field of fluid technology and therefore hydraulics is divided into hydrostatics and hydrodynamics. In hydrostatic systems, the transfer of energy requires static pressure, and so pressures are high but flow rates are low [11.2]. In hydrodynamic systems, the kinetic energy of the flowing fluid is used, so pressures are low but flow rates are high. Fluids designed for hydrodynamic applications are known as power-transmission oils and fluids for hydrostatic applications are known as hydrau-

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lic oils [11.2–11.4]. The fluid is the most important element in hydrostatic and hydrodynamic systems and must be treated like a machine element in the planning, realization, and commissioning of hydraulic systems.

After engine oils, hydraulic oils are the second most important group of lubricants. They account for approximately 13–14 % of total lubricant consumption [11.2, 11.5, 11.6]. In 2005, mineral-based hydraulic oils accounted for approximately 80–85 % of all hydraulic oils in Germany. Fire-resistant fluids had a market share of ca. 7 %, rapidly biodegradable hydraulic fluids ca. 7 %, and synthetic PAO- or HC-based fluids ca. 1 % [11.2, 11.5, 11.6].

Since the early 1950s, hydraulics have been growing rapidly. The German VDMA (Verein Deutscher Maschinen- und Anlagenbau e.V., German Machinery and Plant Manufacturers' Association) founded an 'Oil Hydraulics and Pneumatics' consultative group in 1959. According to the VDMA [11.1] in 2003, sales of hydraulic equipment and machinery in Germany totaled approximately 4.1 billion Euro with exports making up ~ 50 %. Fluid technology is thus a rapidly expanding industry and its growth is significantly larger than that of engineering.

Modern hydraulics can be divided into three principal areas: stationary, mobile, and aviation hydraulics. Each of these areas makes special demands on its components and the hydraulic medium. In recent years, the performance of hydraulic systems has increased significantly. This is reflected in higher pressures, higher system temperatures, and lower system volumes, which increase circulation and thus the stress on the medium.

Hydraulic fluid developments both to date and in the future, and their correct application, are of enormous economic significance. Optimum applications save energy, reduce maintenance intervals, reduce wear, increase machine life, and thus enable worthwhile savings.

## 11.2
## Hydraulic Principle–Pascal's Law

The principle of the hydrostatic displacement machine is based on Pascal's Law from the 17th century which states, 'Pressure applied anywhere to a body of fluid causes a force to be transmitted equally in all directions. This force acts at right angles to any surface within, or in contact with, the fluid.' The static pressure in a fluid thus enables force to be transferred. Figure 11.1 illustrates Pascal's hydrostatic principle. Figure 11.2 shows the principle of a hydraulic press [11.6].

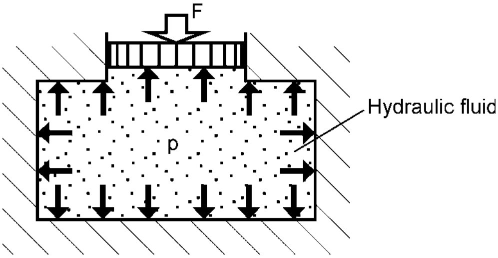


**Fig. 11.1** Pascal's hydrostatic principle.

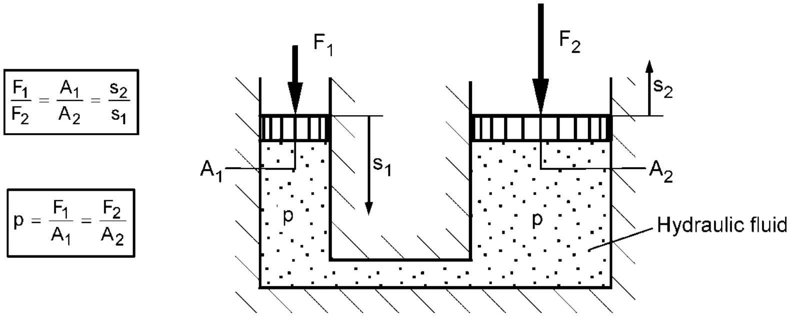


**Fig. 11.2** Principle of a hydraulic press.

## 11.3
## Hydraulic Systems, Circuits, Components

The hydraulic transfer of power is characterized by the simplicity of its elements, long life, high performance, and economy. The variety of hydraulic applications is largely determined by the behavior of the hydraulic fluid.

### 11.3.1
### Elements of a Hydraulic System

The most important elements of a hydraulic system are:

- pumps and motors (e.g. gear, rotary vane and piston pumps)
- hydraulic cylinders (e.g. single- and double-action)
- valves (e.g. pressure limiters and control valves)
- circuit components (e.g. fluid tanks, filter systems, pressure tanks, pipework etc.)
- seals, gaskets and elastomers

Figure 11.3 shows a schematic illustration of a simple hydraulic circuit [11.6].

#### 11.3.1.1 Pumps and Motors

Pumps and motors are used in hydraulic systems to transfer energy. Electricity or mechanical energy is transformed into hydraulic energy by hydrostatic machines such as displacement pumps. The most important types of pump are gear, rotary vane, and axial and radial piston pumps. Gear pumps are used for flow rates of 0.4 to 1200 $cm^3$ $revolution^{-1}$ and pressures of 160 to 250 bar (internal gear pumps up to 350 bar) [11.2–11.4, 11.6].

Rotary vane pumps can generally create pressures up to 160 bar but new developments enable use of pressures between 210 and 290 bar. Flow rates are ca. 30 to 800 $cm^3$ $revolution^{-1}$ [11.2–11.4, 11.6]. Radial piston pumps cover the range up to 700 bar but this type of pump is used for pressures of ca. 480 bar [11.2–11.4, 11.6]. Axial piston pumps are divided into swashplate and bent-axis pumps. These can create pressures of up to 450 bar and higher. Flow rates vary greatly depending on pump dimensions [11.2–11.4, 11.6].

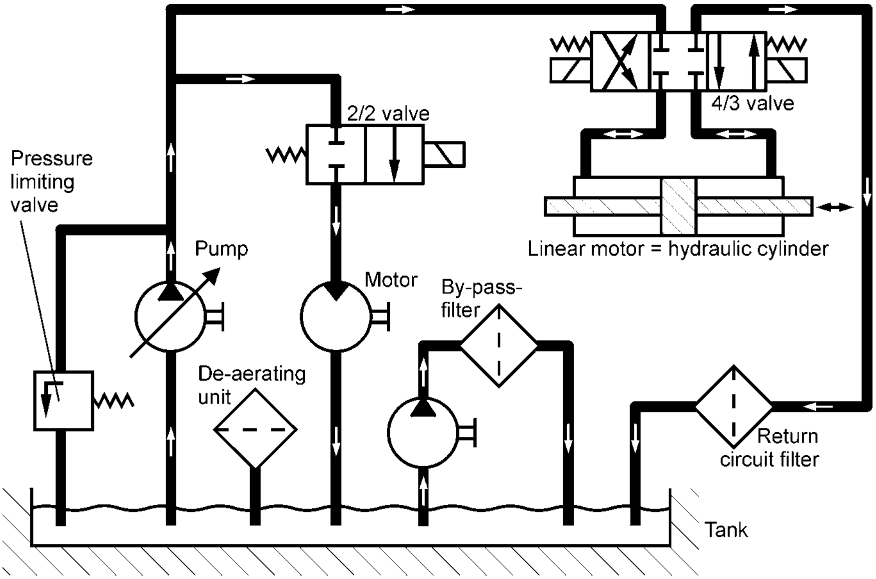


**Fig. 11.3** Schematic illustration of a simple hydraulic circuit.

Pumps and motors are subject to great hydraulic stress. The main functions of hydraulic fluids are to protect (drive components and bearings) from wear and corrosion and reduce friction, thus reducing the accumulation of deposits.

Figures 11.4–11.7 illustrate the most important types of hydraulic pumps and motors: Gear pumps, rotary vane pumps, radial piston pumps and axial piston pumps.

### 11.3.1.2 **Hydraulic Cylinders**

Hydraulic cylinders transform hydraulic pressure into linear movement. This can then perform mechanical work. Hydraulic cylinders can be single- or double-action. In these the primary functions of the fluid are to seal and lubricate the piston and guides, avoid stick–slip, minimize wear, and avoid corrosion [11.2, 11.4, 11.6].

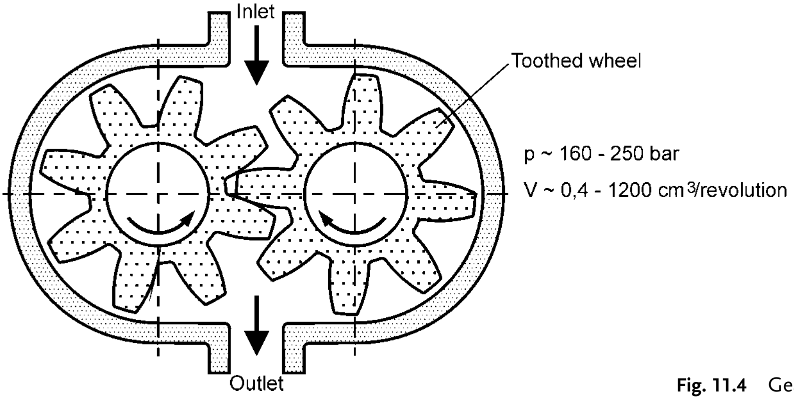


**Fig. 11.4** Gear pump.

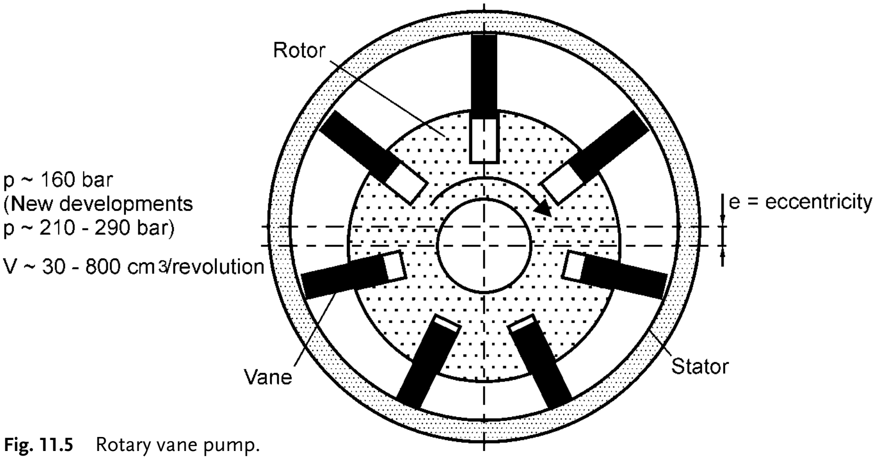


**Fig. 11.5** Rotary vane pump.

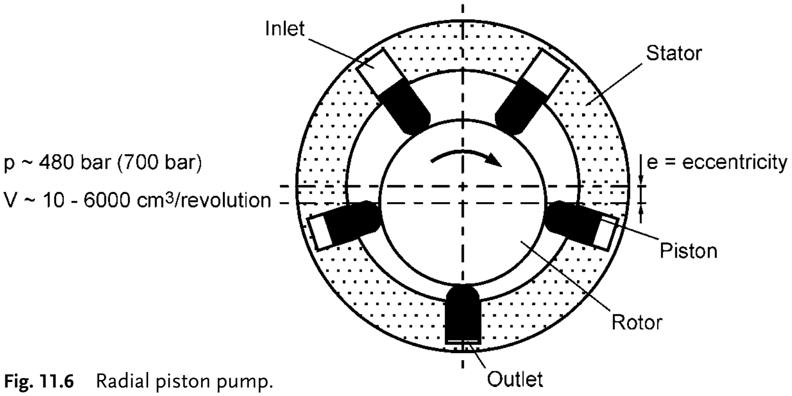


**Fig. 11.6** Radial piston pump.

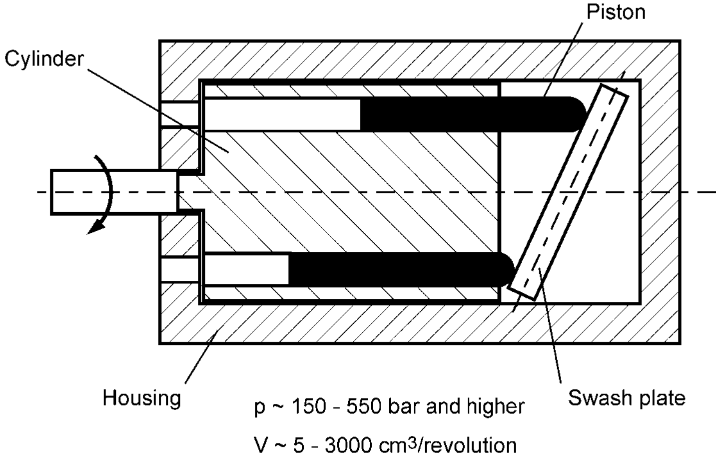


**Fig. 11.7** Axial piston pump.

#### 11.3.1.3 **Valves**

Valves are mechanisms which control the start, stop, direction, or flow of a hydraulic medium from a pump or from a pressure vessel. Flow valves have preset switching points. Proportional and servo valves are electro-hydraulic, i.e. their movement is proportional to the electrical input signal. The differences between these valves are their mechanical design, their static and dynamic properties, and their price. The hydraulic fluid in a valve should dissipate heat, reduce wear, minimize friction, and avoid corrosion. Equally important, no deposits must form in the narrow tolerances found in valves. Long fluid-change intervals and high thermal loads (e.g. caused by solenoid magnets) must not lead to deposits in, or the gumming of, flow valves [11.2, 11.4, 11.6].

#### 11.3.1.4 **Circuit Components**

These include fluid tanks, filter systems, pressure tanks, and pipework. The hydraulic fluid must be compatible with all of the materials used in these elements including all coatings and paint finishes [11.6].

#### 11.3.1.5 **Seals, Gaskets and Elastomers**

Every seal or elastomer in a hydraulic system is fully or partially exposed to the hydraulic fluid when the system is in operation. Interaction between the seal material and the hydraulic medium is thus unavoidable.

The hydraulic medium can influence the sealing material insofar as it causes shrinkage or swelling. This, in turn, effects the volume of the seal and also alters mechanical properties such as hardness, elasticity, tensile strength, and elongation behavior.

Elastomeric seals are influenced chemically by temperature, oxygen, water, additives, and the oxidation by-products of the hydraulic fluid. It is therefore vital that the seals and the hydraulic fluid are chemically compatible.

A seal is mechanically stressed by the pressure and pulsation of the fluid. In addition, dynamically stressed seals such as piston and rod seals are subject to sliding friction [11.6–11.8].

Physical and chemical factors have a direct influence on the mechanical wear of a seal. Swelling causes the seal material to soften. This can lead to higher friction and thus greater wear and power consumption. Normally it is accepted that seals can swell within defined limits to avoid leakage.

Ideally, a hydraulic fluid should have no effect on seal materials and elastomers while at the same time as protecting them from wear, dissipating heat, reducing friction, and avoiding the accumulation of deposits in crevices. Seal manufacturers usually test the compatibility of their products with different hydraulic fluids and publish these in compatibility lists. Lubricant manufacturers are primarily concerned with the behavior of seals in contact with hydraulic fluids as detailed in DIN 51 524 (dated 2006) and the effect of mineral oil-based fluids on SRE NBR 1 seal material as detailed in DIN 53 521 and DIN 53 502. These standards establish thresholds for volume and hardness changes to seal materials. Fundamental and comparison tests are performed with reference fluids. These reference fluids are specified and classified according to an ASTM standard. ASTM Fluid 2 and Fluid 3 and their successor products, IRM 2 and IRM 3 are used for testing purposes [11.7–

11.9]. Basically, a seal must not shrink when in contact with a hydraulic fluid because of the danger of leakage, although slight swelling is permissible. The seal must not harden, but slight softening is allowed. The duration of compatibility tests on seals with hydraulic media is seven days at 100 °C [11.10].

Rapidly biodegradable hydraulic fluids based on vegetable oils, esters, and polyglycols are tested for longer periods for more practically relevant results. The behavior of rapidly biodegradable hydraulic fluids in contact with standard reference elastomers (SRE) is tested for 1000 h at temperatures ranging from 80 °C to 100 °C, as laid down in CETOP R81H, ISO 6072. The seal materials listed in this test include HNBR, FPM AC 6, NBR 1 and AU grades. The properties tested now include hardness changes, volume changes, tensile strength and elongation breakage-point. According to this test, the tensile strength and elongation breakage-point must not exceed 30 % in the presence of rapidly biodegradable hydraulic fluids. [11.9, 11.11–11.13]. Table 11.1 shows the general compatibility of elastomers with different hydraulic fluids [11.7, 11.8, 11.13].

**Tab. 11.1** Compatibility of elastomers with hydraulic fluids.

| | ***NBR*** | ***HNBR*** | ***AU*** | ***FPM*** | ***EPDM mineral-oil-free*** |
|---|---|---|---|---|---|
| | ***Average temperature range of the elastomers in °C (permanent operating range)*** | | | | |
| | ***−30/(−40)/+100*** | ***−20(−30)/+140*** | ***−30/+80 (+100)*** | ***−20/+200***** | ***−50/+150***** |
| HL/HLP/HLPD mineral oils | + | + | + | + | − |
| HFD | − | − | − | +* | +* |
| HFC | + | + | +* | − | + |
| HFB | + | + | − | + | − |
| HFA | + | + | +* | + | − |
| HETG | + | + | + | + | − |
| HEES | +* | +* | +* | + | − |
| HEPG | +* | + | − | + | + |

* Check the application in case of dynamic stress of elastomer material  ** Max. Tmp. in air.

| ***Symbol according to DIN/ISO 1629 resp. ASTM D 1418*** | ***Chemical name*** | ***Trade name*** |
|---|---|---|
| NBR | Acrylonitrile–butadiene–caoutchouk | Perbunan, Nipol, Europrene |
| HNBR | Hydro-treated acrylonitrile–butadiene–caoutchouk | Zetpol, Theiban |
| AU | Polyurethane–caoutchouk (Polyesterurethane–caoutchouk) | Desmopan/Urepan |
| FPM | Fluoro–caoutchouk (FKM) | Viton, Fluorel, Tecnoflon |
| EPDM | Ethylene–propylenediene–caoutchouk | Vistalon, Buna EPG, Keltan |
| SBR | Styrene–butadiene–caoutchouk | Buna SB |
| CR | Chlorobutadiene–caoutchouk | Neoprene/Chloroprene |
| PTFE | Polytetrafluoroethylene | Hostaflon/Teflon |

In principle, the saying 'like dissolves like' applies. To illustrate this, non-polar elastomers such as EPDM rubber swell to the point of dissolving in non-polar hydrocarbons contained in mineral oil-based hydraulic fluids. Conversely, polar fluids such as HEPG fluids (polyglycols) work perfectly with these non-polar elastomer rubbers [11.7, 11.8, 11.13].

Elastomer tests with fire-resistant hydraulic fluids are described in the 7th Luxembourg Report. As food-grade lubricants are based on white oils and polyalphaolefins, they are used in the DIN 51 524 and ISO 6743/4 elastomer test procedures [11.10, 11.14].

General seal compatibility statements must consider the different manufacturing processes and the different composition of elastomers in practice. For example, NBR materials can contain different amounts of acrylic nitrile and this has to be kept in mind when testing and evaluating elastomers and lubricants. The new ISO working paper 'Hydraulic fluid power – compatibility between fluids and standard elastomeric materials' (ISO TC 131/SC7N343 Draft), dated 1999, lists methods which describe the influence of fluids on standard elastomeric materials [11.9].

## 11.4
## Hydraulic Fluids

### 11.4.1
### Composition of Hydraulic Fluids (Base fluids, additives)

#### 11.4.1.1 Base Oil, Base Fluid

In general, a hydraulic fluid consists of a base fluid, usually called a base oil, and chemical substances, usually called additives. The quality and performance of a hydraulic fluid depends on the quality of the base fluid and the combination of the additives or additive systems used. Additives improve certain characteristics which the base fluid cannot provide, or cannot provide not to a sufficient extent. Bearing in mind technical and ecological aspects, the type of base fluid and the types of additive ultimately decide the classification of a hydraulic oil [11.5].

Mineral oil-based fluids (paraffinic oils, naphthenic oils and white oils) and/or mixtures thereof are used as base fluids or base oils. Synthetic fluids based on hydrocracked oils (HC oils or so-called group III oils), polyalphaolefins (PAO), ester oils (POE) and polyglycols (PAG) are mainly used in fire-resistant, rapidly biodegradable fluids or special hydraulic fluids. Natural vegetable oils such as rapeseed oil are often found in rapidly biodegradable fluids. Food-grade hydraulic oils are generally based on special white oils, polyalphaolefins and polyglycols (see Chapters 4 and 5, Base Oils and Synthetic Base Oils) [11.2, 11.5, 11.6].

Mineral oils account for ~ 88 % (mainly paraffinic Group I Oils); synthetic oils account for 12 % ( 80 % Ester, 15 % Polyglycols, etc.).

#### 11.4.1.2 Hydraulic Fluid Additives

The required lubricity of hydraulic fluids is normally provided by special additives. The additives are usually included in the form of additive packages (mixtures) to achieve the desired results. The additive components can either complement each other or counter each other. The characteristics which can be improved by the use of additives include aging stability, corrosion protection, wear protection, EP behavior, viscosity–temperature behavior, foaming, detergency, water separation, friction coefficient and many more.

The most important additives for hydraulic fluids are:

- 'surface-active additives' such as rust inhibitors, metal deactivators, wear inhibitors, friction modifiers, detergents/dispersants, etc.; and
- 'base oil active additives' such as antioxidants, de-foamers, *VI* improvers, pour-point improvers, etc.;

A rough classification of hydraulic fluid additive systems can be made by differentiating between additive systems containing zinc and ash and zinc- and ash-free (ZAF) systems (see Chapter 6: Additives) [11.5, 11.15]. On average, zinc-containing hydraulic oils account for 70–80 % of total volume, zinc and ash-free fluids for 20–30 % of total volume.

### 11.4.2
### Primary, Secondary and Tertiary Characteristics of a Hydraulic Fluid

The primary functions and properties of a hydraulic fluid are [11.2, 11.5, 11.6]:

- transferring pressure and motion energy;
- transferring forces and moments when used as a lubricant;
- minimization of wear to sliding surfaces under boundary friction conditions;
- minimization of friction;
- protection of components against corrosion (ferrous and non-ferrous metals);
- dissipation of heat;
- suitability for a wide range of temperatures, good viscosity–temperature behavior; and
- prolonging the life of machinery, etc.

A hydraulic fluid must satisfy the following

*Secondary characteristics*: high aging stability, good thermal stability, inactive to materials, compatibility with metals and elastomers, good air separation, low foaming, good filterability, good water release, good shear stability in the case of non-Newtonian fluids and many more. [11.2, 11.5, 11.6].

*Tertiary characteristics*: low evaporation as a result of low vapor pressure, toxicologically harmless, ecologically safe, low flammability (fire-resistance), etc. [11.2, 11.5, 11.6].

The wide variety of different characteristics required of hydraulic fluids necessitates special performance which cannot be satisfied by just one base oil. Special chemical substances (additives) improve and complement the technical performance of hydraulic fluids. Synthetic base fluids can satisfy specific performance requirements such as environmental compatibility, high thermal stability, fire-resistance and use in food-grade applications [11.5].

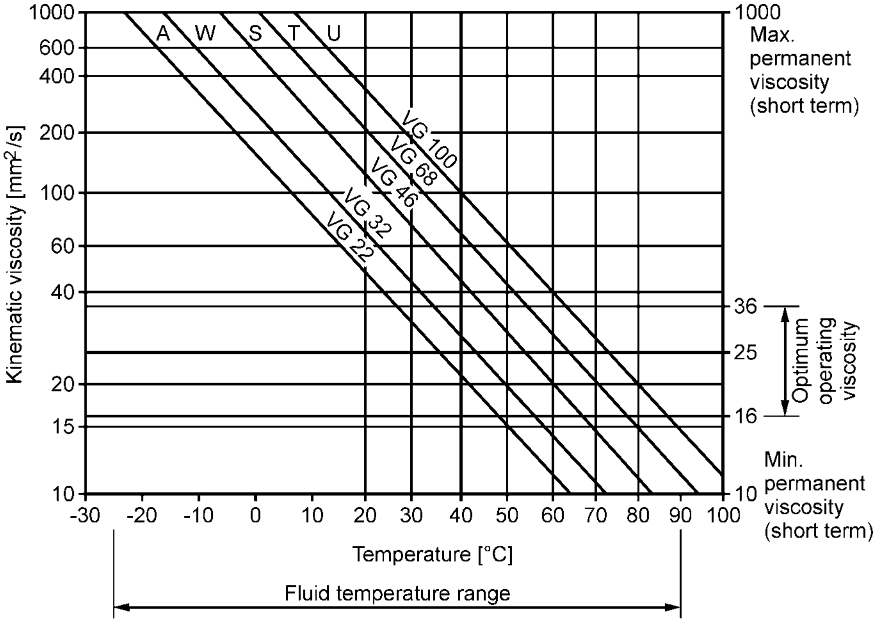
### 11.4.3
### Selection Criteria for Hydraulic Fluids

Fluid selection depends on the application, such as the working temperature range, design of the hydraulic system, type of pump, working pressure, and environmental considerations. The required fluid life, availability, and economic and ecological factors also determine the type of hydraulic oil used. From a rheological standpoint, the viscosity of the fluid selected should be as low as possible. This guarantees instant hydraulic response when the system is activated. On the other hand, a minimum viscosity is required to reduce leakages and to guarantee adequate lubrication of the pump and other moving parts [11.2–11.6]. Any change in hydraulic fluid temperature has a direct effect on viscosity. For this reason, the working-temperature range of a hydraulic system should be kept relatively narrow to keep viscosity fluctuations as small as possible. For accurate fluid selection, it is assumed that the working and ambient temperatures are known. In sealed systems this is the circuit temperature and in open systems, the tank temperature. The viscosity of the fluid selected should be in the optimum range, between 16 and 36 $\text{mm}^2\ \text{s}^{-1}$ ($V_{\text{optimum}}$ = optimum operating viscosity = 16–36 $\text{mm}^2\ \text{s}^{-1}$). Under threshold conditions (during cold starts, short-term overloading), the viscosities shown in Table 11.2 could apply, depending on the type of pump used [11.2, 11.4, 11.16–11.21]:

**Tab. 11.2** Viscosities by the use of different type of pumps

| *Type of pump* | *Max. permissible viscosity in $\text{mm}^2\ \text{s}^{-1}$ (during cold starts)* | *Min. permissible viscosity (during load conditions, max. permissible oil temperature of 90 °C) in $\text{mm}^2\ \text{s}^{-1}$* |
|---|---|---|
| Gear pumps | ca 1.000 | 10–25 |
| Piston pumps | 1.000–2.000 | 10–16 |
| Rotary vane pumps | 200–800 | 16–25 |

Normal operating temperature depends on ambient temperature, pressure and other factors. In low- and medium-pressure stationary hydraulic systems, the operating temperature should be ca. 40–50 °C (tank temperature). For systems which operate at higher pressures (over 400 bar), the average system temperature can be ca. 10–20 °C higher [11.2, 11.6, 11.22]. It must be remembered that the fluid temperature at the pump outlet and downstream of motors or valves is higher than in the tank or the system's average temperature. The leakage temperature, which is influenced by pressure and pump speed, is always above the system or tank temperature. The fluid temperature should never exceed 90 °C (max. 100 °C) in any part of the system [11.22, 11.23]. If these conditions cannot be met, owing to extreme circumstances, at lower ambient temperatures it is recommended that pumps and motors are flushed [11.4, 11.6]. Start-up and working viscosity (operating viscosity of



A = for arctic conditions or for extremely long pipelines
W= for winter conditions in Central Europe
S = for summer conditions in Central Europe or for enclosed areas
T = for tropical conditions or for areas with high temperatures
U = for excessively high temperatures (e.g. due to internal combustion engines)

Depending on the construction of the pumps (axial piston units) and the operating conditions the following viscosity ranges are valid:

10 mm2/s ($t_{max}$ = +90°C) ..... 1000 mm2/s ($t_{min}$ = -25°C)

[5 mm2/s ($t_{max}$ = +115°C) ..... 1600 mm2/s ($t_{min}$ = -40°C)] special components

Figure: Brueninghaus Hydromatik

**Fig. 11.8** Selection diagram for hydraulic fluids.

hydraulic oils) are set by the various ISO Viscosity Grades. Most applications are satisfied by the viscosity grades 15, 22 (at low ambient temperatures), 32, 46 and 68 [11.17]. Normally oils with a Viscosity Index (*VI*) of about 100 are used. Hydraulic oils with higher VIs (better viscosity–temperature range) are recommended for special hydraulic systems, at higher temperatures, at lower ambient temperatures and in mobile applications [11.2, 11.5, 11.6]. If viscosity index improvers (*VI* improvers) are used, they must be shear-stable (over the lifetime of the fluid) to ensure that the oil retains its mechanical properties throughout its life. Higher-viscosity oils can be used in older systems to reduce leakages and wear. High viscosity-index hydraulic oils can enable grade rationalization in industrial applications. For example, an HVLP 46 can replace up to five viscosity grades (ISO VG 15–68) in industrial applications. The selection diagram (Fig. 11.8) shows recommended viscosity grades in relation to ambient temperature [11.17].

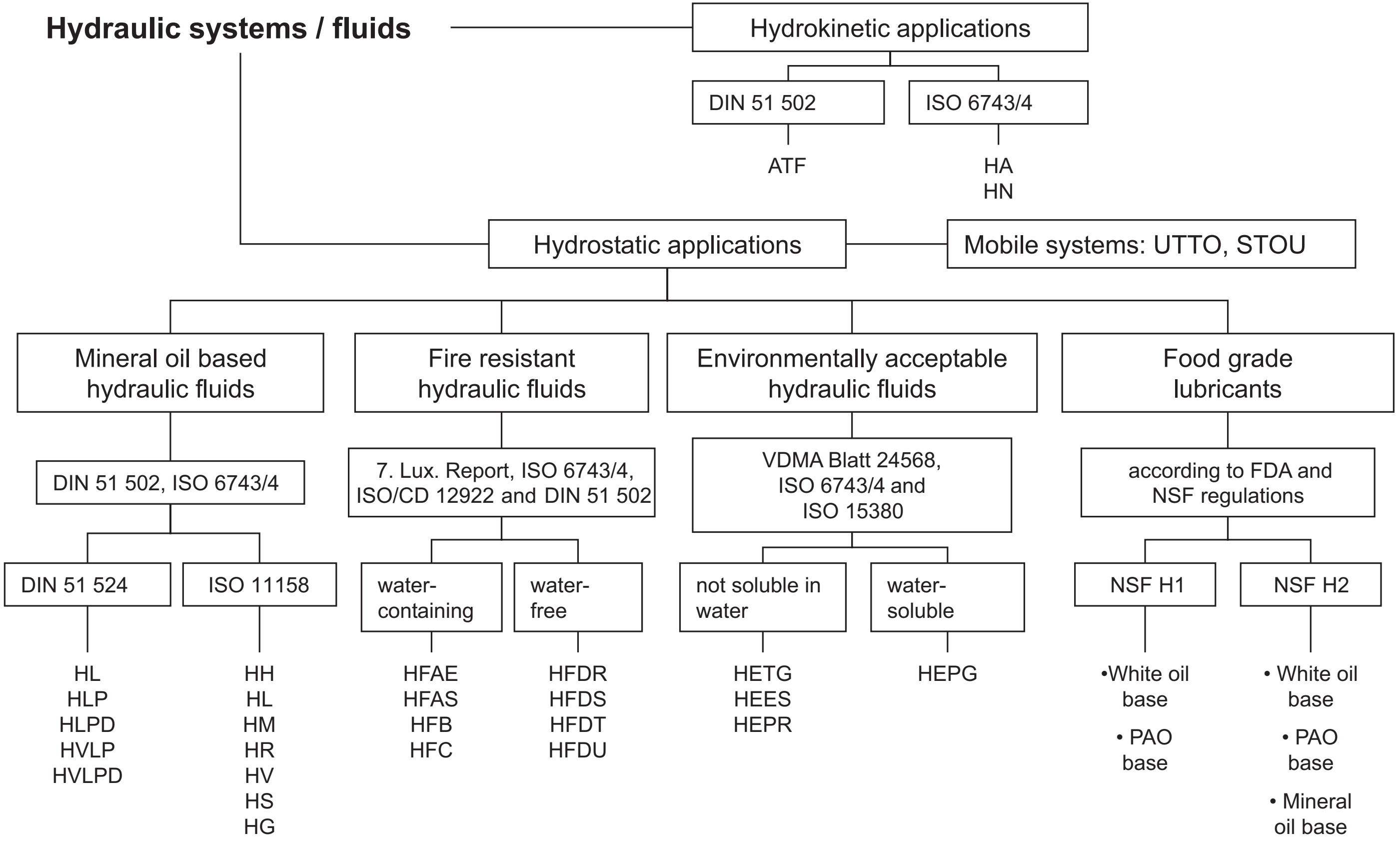


**Fig. 11.9** Classification of hydraulic fluids – overview

The average temperature of mineral-based hydraulic oils in stationary systems should not exceed 50–60 °C and 80–90 °C in mobile systems. Fluids containing water (e. g. HFC fluids) should be kept down to 35–50 °C (vapor pressure of water) [11.6].

The volume of fluid in stationary systems should be three- to five-times the volume pumped per minute. In mobile systems, the tank volume should be one- to two-times the pumped volume but in special circumstances, it can be less [11.2, 11.4, 11.6].

## 11.4.4
## Classification of Hydraulic Fluids – Standardization of Hydraulic Fluids

### 11.4.4.1 Classification of Hydraulic Fluids

Depending on their ultimate use, hydraulic oils can be allocated to one of two main groups – fluids for hydrostatic applications and fluids for hydrodynamic applications.

Hydrostatic applications can be divided into sub-groups with regard to ISO, CETOP and national (e.g. DIN) classifications [11.24]:

- DIN 51 524 or ISO 6743/4 hydraulic oils [11.10, 11.24].
- ISO/CD 12922, VDMA 24317, CETOP RP97H and DIN 51 502 fire-resistant hydraulic fluids according to the 7th Luxembourg Report or Factory Mutual USA Factory Mutual System-Insurance Company USA (FM Global) [11.14, 11.24–11.28].
- ISO 15 380 (VDMA 24568) and ISO 6743/4 rapidly biodegradable hydraulic fluids [11.11, 11.12, 11.25] ISO 15 380: Lubricants, industrial oils, and related products (class L) Family H – Specification for environmentally acceptable fluids – HETG, HEES, HEPG, HEPR.
- NSF H1, H2 and FDA food-grade hydraulic oils [11.29] NSF Internatrional – The Public Health and Safety Company (a not-for-profit, nongovernmental organization – USA) .
- STOU and UTTO universal mobile hydraulic oils [11.30].

The hydraulic oils used in hydrodynamic applications can be allocated to ATF, coupling and converter fluids (see Chapter 10).

Figure 11.9 shows the different categories of hydraulic fluids and their principal uses.

## 11.4.5
## Mineral Oil-Based Hydraulic Fluids

According to DIN 51 524 and ISO 6743/4, these fluids can be categorized as in Table 11.3 [11.10, 11.25]:

**Tab. 11.3** Classification of mineral oil-based hydraulic fluids (Categories according to DIN 51 502 and ISO 6743/4).

| ***Category (Symbol)*** | | ***Composition*** <br> ***Typical characteristics*** | ***Field of application*** <br> ***Operating temperatures*** |
|---|---|---|---|
| ***DIN*** | ***ISO-L*** | | |
| – | HH | Non-inhibited refined mineral oils | Hydraulic systems without specific requirements (rarely used nowadays)/ –10 to 90 °C |
| HL | HL | Refined mineral oils with improved anti-rust and anti-oxidation properties | Hydrostatic drive systems with high thermal stress; need for good water separation / –10 to 90 °C |
| HLP | HM | Oils of HL type with improved anti-wear properties | General hydraulic systems which include highly loaded components, need for good water separation/ –20 to 90 °C |
| – | HR | Oils of HL type with additives to improve viscosity–temperature behavior | Enlarged range of operating temperatures compared with HL oils / –35 to 120 °C |
| HVLP | HV | Oils of HM type with additives to improve viscosity–temperature behavior | For example, hydrostatic power units in construction and marine equipment –35 to 120 °C |
| – | HS | Synthetic fluids with no specific inflammability characteristics and no specific fire resistance properties | Special applications in hydrostatic systems, special properties / –35 to 120 °C |
| – | HG | Oils of HM type with additives to improve stick–slip-behavior, and anti-stick–slip properties | Machines with combined hydraulic and plain bearing way lubrication systems where vibration or intermittent sliding (stick/slip) at low speed must be minimized / –30 to 120 °C |
| HLPD | – | Oils of HM type with detergent / dispersant (DD) additives<br>DD additives reduce friction | Hydrostatic drive units with high thermal stress, which require EP/ AW additives, DD additives keep contaminants in suspension, e.g. machine tools and mobile hydraulic equipment |

*Category L*: Lubricants, Industrial oils and related products
*Category H*: Hydrostatic hydraulic systems

#### 11.4.5.1 **H Hydraulic Oils**

Type H, mineral-based hydraulic oils are generally base oils without additives. Accordingly, they are hardly used in Western Europe.

Classification: ISO 6743/4–HH [11.25]

#### 11.4.5.2 **HL Hydraulic Oils**

Hydraulic fluids with additives to improve aging stability and corrosion protection.

These oils are used in hydraulic systems which need not be protected from wear such as in steel and rolling mills where the prevailing conditions cause the fluids to be contaminated with water. Thus these types of fluid can separate water well, usually also release air rapidly, and are compatible with special–white metal and Morgan–bearings (Spec. Section 1: HL/CL, Spec. Section 2: HLP/CLP). If higher viscosities are used as general lubricating oils, they must fulfil the corresponding CL and CKB specifications according to DIN 51 517-2 [11.31] and ISO 6743/6 [11.32] respectively.

HL hydraulic oils according to DIN 51 524-1 [11.10].

HL hydraulic oils according to ISO 6743/4 [11.25].

Table 11.4 shows the specification profile of DIN 51 524-1 hydraulic oils (2006).

#### 11.4.5.3 **HLP Hydraulic Oils**

Compared with HL fluids, these oils contain additional agents to reduce wear and/or improve EP behavior. This is the dominant group of hydraulic oils in Europe and the rest of the world. They are universal hydraulic oils for a wide range of applications and highly loaded components and are used for applications which require good aging stability, corrosion protection, and wear protection. At the same time, these lubricants offer good demulsifying properties. These fluids are used as universal products in hydraulic presses, die-casting machines and in steel mills when a fluid with wear protection is specified.

HLP according to DIN 51 524-2 [11.10].

HM according to ISO 6743/4 [11.25].

Table 11.5 illustrates the specifications of DIN 51 524-2 hydraulic oils (April 2006). Table 11.6 shows the specifications for HVI-hydraulic oils according to DIN 51 524-3.

#### 11.4.5.4 **HVLP Hydraulic Oils**

Compared with HLP grades, these fluids have a high viscosity index ($VI > 140$, HLP oils: $VI \approx 100$). They therefore have good viscosity–temperature behavior. The high $VI$ is achieved by use of additives and/or by use of an appropriate base oil. The natural high $VI$ of the base oil is preferred because shear losses do not happen. According to DIN 51 524-3 in conjungtion with DIN 51 350-6 (VRA – Determination of shear stability of Lubricating oils containing polymers) the shear loss has to be reported (recommended max 15–20 %, OEMS < 15 %). A low start-up viscosity at low temperatures and a high operating viscosity at higher temperatures is a significant technical advantage over equi-viscous HLP oils. The absorption of energy is low, preheating the system is usually not necessary, and operating temperature is achieved rapidly. The use of HVLP oils affords significant rationalization potential. Generally speaking, HVLP oils have a multigrade character [11.2, 11.4, 11.5]. The use of these oils has grown rapidly over the last few years. They are perfect for fluctuating operating temperatures such as those found in mobile hydraulic systems, canal locks and cable-car hydraulics.

HVLP according to DIN 51 524-3 [11.10]

**Tab. 11.4** Minimum requirements of hydraulic fluids – DIN 51 524, Part 1, HL (Revised Version 2006 – April).

***DIN 51 524 Part 1 – Hydraulic fluids with improved anti-rust and anti-oxidant properties***

| Grade (DIN 51 502) | | HL 10 | HL 15 | HL 22 | HL 32 | HL 46 | HL 68 | HL 100 | HL 150 |
|---|---|---|---|---|---|---|---|---|---|
| ISO viscosity grade (DIN 51 519) | | VG 10 | VG 15 | VG 22 | VG 32 | VG 46 | VG 68 | VG 100 | VG 150 |
| Viscosity at 0 °C and ($mm^2 s^{-1}$), max. (DIN 51 562-1) | | 90 (600) | 150 | 300 | 420 | 780 | 1400 | 2560 | 4500 |
| Viscosity at 40 °C, $mm^2 s^{-1}$, min.–max. (DIN 51 562-1) | | 9.0–11.0 | 13.5–16.5 | 19.8–24.2 | 28.8–35.2 | 41.4–50.6 | 61.2–74.8 | 90.0–110 | 135–165 |
| Viscosity at 100 °C, $mm^2 s^{-1}$, min. (DIN 51 562-1) | | 2.5 | 3.2 | 4.1 | 5.0 | 6.1 | 7.8 | 9.9 | 14.0 |
| Pour point, °C, max. (DIN ISO 3016) | | –30 | –27 | –21 | –18 | –15 | –12 | –12 | –12 |
| Flash point (COC), °C, min. (DIN EN ISO 2592) | | 125 | 140 | 165 | 175 | 185 | 195 | 205 | 215 |
| Purity class (ISO 4406:1999) | | 21/19/16 (defined requirements depend on the system) | | | | | | | |
| Contamination with solid particles, mg $kg^{-1}$, max. (DIN ISO 5884 or ISO 4405:1991) | | 50 | | | | | | | |
| Filterability without $H_2O$, Stage I $F_I$/Stage II $F_{II}$, min. % (E DIN ISO 13357-2) | | 80/60 | | | | | | | |
| Filterability with $H_2O$, Stage I $F_I$/Stage II $F_{II}$, min. % (E DIN ISO 13357-1) | | 70/50 | | | | | | | |
| Demulsibility, 54 °C, minutes, max. | (DIN ISO 6614) | 20 | | | 30 | | | – | |
| Demulsibility, 82 °C, minutes, max. | | – | | | – | | | 30 | |
| Water content in % $m/m$, max. (DIN EN ISO 12937 – method A) | | 0.05 | | | | | | | |
| Steel corrosion, method A (DIN ISO 7120) | | Pass | | | | | | | |
| Copper corrosion, 100 °C, 3 h, degree of corrosion, max. (DIN EN ISO 2160) | | 2 | | | | | | | |
| Ageing properties (maximum increase in neutralization number after 1000 h) in mg KOH $g^{-1}$, max. (DIN 51 587 or DIN EN ISO 4263-1) | | ≤ 2.0 | | | | | | | |
| Behavior toward the SRE-NBR 1 sealant after 7 days ± 2 h at (100 ± 1) °C | Relative change in volume, % | 0 to 18 | 0 to 15 | | 0 to 12 | | | 0 to 10 | |
| (DIN 53 538-1, DIN ISO 1817, and DIN 53 505) | Change in Shore A hardness | 0 to –10 | 0 to –8 | | 0 to –7 | | | 0 to –6 | |
| Air release, 50 °C, minutes, max. (DIN ISO 9120) | | 5 | 5 | 5 | 5 | 10 | 13 | 21 | 28 |
| Foam volume in mL, max. (ISO 6247:1998, incl. Corr. 1: 1999)<br>at 24 °C<br>at 93.5 °C<br>at 25 °C after 95 °C | | <br>150/0<br>75/0<br>150/0 | | | | | | | |

**Tab. 11.5** Minimum requirements of hydraulic fluids – DIN 51 524, Part 2, HLP – April 2006, DIN 51524 Part 3 HVLP.

***DIN 51 524 Part 2 – Anti-wear hydraulic oils***

| Grade (DIN 51 502) | | HLP 10 | HLP 15 | HLP 22 | HLP 32 | HLP 46 | HLP 68 | HLP 100 | HLP 150 |
|---|---|---|---|---|---|---|---|---|---|
| ISO viscosity grade (DIN 51 519) | | VG 10 | VG 15 | VG 22 | VG 32 | VG 46 | VG 68 | VG 100 | VG 150 |
| Viscosity at 0 °C/(–20 °C), $mm^2 s^{-1}$, max. (DIN 51 562-1) | | 90 (600) | 150 | 300 | 420 | 780 | 1400 | 2560 | 4500 |
| Viscosity at 40 °C, $mm^2 s^{-1}$, min.–max. (DIN 51 562-1) | | 9.0–11.0 | 13.5–16.5 | 19.8–24.2 | 28.8–35.2 | 41.4–50.6 | 61.2–74.8 | 90.0–110 | 135–165 |
| Viscosity at 100 °C, $mm^2 s^{-1}$, min. (DIN 51 562-1) | | 2.5 | 3.2 | 4.1 | 5.0 | 6.1 | 7.8 | 9.9 | 14.0 |
| Pour point, °C, max. (DIN ISO 3016) | | –30 | –27 | –21 | –18 | –15 | –12 | –12 | –12 |
| Flash point (COC), °C, min. (DIN EN ISO 2592) | | 125 | 140 | 165 | 175 | 185 | 195 | 205 | 215 |
| Purity class (ISO 4406:1999) | | 21/19/16 (defined requirements depend on the system) | | | | | | | |
| Contamination with solid particles, mg $kg^{-1}$, max. (DIN ISO 5884 or ISO 4405:1991) | | 50 | | | | | | | |
| Filterability without $H_2O$, Stage I $F_I$/Stage II $F_{II}$, min. % (E DIN ISO 13357-2) | | 80/60 | | | | | | | |
| Filterability with $H_2O$, Stage I $F_I$/Stage II $F_{II}$, min. % (E DIN ISO 13357-1) | | 70/50 | | | | | | | |
| Demulsibility, 54 °C, minutes, max. | (DIN ISO 6614) | 20 | | | 30 | | | – | |
| Demulsibility, 82 °C, minutes, max. | | – | | | – | | | 30 | |
| Water content in % $m/m$, max. (DIN EN ISO 12937 – method A) | | 0.05 | | | | | | | |
| Steel corrosion, method A (DIN ISO 7120) | | Pass | | | | | | | |
| Copper corrosion, 100 °C, 3 h, degree of corrosion, max. (DIN EN ISO 2160) | | 2 | | | | | | | |
| Ageing properties (maximum increase in neutralization number after 1000 h) in mg KOH $g^{-1}$, max. (DIN 51 587 or DIN EN ISO 4263-1) | | ≤ 2.0 | | | | | | | |
| Behavior toward the SRE-NBR 1 sealant after 7 days ± 2 h at (100 ± 1) °C | Relative change in volume, % | 0 to 18 | 0 to 15 | | 0 to 12 | | 0 to 10 | | |
| (DIN 53 538-1, DIN ISO 1817, and DIN 53 505) | Change in Shore A hardness | 0 to –10 | 0 to –8 | | 0 to –7 | | 0 to –6 | | |
| Air release, 50 °C, minutes , max. (DIN ISO 9120) | | 5 | 5 | 5 | 5 | 10 | 13 | 21 | 32 |
| Foam volume, in mL, max. (ISO 6247:1998, incl. Corr. 1: 1999)<br>at 24 °C<br>at 93.5 °C<br>at 25 °C after 95 °C | | <br>150/0<br>75/0<br>150/0 | | | | | | | |
| FZG mechanical gear test rig, A/8.3/90: failure load stage, min. (DIN 51 354-2 or DIN ISO 14635-1) | | – | – | – | 10 | | | | |
| Vane pump wear, mg (DIN EN ISO 20763) | Ring, max. | – | – | – | 120 | | | – | – |
| | Vanes, max. | – | – | – | 30 | | | – | – |

### DIN 51 524 Part 3 – High-VI hydraulic oils

| Grade (DIN 51 502) | | HVLP 10 | HVLP 15 | HVLP 22 | HVLP 32 | HVLP 46 | HVLP 68 | HVLP 100 | HVLP 150 |
|---|---|---|---|---|---|---|---|---|---|
| ISO viscosity grade (DIN 51 519) | | VG 10 | VG 15 | VG 22 | VG 32 | VG 46 | VG 68 | VG 100 | VG 150 |
| Viscosity at 0 °C/(–20 °C), mm$^2$ s$^{-1}$, max. (DIN 51 562-1) | | To be reported by the supplier | | | | | | | |
| Viscosity at 40 °C, mm$^2$ s$^{-1}$, min.–max. (DIN 51 562-1) | | 9.0–11.0 | 13.5–16.5 | 19.8–24.2 | 28.8–35.2 | 41.4–50.6 | 61.2–74.8 | 90.0–110 | 135–165 |
| Viscosity at 100 °C, mm$^2$ s$^{-1}$, min. (DIN 51 562-1) | | To be reported by the supplier | | | | | | | |
| Viscosity index, min. (DIN ISO 2909) | | 140 | | | | | | | 120 |
| Pour point, °C, max. (DIN ISO 3016) | | –39 | –39 | –39 | –30 | –27 | –24 | –21 | –18 |
| Flash point (COC), °C, min. (DIN EN ISO 2592) | | 125 | 125 | 175 | 175 | 180 | 180 | 190 | 200 |
| Purity class (ISO 4406:1999) | | 21/19/16 (defined requirements depend on the system) | | | | | | | |
| Contamination with solid particles, mg kg$^{-1}$, max. (DIN ISO 5884 or ISO 4405:1991) | | 50 | | | | | | | |
| Filterability without $H_2O$, Stage I $F_I$/Stage II $F_{II}$, min. % (E DIN ISO 13357-2) | | 80/60 | | | | | | | |
| Filterability with $H_2O$, Stage I $F_I$/Stage II $F_{II}$, min. % (E DIN ISO 13357-1) | | 70/50 | | | | | | | |
| Demulsibility, 54 °C, minutes, max. | (DIN ISO 6614) | 20 | | | 30 | | | – | |
| Demulsibility, 82 °C, minutes, max. | | – | | | – | | | 30 | |
| Water content in % $m/m$, max. (DIN EN ISO 12937 – method A) | | 0.05 | | | | | | | |
| Steel corrosion, method A (DIN ISO 7120) | | Pass | | | | | | | |
| Copper corrosion, 100 °C, 3 h, degree of corrosion, max. (DIN EN ISO 2160) | | 2 | | | | | | | |
| Ageing properties (maximum increase in neutralization number after 1000 h), mg KOH g$^{-1}$, max. (DIN 51 587 or DIN EN ISO 4263-1) | | ≤ 2.0 | | | | | | | |
| Behavior toward the SRE-NBR 1 sealant after 7 days ± 2 h at (100 ± 1) °C | Relative change in volume, % | 0 to 18 | 0 to 15 | | 0 to 12 | | 0 to 10 | | |
| (DIN 53 521, DIN ISO 1817, and DIN 53 505) | Change in Shore A hardness | 0 to –10 | 0 to –8 | | 0 to –7 | | 0 to –6 | | |
| Air release, 50 °C, minutes , max. (DIN ISO 9120) | | 5 | | | | | 13 | 21 | 32 |
| Foam volume, in mL, max. (ISO 6247:1998, incl. Corr. 1: 1999)<br>at 24 °C<br>at 93.5 °C<br>at 25 °C after 95 °C | | 150/0<br>75/0<br>150/0 | | | | | | | |
| FZG mechanical gear test rig, A/8.3/90: failure load stage, min. (DIN 51 354-2 or DIN ISO 14635-1) | | – | – | – | 10 | | | | |
| Vane pump wear, mg (DIN 51 389-2) | Ring, max. | – | – | – | 120 | | | – | – |
| | Vanes, max. | – | – | – | 30 | | | – | – |
| Relative viscosity reduction due to shearing after 20 h (DIN 51 350-6) | at 40 °C<br>at 100 °C | To be reported by the supplier<br>To be reported by the supplier | | | | | | | |

**Tab. 11.6** Minimum requirements of hydraulic fluids – ISO 6743. Part 4 – HM, ISO 11158.

***ISO 6743/4, ISO 11158: Specification for type HM mineral oil hydraulic fluids***
***Oils with improved anti-rust and anti-oxidant and anti-wear properties (a typical application is for general hydraulics)***

| Characteristics or Test | Units | Specifications | | | | | | | | Standard or Test Method |
|---|---|---|---|---|---|---|---|---|---|---|
| Viscosity grade | | VG 10 | VG 15 | VG 22 | VG 32 | VG 46 | VG 68 | VG 100 | VG 150 | ISO 3448 |
| Density at 15°C | kg/dm$^3$ | 1) | 1) | 1) | 1) | 1) | 1) | 1) | 1) | ISO 3675 |
| Colour 2) | | 1) | 1) | 1) | 1) | 1) | 1) | 1) | 1) | ISO 2049 |
| Appearance at 25°C 3) | | Clbr | Clbr | Clbr | Clbr | Clbr | Clbr | Clbr | Clbr | |
| Flash point, | | | | | | | | | | |
| Cleveland open cup, min. | °C | 100 | 140 | 140 | 160 | 180 | 180 | 180 | 180 | ISO 2592 |
| Pensky-Martens closed cup, min. | °C | 88 | 128 | 148 | 168 | 168 | 168 | 168 | 168 | ISO 2719 |
| Kinematic viscosity at 40°C, min.-max. 4) | mm$^2$/s 4) | 9,0–11,0 | 13,5–16,5 | 19,8–24,2 | 28,8–35,2 | 41,4–50,6 | 61,2–74,8 | 90,0–110 | 135–165 | ISO 3104 & ISO 3105 |
| Viscosity Index | | 1) | 1) | 1) | 1) | 1) | 1) | 1) | 1) | ISO 2909 |
| Pourpoint, max. | °C | –30 | –21 | –18 | –15 | –12 | –12 | –12 | –12 | ISO 3016 |
| Neutralisation value 5) | mg KOH/g | 1) | 1) | 1) | 1) | 1) | 1) | 1) | 1) | ISO 6618 |
| Water content | % (m/m) | 0,05 | 0,05 | 0,05 | 0,05 | 0,05 | 0,05 | 0,05 | 0,05 | ISO/DIS 6296 or DIN 51777-2 6) |
| Copper corrosion, 100°C, 3 h, max. | | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | ISO 2160 |
| Rust prevention, procedure A | | Pass | Pass | Pass | Pass | Pass | Pass | Pass | Pass | ISO 7120 |
| Foam: at 24°C, max. | ml | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | ISO/DIS 6247 |
| at 93°C, max. | ml | 75/0 | 75/0 | 75/0 | 75/0 | 75/0 | 75/0 | 75/0 | 75/0 | |
| at 24°C, max. | ml | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | |

**Tab. 11.6** Continued.

***ISO 6743/4, ISO 11158: Specification for type HM mineral oil hydraulic fluids***
***Oils with improved anti-rust and anti-oxidant and anti-wear properties (a typical application is for general hydraulics)***

| Characteristics or Test | Units | Specifications | | | | | | | | Standard or Test Method |
|---|---|---|---|---|---|---|---|---|---|---|
| Air release, 50°C, max. | min. | 5 | 5 | 5 | 5 | 10 | 13 | 21 | 32 | ISO/DIS 9120 |
| Water separation | | | | | | | | | | |
| time to 3 ml emulsion at 54°C | min. | 30 | 30 | 30 | 30 | 30 | 30 | – | – | ISO 6614 |
| time to 3 ml emulsion at 82°C | min. | – | – | – | – | – | – | 30 | 30 | |
| Elastomer compatibility 7) | | 1) | 1) | 1) | 1) | 1) | 1) | 1) | 1) | ISO 6072 |
| Oxidation stability, 1 000 h: | | | | | | | | | | |
| delta neut. number, max. | mg KOH/g | – | – | 2,0 | 2,0 | 2,0 | 2,0 | 2,0 | 2,0 | ASTM D 4310 |
| insoluble sludge | mg | – | – | 1) | 1) | 1) | 1) | 1) | 1) | |
| Wear protection, FZG A/8.3/90, min. 8) | Fail stage | – | – | – | 10 | 10 | 10 | 10 | 10 | DIN 51354 |
| Vane pump 9) | | – | – | 1) | 1) | 1) | 1) | – | – | BS 2000: part 281 or ASTM D 2882 |

1) Report only
2) For purposes of identification, dye may be used by agreement between supplier and end user.
3) Clear-bright is abbreviated as Clbr. Cleanliness level expressed according to ISO 4406 may be used by agreement between supplier and end user.
4) mm$^2$/s is equivalent to cSt
5) Initial neutralization number is influenced by the presence of functional moieties in the total additive package.
6) DIN 51777-2 is applied to instances where interference by certain chemicals can be avoided.
7) The type of elastomer and definition of compatibility are to be agreed between supplier and end user.
8) Applicable from ISO VG 32 to ISO VG 150
9) Applicable from ISO VG 22 to ISO VG 68

HV according to ISO 6743/4 (HR = HVL, without EP additives ISO 6743/4) [11.25].

The hydraulic oil grades described above which fulfil DIN 51 524 and ISO 6743/4 all have demulsifying properties. They meet the demulsifying thresholds laid down in DIN 51 599 and ISO 6614.

In contrast with this, detergent and dispersing lubricants cannot fulfil the requirements relating to water separation.

The classification standard, DIN 51 502, identifies detergent/dispersant oils with the letter D. Because tangible requirements are not specified, direct comparison of ISO classifications is not possible. In the broad sense, HG hydraulic oils according to ISO 6743/4 can be allocated to this group [11.24, 11.25].

#### 11.4.5.5 **HLPD Hydraulic Oils**

Compared with HLP products, these hydraulic oils contain DD additives which finely disperse, suspend, and emulsify water, dirt, aging products and contaminants and thus hinder the accumulation of deposits on hydraulic components. The mostly polar DD additives also reduce friction and wear. Their polarity also affords good wetting, reduces stick–slip and leads to a lower coefficient of friction. These oils are often used in machine tools and other systems with sensitive control valves because they combat deposits. For machine tools, where cutting fluids can contaminate the hydraulic system, HLPD oils combat valve and cylinder gumming by emulsifying ingressed cutting fluid. HLPD oils hinder the precipitation of solid and fluid impurities. These are kept in suspension and are removed at the filtering stage. This is why filter capacities must often be increased if HLPD oils are used [11.2, 11.4, 11.6].

### 11.4.6
### Fire-Resistant Hydraulic Fluids

Fire-resistant fluids have been developed for mining, die-casting, steel mill, and aviation applications. These fluids have significantly higher ignition temperatures or fire-resistant properties and thus afford better fire protection than mineral oils. Use of these fluids is compulsory for some applications, for example underground coal mining.

Fire-resistant hydraulic fluids are classified according to the Luxembourg Report [11.14], ISO 6743/4 [11.25], VDMA sheets [11.26, 11.27], CETOP RP97H [11.28], DIN 51 502 and Factory Mutual (FM-USA) [11.10]. Evaluated factors include the physical characteristics of the different types of fluid and their fire-resistance and technical specifications. The grading of the different types of fluid are identical in DIN 51 502, ISO 6743/4 and the Luxembourg Report [11.10, 11.25].

- HFAE: Oil-in-water emulsions (mineral oil-based)
- HFAS: Synthetic, water-based solutions (ester or/and polyglycol-based)
- HFB: Water-in-oil emulsions (mineral oil-based)
- HFC: Water-based monomer and polymer solutions (polyglycol-based)
- HFD: Water-free fluids (ester-based)

#### 11.4.6.1 HFA Fluids

HFA fluids are seldom used in industrial applications because of their poor anti-wear characteristics and very low viscosity. The additives used in HFA fluids usually improve corrosion protection for steels and non-ferrous metals, guarantee the bio-stability of the fluid and ensure compatibility with sealing materials (seal wear-reduction). HFA fluids also contain friction-reducing additives but these only give limited protection against wear. Their principal use is in mining hydraulics and in some non-European steel mills [11.33]. The practical concentration of mineral oil-based HFAE emulsions or synthetic HFAS solutions is between 1 and 5 % in water (depending on water quality). Demands in Germany and other parts of the world for better HFA fluid biodegradability and less water pollution caused by HFA fluids, because large industrial leakages of these fluids pollute the soil and ground water, have led to new product developments. HFA fluids are gaining acceptance as hydraulic fluids in hydroforming machinery and in industrial robots used in the automobile industry (concentration 5–7 % in water). HFA concentrates are also recommended for plunger-pump-systems to protect the circuit against corrosion.

HFA fluids have to be carefully matched to the hardness of the water used to ensure adequate stability. This is particularly important in the mining industry [11.14, 11.33]. HFA fluids should be used at their recommended concentrations to ensure optimum technical performance and bio-stability (e.g. crevice corrosion).

#### 11.4.6.2 HFB Fluids

HFB fluids are water-in-oil emulsions with a (flammable) mineral oil content of ca. 60 % (*m*/*m*). HFB fluids are used almost only in the UK mining industry and in other UK-influenced countries (Commonwealth countries). Because of their high mineral oil content, these fluids do not pass prescribed spray-ignition tests in Germany and several other countries [11.14].

#### 11.4.6.3 HFC Fluids

HFC fluids are normally based on a mixture of min. 35–50 % fully demineralized water with polyglycols (monomers or polymers) as thickeners. Low molecular-weight ethylenes and/or propylene glycols are used to improve low-temperature behavior of the fluid. Carefully matched additive systems which improve wear and corrosion protection and foaming behavior ensure the required tribological properties. HFC fluids are normally somewhat alkaline (pH > 9) and contain fluid and gas-phase corrosion inhibitors. HFC hydraulic fluids can be used at pressures of up to and above 200–250 bar (new pumps > 350–400 bar). HFC fluids are used for a wide range of applications in the steel-making industry, in foundries, in forging plants, in die-casting machines and in hydraulic presses and wherever pressurized hydraulic fluid leaks pose a fire hazard. The temperature range of HFC fluids is between –20 (start-up) and +60 °C (working temperature) because higher temperatures would lead to large fluid losses as a result of the high water content and the evaporative loss at high temperatures (vapor pressure) [11.14, 11.33].

#### 11.4.6.4 HFD Fluids

HFD fluids are synthetic, water-free and fire-resistant hydraulic fluids. Usually HFDR fluids based on phosphoric acid esters or HFDU fluids based on carboxylic acid esters or polyolesters are used. Until the 1980s, HFD fluids were the predominant hydraulic fluids. HFDS fluids based on chlorinated hydrocarbons then disappeared from the market because of PCB problems and were replaced by HFC fluids for mining applications and by HFDU fluids for industrial applications. These days, HFDR fluids containing phosphoric acid esters are used as control and regulator fluids in turbines and as hydraulic fluids in aviation systems [11.6]. HFDU fluids based on carboxylic acids and polyolesters make up the largest segment of HFD fluids [11.6]. They are normally used for hydrodynamic clutches and high-performance hydraulic systems at pressures of 250–350 bar and temperatures of 70–100 °C (and higher). HFDU fluids have tribological properties similar to those of mineral oils [11.2, 11.5, 11.6]. They show excellent characteristics in boundary lubrication and in biodegradability. The ester-based HFDU fluids currently on the market are only roughly comparable with HFC fluids in terms of fire protection.

### 11.4.7
### Biodegradable Hydraulic Fluids

Environmentally friendly, rapidly biodegradable hydraulic fluids were originally developed to ensure ecological compatibility. They are used in stationary and mobile systems. Their market share is growing rapidly and they are replacing mineral oil-based hydraulic fluids in several areas. This trend has been underlined by the creation of VDMA guideline 24568 [11.11] which specifies minimum requirements for HETG, HEES and HEPG fluids and the inclusion of these three fluid groups into ISO 6743/4 [11.25] in combination with the new ISO 15 380 specification (enlarged to include HEPR).

Rapidly biodegradable hydraulic fluids are allocated to several product families according to VDMA 24568 (minimum technical requirements) [11.11], VDMA 24569 (change-over guidelines) [11.11] and ISO 6743/4 [11.25] and ISO 15380:

- HETG: triglyceride (non-water-soluble, vegetable-oil types)
- HEES: synthetic ester types (non-water-soluble)
- HEPG: polyglycol types (water-soluble)
- HEPR: polyalphaolefins and related hydrocarbon products.

Table 11.7 shows the range of fire-resistant and environmentally friendly hydraulic fluids.

#### 11.4.7.1 HETG: Triglyceride, Vegetable-Oil Types

The natural esters used for the HETG group of lubricants are primarily triglycerides. These are triple-valence glycerols and (fatty) acids. The most important esters are rapeseed oil but sunflower oil is also used. The physical and chemical properties of these base oils result from their fatty acid distribution. Natural fatty acids have even numbers and are un-branched. Fatty acids can be saturated but also mono or multiply unsaturated fatty acids are used. In general, one can say that a high proportion

**Tab. 11.7** Classification of fire resistant fluids and rapidly biodegradable fluids.

| ***Category according to 7th Luxemb. Report, DIN 51 502, and ISO 6743/4*** | ***Composition<br>Typical characteristics*** | ***Field of Application<br>Operating temperatures*** |
|---|---|---|
| | ***Water containing fire resistant hydraulic fluids*** | |
| HFA E | Oil-in-water-emulsions, mineral oil or synth. ester<br>Water content > 80 % | Power transmissions, about 300 bar, high working pressures, powered roof support<br>hydrostatic drives, about 160 bar, low working pressures<br>5 to < 55 °C |
| HFA S | Mineral oil free aqueous synth. chem. solutions<br>Water content > 80 % | |
| HFB | Water-in-oil-emulsions<br>Mineral oil content about 60 % | e.g. in the British mining industry, not approved in Germany<br>5 to 60 °C |
| HFC | Water polymer solutions<br>water content > 35 % | Hydrostatic drives, Industry and mining applications<br>–20 to 60 °C |
| | ***Water free, synthetic fire resistant hydraulic fluids*** | |
| HFD R | Water-free synthetic fluids, consisting of phosphate esters,<br>not soluble in water | Lubrication and control of turbines, industrial hydraulics<br>–20 to 150 °C,<br>in hydrostatic applications often<br>10 to 70 °C |
| HFD U | Water-free synthetic fluids of other compositions (e.g. carboxylic acid esters) | Hydrostatic drives, industrial hydraulic systems<br>–35 to < 90 °C |
| ***Category according to VDMA 24568 and ISO 15380*** | ***Composition<br>Typical characteristics*** | ***Field of Application<br>Operating temperatures*** |
| | ***Water free, rapidly biodegradable hydraulic fluids*** | |
| HEPG | Polyalkylene glycols<br>soluble in water | Hydrostatic drives, e.g. locks,<br>'water hydraulics'<br>– 30 to < 90 °C (reservoir temp. –20 to 80 °C) |
| HETG | Triglycerides (vegetable oils)<br>not soluble in water | Hydrostatic drives, e.g. mobile hydraulic systems<br>–20 to < 70 °C (reservoir temp. –10 to 70 °C) |
| HEES | Synthetic esters<br>not soluble in water | Hydrostatic drives, mobile and industrial hydraulic systems<br>–30 to < 90 °C (reservoir temp. –20 to 80 °C) |
| HEPR | Polyalphaolefins and/or related hydrocarbons<br>not soluble in water | Hydrostatic drives, mobile and industrial hydraulic systems<br>–35 to < 80 °C (reservoir temp. –30 to 100 °C) |

of unsaturated or short-chain fatty acids produces lubricants with a low pour-point. A higher proportion of double bonds (unsaturated fatty acids) increases the sensitivity of a lubricant to oxygen and higher temperatures. Natural ester triglycerides are obtained from the seeds of oil plants such as rape or sunflower by pressing or extraction. The oil thus obtained is subjected to a number of purification processes. Rapeseed oil and other natural fatty acids can be used as the raw material for HETG lubricants or as a raw material for synthetic esters.

The selection of the most suitable natural ester and the corresponding matched additive system is of fundamental importance to the quality of an environmentally friendly lubricant based on harvestable raw materials. Because HETG hydraulic oils withstand lower thermal and oxidative loads than mineral oils, they are used for medium-temperature and low-pressure applications (often in total-loss application).

The minimum requirements of triglycerides are described in VDMA 24568 and in ISO 15380 Table 1 [11.2, 11.5, 11.6, 11.13, 11.34].

#### 11.4.7.2 **HEES: Synthetic Ester Types**

Synthetic esters are a group of substances with wide variation in structure. Esters are manufactured by chemically altering alcohols and acids. Alcohols and acids from a broad range of raw materials can be randomly combined to achieve the desired product characteristics such as thermal and hydrolytic stability, low-temperature properties, seal compatibility, etc. The chain length and the branching characteristics (linear or branched) of alcohols can be varied, as can the number of OH groups (monoalcohols, diols, polyols). The acids can include monocarboxylic acids, dicarboxylic acids and polycarboxylic acids with aliphatic, aromatic, straight, or branched structures. They can be saturated or partially unsaturated [11.6, 11.13]. The most widely used synthetic esters are probably dicarboxylic acid esters, polyol esters (e.g. TMP esters) and complex esters (manufactured from more than one alcohol or acid component). The raw materials for synthetic esters originate from, on one hand, petrochemicals and, on the other, from natural materials and their chemical transformation. Partially unsaturated ester oils form the largest group of rapidly biodegradable lubricants. Fully synthetic, saturated ester oils based on saturated TMP, polyol and complex esters give the best technical performance. Their advantages include outstanding oxidation stability, good material compatibility and excellent tribological performance. These products, which are much more expensive than mineral oils, are used in high-pressure, high-temperature and highly stressed hydraulic systems, for example mobile hydraulics and hydrostatic drives, under difficult conditions [11.35–11.39].

The minimum requirements for ester oils are specified by VDMA 24568, ISO 15380, Group HEES, synthetic esters [11.11]. This does not differentiate between partially unsaturated and saturated systems. The VDMA paper 24568 is substituted by the official edition of ISO 15380 – dated 2002 [11.12]. Table 11.8 shows the requirements of type HEES hydraulic fluids according to ISO 15380. It lists synthetic esters according to their aging stability (unsaturated systems aging stability according to Baader: 72 h at 110 °C synthetic saturated esters – aging stability in the Tost test without the addition of water). Furthermore, special material compatibility tests (such as nonferrous metal compatibility according to Linde) are included in the specifications [11.13].

#### 11.4.7.3 HEPG: Polyglycol Types

Polyglycols differ from the hydraulic fluids already mentioned by being water soluble. Depending on the substances used to manufacture the polyglycol (polyethylene oxide, polypropylene oxide), their molecular mixture ratio, and the alcohol start-up molecule used, polyglycols of differing make-up can be synthesized. Ethylene oxide-based polyglycols are highly water-soluble, poorly miscible with mineral oil, and of high polarity. Polyglycols with a high proportion of propylene oxide are not water-soluble, or only slightly so, to some extent miscible with mineral oil, and are significantly less polar than polyethylene glycols. As a result of their 'water solubility', glycol-based hydraulic fluids can, and may, contain water. The water-solubility of polyglycols (and thus their mobility in the ground–ground water contamination) and their incompatibility with mineral oils has limited their acceptance. Polyglycols are primarily used in the water-supply industry, in canal lock hydraulics, and in off-shore applications as rapidly biodegradable hydraulic fluids especially when the application leads to unavoidable contamination of the hydraulic fluid with ingressed water [11.13].

#### 11.4.7.4 HEPR: Polyalphaolefin and Related Hydrocarbon Products

Because of their biodegradability, low-molecular weight polyalphaolefins and the correspondingly derived hydrocarbons are classified as environmentally friendly hydraulic fluids. In ISO 15380 [11.12] these products were given the group title HEPR. The inclusion of this new group in ISO 15380 is an extension compared with the "old VDMA 24568" [11.11]. HEPR fluids are more rapidly biodegradable than mineral oils but significantly less biodegradable than most ester oils and natural oils such as rapeseed oil. The technical properties of these oils are similar to those of mineral oils but they have the advantage of faster biodegradability.

### 11.4.8
### Food-Grade Hydraulic Fluids

The classification of foodstuff-compatible hydraulic oils is based on FDA (Food and Drug Administration) and NSF guidelines [11.29]. The NSF issue of H1 or H2 lubricant approvals depends on the FDA classification of components such as base oils and additives (FDA Code of federal regulations, 21 CFR 178.3570). The European hygiene guideline 93/43 EWG and the Hazard Analysis and Critical Control Points (HACCP) Management System, promotes the use of safe lubricants in the food-processing industries. The NSF organization has taken over the activities of the USDA (United States Department of Agriculture) which has closed its activities [NSF International – The Public Health and Safety Company (a not-for-profit, nongovernmental organization – USA)].

**Tab. 11.8** Minimum requirements of hydraulic fluids – ISO 15380 – Part 4 – HEES (2002).

| ***Characteristics of Test*** | ***Units*** | ***Requirements*** | | | | | ***Test Method or Standard*** |
|---|---|---|---|---|---|---|---|
| Viscosity Grade | | 22 | 32 | 46 | 68 | 100 | ISO 3448 |
| Density at 15°C | $kg/m^3$ | a) | a) | a) | a) | a) | ISO 12185 |
| Colour b) | | a) | a) | a) | a) | a) | ISO 2049 |
| Appearance at 25°C c) | | Clbr | Clbr | Clbr | Clbr | Clbr | |
| Ash content, max. | % (m/m) | g) | g) | g) | g) | g) | ISO 6245 |
| Flash point Cleveland open cup, min. | °C | 165 | 175 | 185 | 195 | 205 | ISO 2592 |
| Kinematic viscosity d) | | | | | | | |
| at –20°C, max. | $mm^2/s$ | g) | g) | g) | g) | g) | ISO 3104 |
| at 0°C, max. | $mm^2/s$ | 300 | 420 | 780 | 1 400 | 1 500 | ISO 3104 |
| at 40°C, min.-max. | $mm^2/s$ | 19,8–24,2 | 28,8–35,2 | 41,4–50,6 | 61,2–74,8 | 90,0–110 | ISO 3104 |
| at 100°C, min. | $mm^2/s$ | 4,1 | 5,0 | 6,1 | 7,8 | 10,0 | ISO 3104 |
| Pourpoint, max. | °C | –21 | –18 | –15 | –12 | –9 | ISO 3016 |
| Low temperature fluidity after 7 days | °C | g) | g) | g) | g) | g) | ASTM D 2532 |
| Acid number e), max. | g KOH/g | g) | g) | g) | g) | g) | ISO 6618 |
| Water content, max. | mg/kg | 1 000 | 1 000 | 1 000 | 1 000 | 1 000 | ISO 12937 or<br>ASTM D 1744 or<br>DIN 51777-1 |
| Copper corrosion, 100°C 3 h, max. | rating | 2 | 2 | 2 | 2 | 2 | ISO 2160 |
| Rust prevention, procedure A | | Pass | Pass | Pass | Pass | Pass | ISO 7120 |

**Tab. 11.8** Continued.

| ***Characteristics of Test*** | ***Units*** | ***Requirements*** | | | | | ***Test Method or Standard*** |
|---|---|---|---|---|---|---|---|
| Viscosity grade | | 22 | 32 | 46 | 68 | 100 | ISO 3448 |
| Foam at 24°C, max. | ml | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | ISO 6247 |
| at 93°C, max. | | 75/0 | 75/0 | 75/0 | 75/0 | 75/0 | |
| at 24°C, max. | | 150/0 | 150/0 | 150/0 | 150/0 | 150/0 | |
| Air release, 50°C, max. | min | 7 | 7 | 10 | 10 | 14 | ISO 9120 |
| Water separation, time to 3 ml emulsion at 54°C, max. | min | g) | g) | g) | g) | g) | ISO 6614 |
| Elastomer compatibility f) HNBR, FPM, NBR1, AU after 1.000 h at | °C | 60 | 80 | 80 | 100 | 100 | ISO 6072 |
| Change in shore-A-hardness, max. | IRHD | ± 10 | ± 10 | ± 10 | ± 10 | ± 10 | ISO 6072 |
| Change in volume, max. | % | –3 to +10 | –3 to +10 | –3 to +10 | –3 to +10 | –3 to +10 | ISO 6072 |
| Change in elongation, max. | % | 30 | 30 | 30 | 30 | 30 | |
| Change in tensile strength, max. | % | 30 | 30 | 30 | 30 | 30 | |
| Oxidation stability | | | | | | | |
| Modified TOST, dry TOST | h | g) | g) | g) | g) | g) | ASTM D943 l) |
| Baader test, 110°C, 72 h | | | | | | | |
| Increase in viscosity at 40°C, max. | % | 20 | 20 | 20 | 20 | 20 | DIN 51554-3 |
| Wear protection, FZG A/8.3/90, min. | failure load stage | i) | 10 | 10 | 10 | 10 | DIN 51354-2 |
| Vane pump j) weight loss | | | | | | | IP 281 or ASTM D 2882 |
| Ring, max. | mg | 120 | 120 | 120 | 120 | 120 | |
| Vane, max. | mg | 30 | 30 | 30 | 30 | 30 | |

**Tab. 11.8** Continued.

| ***Characteristics of Test*** | ***Units*** | ***Requirements*** | | | | | ***Test Method or Standard*** |
|---|---|---|---|---|---|---|---|
| Viscosity grade | | 22 | 32 | 46 | 68 | 100 | ISO 3448 |
| Biodegradability [k), m)] | % | 60 | 60 | 60 | 60 | 60 | ISO 14593 or ISO 9439 |
| Toxicity [h)] | | | | | | | |
| Acute fish toxicity, $LL_{50}$, min. | mg/l | 100 | 100 | 100 | 100 | 100 | ISO 7346-2 |
| Acute daphnia toxicity, $EC_{50}$, min. | mg/l | 100 | 100 | 100 | 100 | 100 | ISO 6341 |
| Inhibition, $EC_{50}$, min. | mg/l | 100 | 100 | 100 | 100 | 100 | ISO 8192 |

a) Report
b) For purpose of identification, dye may be used by agreement between supplier and end user
c) Clear-bright is abbreviated as Clbr.
d) $mm^2/s$ is equivalent to cSt
e) Initial acid number is influenced by the presence of functional moieties in the total additive package
f) The type of elastomer and definition of compatibility are to be agreed between supplier and end user
g) Criteria of performance or characteristics values to be negotiated between supplier and end userh)
This standard may involve hazardous materials, operation and equipment. This standard does not purport to address all of the safety problem associated with its use. It is the responsibility of whoever use this standard to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitation prior to use. The fluids must
not present any significant hazard to health when correctly used in hydraulic equipment observing supplier's handling recommendations.
i) Not applicable to viscosity grade 22
j) State test method employed
k) Some national requirements may be more severe
l) Modification consisting in driving the oxidation stability test without water, water is replaced by hydraulic fluid.
m) Without 10 day window requirement

#### 11.4.8.1 **NSF H2 Lubricants**

This classification indicates that these lubricants are suitable for general use in the foodstuffs industry if no direct contact with the foodstuffs can occur [11.29].

#### 11.4.8.2 **NSF H1 Lubricants**

The classification USDA H1 stands for food grade lubricants which can be used in the food-processing industries if occasional, technically-unavoidable, contact between the foods and the lubricant occurs. In other words, if there is a chance of lubricants coming into contact with foods, the lubricants must be NSF H1 approved [11.29].

The base oils for such products are principally special white oils, special polyalphaolefin grades and special polyglycols. NSF H2 lubricants can be standard lubricants if special precautions are taken. Although the USDA is not issuing any new approvals at present, all previously issued approvals remain valid and NSF has taklen over the approval activities. DIN and European activities for this group of lubricants are currently being discussed [11.40].

### 11.4.9
### Automatic Transmission Fluids (ATF)

ATF are functional fluids for automatic transmissions in vehicles and machinery. In special circumstances ATF are used in manual synchromesh gearboxes and hydraulic gearboxes/transmissions. These products have excellent viscosity–temperature behavior, shear stability, high oxidation stability, minimal foaming, and outstanding air release. As a result of different manufacturers' demands on a fluid's friction characteristics (coefficient of friction on the brake and clutch bands), there are several types of AFT (see Chapter 9) [11.15, 11.30].

### 11.4.10
### Fluids in Tractors and Agricultural Machinery

Gear oils and hydraulic fluids are specified for the gearboxes and hydraulic systems of tractors and agricultural machinery fitted with and without 'wet' brakes. The most-commonly used types of oil are UTTO (universal tractor transmission oil), STUO (super tractor universal oil), and MFO (multi-functional oil) (see Chapter 9) [11.15, 11.30].

### 11.4.11
### Hydraulic Fluids for Aircraft

Since the 1930s the use of hydraulic systems in aviation has increased sharply. The principal functions of aircraft hydraulics are controlling the elevator, rudder, ailerons, flaps, landing gear, doors, and brakes, etc. The most commonly used pumps are of the axial piston type with pressures of between 100 and 280 bar. Fluid volumes, between 20 and 40 L, are relatively small. The hydraulic fluids used must be free from particle contaminants because aircraft hydraulics have to work under extreme climatic conditions with enormous fluctuations in temperature. The hydraulic fluids must have outstanding thermal stability and good viscosity–temperature behavior. These fluids should also be fire-resistant. A wide variety of different hydraulic fluids are currently used in aircraft. These include those based on phosphoric acid esters, polyalphaolefins, silicate esters and special mineral oils. One standard they must fulfil is US military specifications, e.g. MIL H 5606 or others. These specifications serve as basis for other specifications issued by manufacturers of aircraft and aircraft hydraulics [11.6].

### 11.4.12
### International Requirements on Hydraulic Oils

The requirements of hydraulic fluids are normally set out in national standards or company-specific specifications. Apart from ISO 6743/4 and DIN 51 524, there are several specifications issued by leading component manufacturers. Although these specifications often overlap, there are differences in detail, normally in relation to thermal/oxidation stability, mechanical/dynamic tests, filterability tests, and in-house tests performed by some manufacturers. Table 11.9 shows US Steel's 126 and 127 requirements and Table 11.10 is a summary of the most important OEM hydraulic oil specifications.

**Tab. 11.9** Performance requirements of US hydraulic specifications US Steel 126 & 127.

| *Performance requirements ASTM-test* | *US Steel 126* | *US Steel 127* |
|---|---|---|
| Viscosity D-88 | Suitable for specified application | Suitable for specified application |
| Viscosity index D-567 | Not less than 80 | Not less than 90 |
| COC Flash point D-92 | 375 °F minimum | 375 °F minimum |
| Hydraulic pump test D-2882 (100 h @ 2000 psig) | 0.05 % Total wear (450 mg maximum) | 0.010 % Total wear (90 mg maximum) |
| Four-ball wear test D-2266 (40 kg, 1800 rpm, 130 °F, 1 h) | No more than 0.80 mm scar diameter | Not more than 0.50 mm scar diameter |
| Rotary bomb oxidation D-2272 | 120 min minimum | 120 min minimum |
| Low-temp. cycling test (US steel method) | Must pass @ 15 °F | Must pass @ 15 °F |
| Water emulsion test D-1401 @ 130 °F | ml ml ml<br>oil water emulsion<br>40 37 3<br>$\leq$ 30 min | ml ml ml<br>oil water emulsion<br>40 37 3<br>$\leq$ 30 min |
| Rust-prevention test D-665 A | No rust | No rust |

**Tab. 11.10** Major OEM hydraulic oil specifications

| ***Performance: ASTM-test*** | ***Denison HF-0 (piston and vane pump test)*** | ***Denison HF-1 (piston pump test)*** | ***Denison HF-2 (vane pump test)*** | ***Vickers1) / Mannesmann Rexroth2)*** | ***Cincinnati Milacron P-68,P-69, P70*** | ***Rust and Oxidation Cincinnati Milacron P-38, P-54, P-55, P-57*** |
|---|---|---|---|---|---|---|
| SLUDGE & METAL CORROSION (1000 hr. D4310) | | | | | | |
| Neutralisation No. (D974) | 1.0 mg KOH/g max. | 1.0 mg KOH/g max. | 1.0 mg KOH/g max. | | | |
| Insoluble Sludge max. | 100 mg max. | 100 mg max. | 100 mg max. | | | |
| total Copper max. | 200 mg max. | 200 mg max. | 200 mg max. | | | |
| C.M. THERMAL STABILITY TEST – 168 hour at 135 °C, Cin. P70 (ISO VG 46) | | | | | 5 % max. change | 5 % max. change |
| Results After Test: | | | | | | |
| Viscosity (D445, D2161) | | | | | | |
| Neutralisation No. (D744) | | | | | + 50 % max. change | 0.15 max. increase |
| Sludge or Precipitate | 100 mg/100 ml max. | 100 mg/100 ml max. | 100 mg/100 ml max. | | 25 mg/100 ml max. | 25 mg/100 ml max. |
| Conditions of Steel Rod | | | | | | |
| Visual | | | | | No discoloration | No discoloration |
| Deposits (per 200 ml) | | | | | 3.5 mg max. | 3.5 mg max. |
| Metal Removed (per 200 ml) | | | | | 1.0 mg max. | 1.0 mg max. |
| Condition of Copper Rod | Report | Report | Report | | 5 max. | 5 max. |
| Copper rod rating Visual (CM) | 10 mg max. | 10 mg max. | 10 mg max. | | 10.0 mg max. | 10.0 mg max. |
| Copper weight loss | | | | | | |
| HYDROLYTIC STABILITY (ASTM D2619) | | | | | | |
| Copper Wt. Loss (mg/cm$^2$) | 0.2 max. | 0.2 max. | 0.2 max. | | | |
| $H_2O$ Acidity (mg KOH) | 4.0 max. | 4.0 max. | 4.0 max. | | | |

**Tab. 11.10** Continued.

| *Performance: ASTM-test* | *Denison HF-0 (piston and vane pump test)* | *Denison HF-1 (piston pump test)* | *Denison HF-2 (vane pump test)* | *Vickers*[1) ] */ Mannesmann Rexroth*[2)] | *Cincinnati Milacron P-68,P-69, P70* | *Rust and Oxidation Cincinnati Milacron P-38, P-54, P-55, P-57* |
|---|---|---|---|---|---|---|
| PUMP WEAR TEST (ASTM D2882) | | | | | 50 mg max.<br>100 hours | |
| DENISON AXIAL PISTON & VANE PUMP WEAR TEST (in co-operation with Denison when deemed necessary) | T6H20C according to TP 30533 | P-pump | T-pump | | | |
| VICKERS HP VANE PUMP TEST[1)]<br>MANNESMANN REXROTH INHOUSE PUMP TEST[2)] | | | | Total-90 mg max.[1)]<br>Inhouse Report[2)] | | |
| FILTERABILITY (DENISON PROCEDURE) | | | | | | |
| Filtration Time w/o Water | 600 secs max. | | 600 secs max. | | | |
| Filtration Time with Water | Not to exceed two times the filtration rate w/o water | | Not to exceed two times the filtration rate w/o water | | | |
| (PALL Filtration) | | | 80 min. | | | |
| RUST TEST (ASTM D665A) 24 hrs. | Procedure A&B | Procedure A&B | Procedure A&B | | Procedure A | Procedure A |
| FOAM (ASTM D892) after 10 min. | No foam | No foam | No foam | | | |
| NEUTRALISATION NUMBER (ASTM D974) mg KOH/g | | | | | 1.5 max. | 0.2 max. |

## 11.4.13
## Physical Properties of Hydraulic Oils and Their Effect on Performance

### 11.4.13.1 Viscosity, V–T Behavior

Viscosity is the most important measure of the load-carrying properties of a hydraulic oil. A differentiation is made between dynamic and kinematic viscosity [11.2, 11.5, 11.41, 11.42].

Industrial lubricants and hydraulic oils are allocated ISO viscosity grades on the basis of their kinematic viscosity which in turn is described by the ratio of dynamic viscosity and density. The reference temperature is 40 °C. The official SI unit for kinematic viscosity is $m^2 s^{-1}$. In the petroleum industry the unit cSt or $mm^2 s^{-1}$ is in use. The ISO viscosity classification, DIN 51 519, for liquid industrial lubricants defines 18 viscosity grades from 2–1500 $mm^2 s^{-1}$ at 40 °C. Every grade is defined by the average mean viscosity at 40 °C and the permissible deviation of ± 10 % of this value [11.43]. The viscosity–temperature behavior (*V–T*) is of great importance to hydraulic oils. Viscosity increases sharply as temperature falls and decreases as the temperature rises. In practical terms, the threshold viscosity of a fluid (permissible start-up viscosity, ca. 800–2000 $mm^2 s^{-1}$) must be observed for the different types of pump [11.16–11.18, 11.36, 11.37, 11.44]. The minimum permissible viscosity at higher temperatures is defined by the start of the boundary friction phase. The minimum viscosity should not be less than 7–10 $mm^2 s^{-1}$ to avoid unacceptable wear to pumps and motors [11.16–11.18, 11.21, 11.36, 11.37, 11.44]. In viscosity–temperature diagrams, the viscosity of a hydraulic fluid is plotted against temperature. In linear terms, *V–T* graphs are hyperbolic. By mathematical transformation, these *V–T* graphs can be shown as straight lines. These lines enable the exact viscosity to be determined over a wide temperature range. The measure of *V–T* behavior is the viscosity index (*VI*) and the *V–T* line gradient in the diagram. The higher the *VI* of a hydraulic oil, the less its viscosity changes with temperature, i.e. the flatter the *V–T* line (see Chapter 3). Mineral oil-based hydraulic oils usually have natural *VI* of 95–100. Synthetic, ester-based hydraulic oils have natural *VI* of 140–180 and polyglycols have natural *VI* of 180–200 (Fig. 11.10) [11.2, 11.5, 11.6, 11.44].

The Viscosity Index can also be increased by additives (polymeric additives which must be shear-stable) called *VI* improvers. Hydraulic oils of high *VI* enable easy pump start-up, less loss in performance at low ambient temperatures, and improved sealing and wear protection at high operating temperatures. High-*VI* oils improve the efficiency of a system and increase the life of components subject to wear (higher viscosity at operating temperatures will result in better volume efficiency).

### 11.4.13.2 Viscosity–Pressure Behavior

The viscosity–pressure behavior of a lubricant is principally responsible for the load-carrying properties of a lubricating film. The dynamic viscosity of fluid media increases with pressure. The following relationship governs the pressure-dependence of dynamic viscosity at constant temperature.

Viscosity–pressure behavior and the increase in viscosity at increasing pressures has a high positive effect on specific loading (such as on bearings) because the visc-

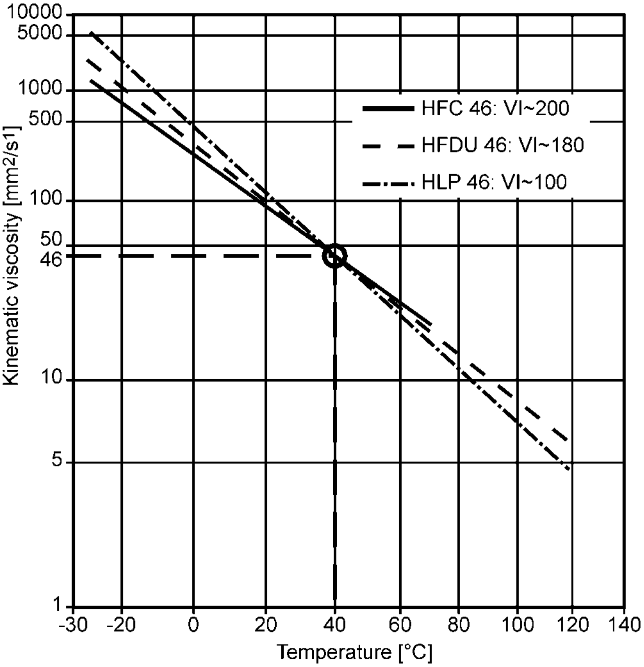


**Fig. 11.10** Viscosity index of different hydraulic fluids.

osity of the lubricating film increases when exposed to high partial pressure and this increases load-carrying capacity. Assuming an increase in pressure from 0 to 2000 bar, the viscosity would increase by factors of 2 for an HFC fluid, 30 for a mineral oil, and 60 for an HFD fluid, [11.2, 11.4]. This explains why roller bearings have a relatively short life if water-based (HFA, HFC) lubricants are used. Figures 11.12 and 11.13 show the dependence of viscosity on pressure for different hydraulic fluids. Viscosity–temperature behavior can also be described by an exponential statement (see Chapter 3) [11.2, 11.4, 11.6, 11.45, 11.46]:

$$\eta = \eta_0 \times e^{\alpha P}$$

where $\eta_0$ is the dynamic viscosity at atmospheric pressure, $\alpha$ is the viscosity–pressure coefficient, and $P$ is the pressure. For HFC, $\alpha = 3.5 \times 10^{-4}$ bar$^{-1}$; for HFD, $\alpha = 2.2 \times 10^{-3}$ bar$^{-1}$; and for HLP, $\alpha = 1.7 \times 10^{-3}$ bar$^{-1}$.

### 11.4.13.3 **Density**

Losses in the pipework and elements of a hydraulic system are directly proportional to the density of the fluid [11.2, 11.42]. For example, pressure losses are directly proportional to density:

$$\Delta P = (\rho/2) \times \zeta \times c^2$$

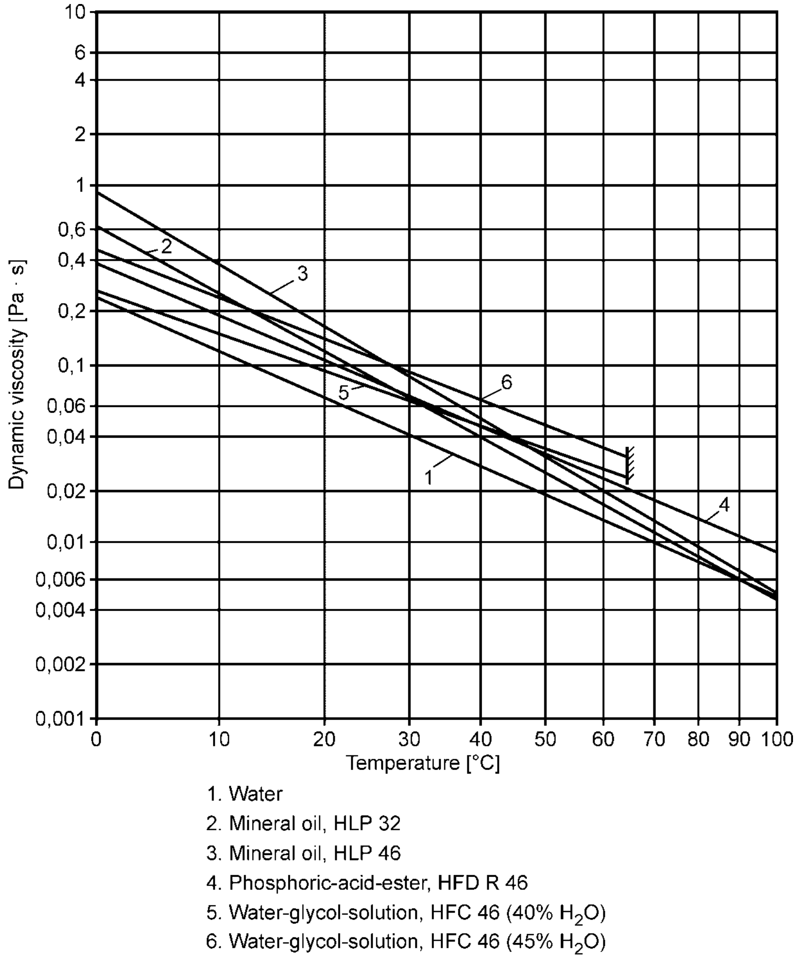


**Fig. 11.11** Viscosity–temperature characteristics of hydraulic fluids [11.2, 11.4, 11.5].

where $\rho$ is the density of the fluid, $\zeta$ is the resistance coefficient; $C$ is the fluid flow velocity, and $\Delta P$ the pressure loss. The density $\rho$, is the mass per unit volume of fluid.

$$\rho = m/V\,[\mathrm{kg\ m^{-3}}]$$

The density of hydraulic fluids is measured at 15 °C. The density of a hydraulic fluid is dependent on temperature and pressure because the volume of a fluid expands as its temperature increases. The change in volume of a fluid as a result of heating is thus:

$$\Delta V = V \times \beta_{(\mathrm{Temp.})} \times \Delta T$$

and this causes the change in density:

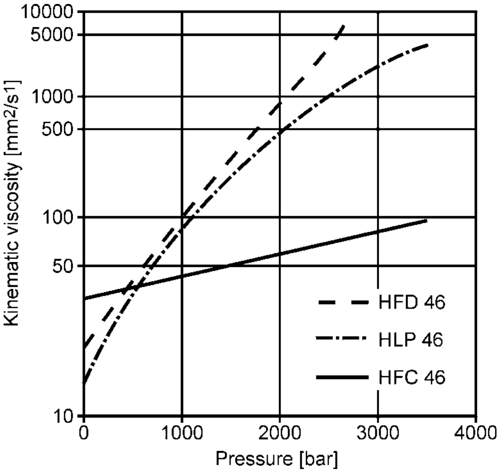


**Fig. 11.12** The pressure-dependence of kinematic viscosity.

$$\Delta\rho = \rho \times \beta_{(\text{Temp.})} \times \Delta T$$

In hydrostatics, it is sufficient to apply the linear formula to the above equation at temperatures between –50 and +150 °C. The thermal volume-expansion coefficient, $\beta_{(\text{Temp.})}$, can be applied to all different types of hydraulic fluid [11.2, 11.4–11.6, 11.45, 11.46].

| Base oil | $\beta_{(\text{Temp.})}$ [$K^{-1}$] Thermal volume-expansion coefficient at atmospheric pressure |
|---|---|
| HLP 46 Mineral oil | $0.65–0.70 \times 10^{-3}$ |
| HFC 46 Water, glycol | $0.75 \times 10^{-3}$ |
| HFD 46 Ester oil | $0.75 \times 10^{-3}$ |

As the coefficient of expansion for mineral oils is approximately $7 \times 10^{-4}\ K^{-1}$, the volume of a hydraulic oil would increase by 0.7 % (vol.) if its temperature increased by 10 °C. Figure 11.14 shows the temperature-dependence of the density of common hydraulic fluids [11.2, 11.4].

The density–pressure behavior of hydraulic fluids should also be included in a hydrostatic evaluation because the compressibility of fluids affect their dynamic performance. The dependence of density on pressure can simply be taken from corresponding diagrams (Fig. 11.15) [11.2, 11.4, 11.45, 11.46].

### 11.4.13.4 **Compressibility**

The compressibility of mineral oil-based hydraulic fluids depends on temperature and pressure. At pressures up to 400 bar and temperatures up to 70 °C which are top-end values in industrial systems, compressibility is irrelevant to the system. The hydraulic fluids used can be viewed as incompressible. At pressures from 1000 to 10 000 bar, however, changes in the compressibility of the medium can be observed. Compressibility is expressed in terms of the compressibility coefficient $\beta$ or the compressibility modulus $M$ (Fig. 11.16, M=K) [11.2, 11.45, 11.46].

$$M = 1/\beta \text{ bar} = 1/\beta \times 10^5 \text{ N m}^2 = 1/\beta \times 10^5 \text{ Pa}$$

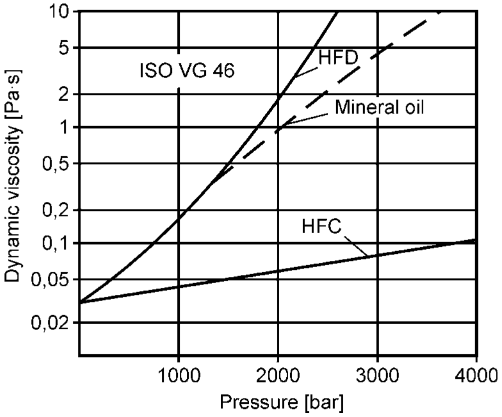


**Fig. 11.13** The pressure-dependence of dynamic viscosity.

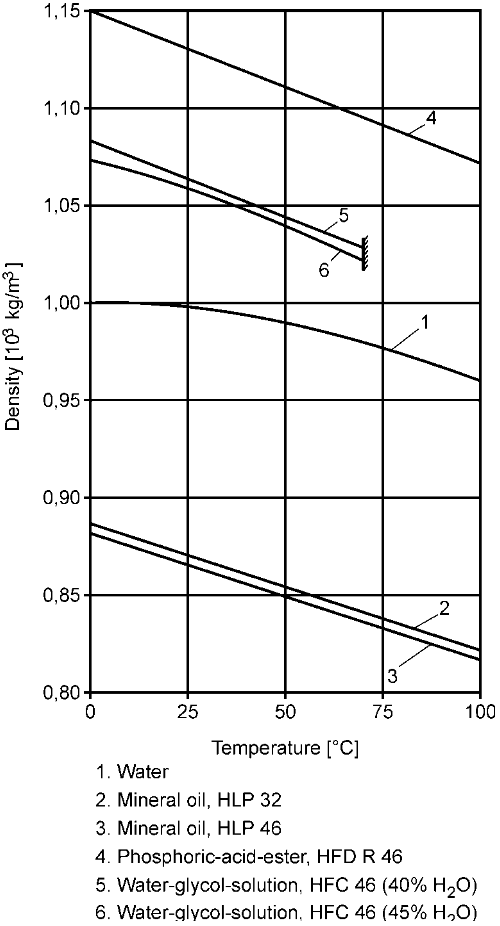


**Fig. 11.14** Temperature-dependence of the density for different hydraulic fluids.

The change in volume can be determined by use of the equation:

$$\Delta V = V \times \beta \times (P_x - P_{start})$$

where $\Delta V$ is the volume change, $P_x$ the maximum pressure, and $P_{Start}$ the initial pressure.

#### 11.4.13.5 **Gas Solubility, Cavitation**

Air and other gases can dissolve in fluids. A fluid can absorb a gas until a saturation is reached; this need not negatively affect the characteristics of the fluid. The solubility of a gas in a fluid depends on the base fluid, the type of gas, the pressure, and the temperature. Up to ca. 300 bar, gas solubility is proportional to pressure, and Henry's law applies [11.2, 11.6].

$$V_G = V_F \times \alpha_V \times P/P_0$$

where $V_G$ is the volume of gas dissolved, $V_F$ the volume of fluid, $P_0$ the atmospheric pressure, $P$ the fluid pressure, and $\alpha_V$ the Bunsen coefficient (1.013 mbar, 20 °C)

The Bunsen coefficient is highly dependent on the base fluid and indicates how much (% v/v) gas is dissolved in a unit volume of the fluid under normal conditions. The dissolved gas can escape from the hydraulic fluid at low static pressure (high flow velocity and shearing stress) until a new saturation point is reached. The speed at which the gas escapes is usually higher than the speed at which the gas is absorbed. Gas which escapes as bubbles changes the compressibility of the fluid similarly to air bubbles. Even at low pressures, small quantities of air can drastically reduce the incompressibility of a fluid. Up to ca. 5% (v/v) undissolved air can be found in mobile systems with high fluid circulation. This undissolved air has a highly negative effect on the performance, the load rigidity, and dynamics of a system (see Section 11.4.13.6, Air Release, and Section 11.4.13.7, Foaming). As the pressurization of fluids in systems is usually very rapid, any air bubbles can suddenly heat up to a high temperature (adiabatic compression). In extreme cases the ignition temperature of the fluid might be reached and micro-diesel effects can occur. The gas bubbles can also collapse in pumps as a result of pressurization and this can cause erosion damage (sometimes called cavitation or pseudo-cavitation) [11.2, 11.6]. The situation can become worse if vapor bubbles are created in the fluid. Cavitation thus occurs where the pressure falls below the gas solubility or vapor pressure of the fluid. Cavitation mainly occurs in open systems with constant volume. These can include inlet and intake circuits and pumps. The reasons include too low an absolute pressure as a result of flow losses in narrow cross-sections, filters, manifolds and chokes, excessive inlet head, or pressure losses resulting from excessive fluid viscosity. Cavitation can cause pump erosion, poor efficiency, pressure peaks, and excessive noise. It can also detrimentally affect the stability of choke controls and cause foaming in tanks if the fluid–air mixture is returned to the tank at atmospheric pressure [11.2, 11.4, 11.6, 11.47].

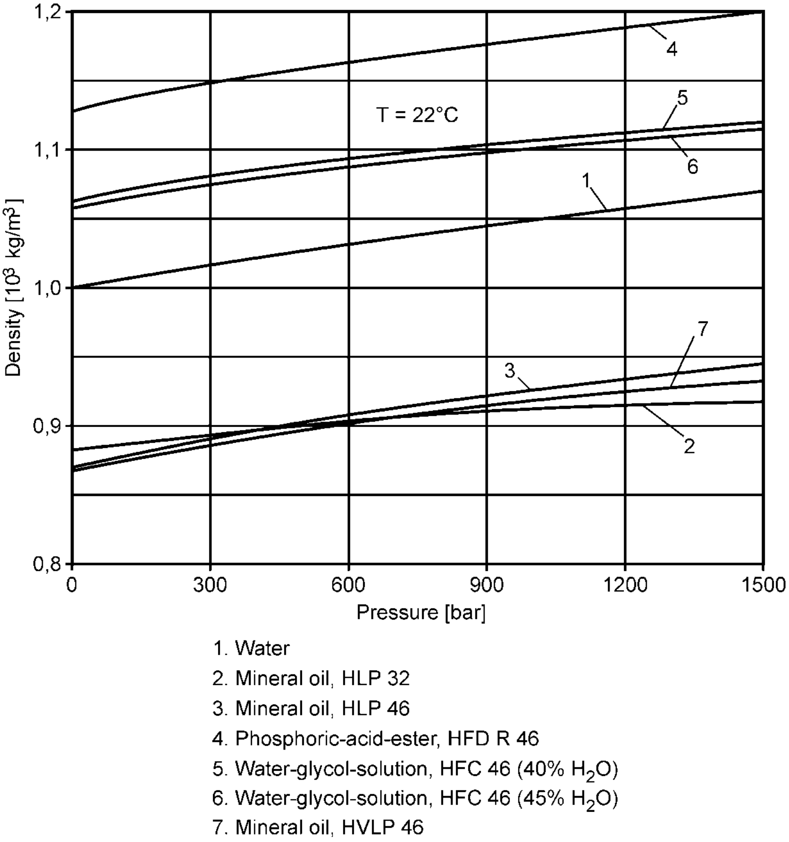


**Fig. 11.15** Pressure-dependence of the density for different hydraulic fluids.

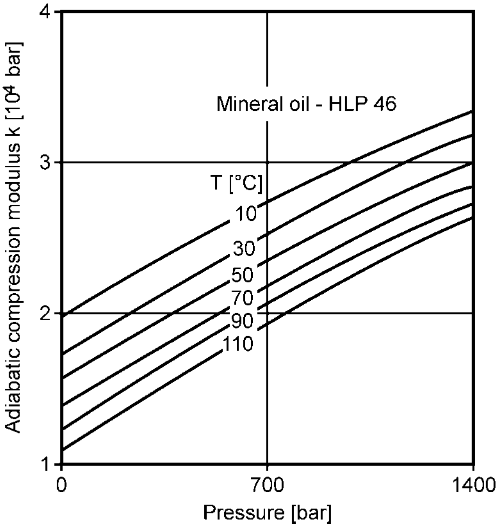


**Fig. 11.16** Adiabatic compression module of hydraulic oil.

#### 11.4.13.6 **Air Release**

The flow of hydraulic fluids back into tanks can drag air into the fluid. In addition, leaks in pipework at restrictions and partial vacuums can draw air bubbles into the system. Turbulence in the tank or local cavitation promote the formation of air bubbles in the fluid.

This air has to separate to the surface of the fluid or it will be drawn into the pump and this can cause other system or component damage. The speed at which the air bubbles rise to the surface depends on the diameter of the bubbles, the viscosity of the fluid, the density, and the quality of the base oils. The higher the quality and purity of the base oils, the faster is air release. Low viscosity oils usually release air more rapidly than high viscosity oils because the speed at which the bubbles rise.

$$C = (\rho_{FL} - \rho_L) \times X/\eta$$

where $\rho_{FL}$ is the density of the fluid, $\rho_L$ is the density of the air, $\eta$ is the dynamic viscosity and $X$ is a constant which depends on the density and viscosity of the fluid [11.2, 11.6].

Systems should be designed to hinder the ingress of air into the fluid and to assist the release of any trapped air bubbles. Critical areas include the tank, which should be fitted with baffles and deflectors, and the positioning of return pipes and circuits. Additives cannot positively influence air-release properties. Surface-active components (such as silicone-based defoamers) and contaminants (such as greases and corrosion preventives) detrimentally affect the air-release properties of hydraulic oils [11.5, 11.48, 11.49]. The air-release properties of mineral oils is generally better than that of fire-resistant fluids. The air-release properties of HPLD hydraulic fluid can be comparable with those of HLP hydraulic fluids.

Air-release properties are defined by DIN 51 381. The test involves air being blown into the oil. The air-release figure is the time the air (down to 0.2 % v/v) takes to escape from the oil at 50 °C under given conditions. The proportion of dispersed air is determined by measuring the density of the oil–air mixture [11.4] (see Chapter 18).

#### 11.4.13.7 **Foaming**

Surface foaming occurs when the air release speed is greater than the speed at which the bubbles at the surface of the fluid collapse, i.e. more bubbles are created than collapse. In the worst cases, this foam can be forced out of tank vents or openings or can be drawn into the pump. Additives, silicone-based or silicone-free defoamers can accelerate the collapse of bubbles by reducing the surface tension of the foam. They also negatively influence the air-release properties of the fluid, however, and this can cause compression and cavitation problems. Defoamers are thus used in very small concentrations (ca 0.001 %). The concentration of defoamer in a fluid can progressively decline as a result of aging and settling on metal surfaces and foaming problems often affect old, used fluids. Subsequent addition of defoamer should only be performed after consultation with the fluid manufacturer. Foaming behavior is defined by DIN 51 566. This involves defined air being passed through a porous stone which is submerged in the fluid. The volume of foam which gathers on the surface of the fluid is measured against time (at once, after 10 min) and at different temperatures (25 and 95 °C) [11.42]. Surface-active substances, detergent or dispersing additives, contamination in the form of grease, corrosion preventives, cleaners, cutting fluids, aging by-products, etc., can all have a negative affect on foaming behavior [11.5, 11.22, 11.48, 11.49] (see Chapter 18).

#### 11.4.13.8 Demulsification

Demulsification is the capacity of a hydraulic fluid to repel ingressed water. Water ingress can result from a leak in a heat exchanger, the formation of condensation water in tanks as a result of considerable changes of the oil level, poor filtering, water contamination through faulty seals, and extreme ambient conditions. Water in a hydraulic fluid can cause corrosion, cavitation in pumps, increases friction and wear, and accelerate the decay of elastomers and plastics. Free water should be removed from hydraulic fluid tanks as soon as possible by means of drain cocks. Contamination with water-miscible cutting fluids which is especially possible in machine tools can promote the formation of sticky residues after the water has evaporated. This can cause problems in pumps, valves, and cylinders. A hydraulic fluid should rapidly and completely repel ingressed water. Demulsification is defined by DIN 51 599 but this cannot be used for hydraulic oils which contain detergent/dispersing additives. Demulsification is the time taken to separate an oil–water mixture. The parameters are:

- viscosity up to 95 $mm^2\ s^{-1}$ at 40 °C; test temperature 54 °C;
- viscosity > 95 $mm^2\ s^{-1}$; test temperature 82 °C [11.42].

In detergent/dispersing (DD) hydraulic oils, fluid and solid contaminants and water are held in fine suspension and can be removed by use of suitable filter systems without the hydraulic function of machine and the fluid being negatively affected. DD hydraulic oils are thus often used in hydrostatic machine-tool equipment and in mobile hydraulics. Such machinery, for which circulation times are rapid, which need to be constantly available, and which are permanently subject to the danger of water and other contamination, is the primary area of application of detergent hydraulic fluids. Hydraulic fluids with demulsifying properties are often recommended for application in steel and rolling mills where large amounts of water are present and where low circulation times enable tank separation. Demulsifying properties in a modified form are used to determine the compatibility and demulsification of water-miscible cutting fluids with slideway oils and hydraulic oils. The aging of a hydraulic fluid negatively affects its demulsifying properties [11.5, 11.22, 11.48, 11.49] (see Chapter 18).

#### 11.4.13.9 Pour-point

The pour-point is the lowest temperature at which an oil still flows. A sample is systematically cooled and its flowing properties are tested every 3 °C [11.42]. The pour-point and the boundary viscosity define the lowest temperature at which an oil can normally be used (see Chapter 18).

#### 11.4.13.10 Copper Corrosion Behavior (Copper-Strip Test)

Copper or copper-containing materials are often used in hydraulic components. Materials such as brass, cast bronze or sintered bronze are found in the bearing elements, guides, or control units or sliding blocks of hydraulic pumps and motors. Copper pipes are used in cooling systems. Because copper corrosion can cause an entire hydraulic system to fail, the copper-strip test provides information about the corrosive effect of base fluids and additives on copper-containing materials.. A procedure for testing the corrosive effect of mineral oil-based and rapidly biodegradable hydraulic fluids on non-ferrous metals is known as the so called “Linde-Test” (A Screening Method for Biodegradable Oils with Respect to Copper-Alloy-Corrosion) [SAE Technical Paper 981516, April 1998] also known as VDMA 24570 [VDMA 24570 – Biologisch schnell abbaubare Druckflüssigkeiten – Einwirkung auf Legierungen aus Buntmetallen 03-1999] [11.11].

According to DIN 51 759, corrosion to a copper strip can take the form of discoloration or flaking. A ground copper strip is submerged in the fluid to be tested for a given time and at a given temperature. Hydraulic and lubricating oils are normally tested for three hours at 100 °C. The degrees of corrosion are: 1, slightly discolored; 2, moderately discolored; 3, heavily discolored; 4, corrosion (dark discoloration) [11.42] (see Chapter 18).

#### 11.4.13.11 Water Content (Karl Fischer method)

If water enters a hydraulic system, part might be so finely dispersed that it enters the oil phase and, depending on the density of the hydraulic oil, the water can also separate from the oil phase. This possibility must be considered when samples are being taken to determine water content.

Determining water content in mg $kg^{-1}$ (mass) with the Karl Fischer method involves the addition of a Karl Fischer solution then direct or indirect titration [11.42] (see Chapter 18).

#### 11.4.13.12 Aging Stability (Baader method)

This is an attempt to replicate the influence of temperature and oxygen, in the form of air, on hydraulic fluids under laboratory conditions. By increasing the temperature above practical usage levels, increasing oxygen levels, and including metal catalysts, an attempt is made to artificially accelerate the aging of hydraulic oils. The viscosity increase and the increase in the neutralization number (free acid) are recorded and evaluated. The laboratory test results are transferred to practical conditions. The Baader procedure is a practical means of testing the aging of hydraulic and lubricating oils.

For a given period of time, samples are aged at a predetermined temperature and in the presence of moving air while a copper coil which acts as an aging accelerator is periodically submerged in the oil. According to DIN 51 554-3, the aging stability of C, CL and CLP and hydraulic oils HL, HLP, HM is tested at 95 °C. The saponification number is given in mg KOH $g^{-1}$ [11.42] (see Chapter 18).

### 11.4.13.13 Aging Stability (TOST test)

The aging stability of steam turbine and hydraulic oils containing additives is determined in accordance with DIN 51 587

The TOST test has been used for many years to test mineral oil-based turbine and hydraulic oils. In a modified form (without water), the dry TOST test is used to determine the aging stability of ester-based hydraulic oils.

The aging of a lubricant is characterized by an increase in the neutralization number when the oil is exposed to oxygen, water, steel, and copper for a maximum of 1000 h at 95 °C (neutralization aging curve). The maximum permissible increase in neutralization number is 2 mg KOH after 1000 h [11.42] (see Chapter 18).

### 11.4.13.14 Neutralization Number

If the neutralization number of a hydraulic oil increases as a result of aging, overheating, or oxidation, the aging products formed can attack system components such as pumps and bearings. This, in turn, can cause serious system failures. The neutralization number is, therefore, an important criterion for evaluation of the condition of a hydraulic fluid [11.42].

The neutralization number indicates the amount of acidic or alkaline substances in a lubricant. Acids in mineral oils can attack materials. A high acid content, which can be the result of oxidation, is, therefore, undesirable [11.42] (see Chapter 18).

### 11.4.13.15 Steel/Ferrous Corrosion Protection Properties

The steel/ferrous corrosion protection-properties of steam turbine and hydraulic oils containing additives are determined in accordance with DIN 51 585.

Hydraulic fluids often contain dispersed, dissolved, or free water and a hydraulic fluid must provide corrosion protection to all wetted components under all operating conditions including contamination with water. This test examines the performance of the corrosion-protection additives under a number of different operating conditions.

The oil to be tested is mixed with distilled water (Method A) or artificial seawater (Method B) and stirred continuously for 24 h at 60 °C while a steel rod is submerged in the mixture. After 24 hours, the steel rod is examined for corrosion. The results enable assessment of the corrosion protection offered by the oil to steel components which are in contact with water or water vapor: degree of corrosion 0, no corrosion; degree of corrosion 1, little corrosion; degree of corrosion 2, moderate corrosion; degree of corrosion 3, heavy corrosion [11.42] (see Chapter 18).

### 11.4.13.16 Wear Protection (SHELL four-ball apparatus; VKA, DIN 51 350)

The Shell four-ball apparatus is used to measure the antiwear and EP properties of hydraulic fluids. The load-carrying capacity of hydraulic fluids is tested under boundary friction conditions. The procedure serves to determine values for lubricants with additives which withstand high pressures in boundary friction conditions between sliding surfaces. The lubricant is tested in a four-ball apparatus which consists of one (central) revolving ball and three stationary balls arranged as a cup. Under constant test conditions and for a predetermined duration, the contact scar on the three stationary balls is measured or the load on the revolving ball can be increased until it welds to the other three [11.42] (see Chapter 19).

### 11.4.13.17 Shear Stability of Polymer-containing Lubricants

Polymer-containing lubricants, high-molecular mass polymer molecules, are used as viscosity index improvers to improve the viscosity–temperature behavior of oils. As their molecular mass increases, these substances become increasingly sensitive to mechanical stress such as that which exists between a piston and its cylinder. Several tests are used to evaluate shear stability under different conditions [11.42]: DIN 51 350-6, Four-ball test; DIN 51 354-3, FZG test; and DIN 51 382, Diesel fuel injector method.

The drop in kinematic viscosity after shearing provides an indication of the permanent drop in viscosity which can be expected during operation (see Chapter 19).

The relative viscosity reduction due to shearing after 20 h according DIN 51350-6 (Determination of shear stability of lubricating oils containing polymers-tapered roller bearing) is implemented in DIN 51524-3 (2006); recommended shear loss below 15 %.

### 11.4.13.18 Mechanical Testing of Hydraulic Fluids in Rotary Vane Pumps (DIN 51 389-2)

The Vickers pump test and a variety of other manufacturers' pump tests realistically evaluate the performance of a hydraulic fluid. At present however, alternative tests (such as the DGMK 514 project, Mechanical Testing of Hydraulic Fluids) are being developed [11.50].

The Vickers test serves to determine wear protection in a rotary vane pump. The oil to be tested is circulated through a rotary vane pump at a given temperature and pressure (the test conditions are 140 bar, 250 h, variable temperature, operating fluid viscosity 13 $mm^2\ s^{-1}$). After completion of the test the ring and vanes are examined for wear (Vickers V-104 C10 or Vickers V-105 C10). The maximum permissible wear values are $< 120$ mg for the ring and $< 30$ mg for the vanes [11.42] (see Chapter 19).

### 11.4.13.19 Wear Protection (FZG Gear Rig Test; DIN 51 354-1 and -2)

Hydraulic fluids, particularly higher-viscosity grades, are used as hydraulic and lubricating oils in combined systems. Dynamic viscosity is the key wear-protection factor in hydrodynamic lubrication. At low sliding speeds or high pressures under boundary-friction conditions the wear protection offered by a fluid depends on the additives used (reactive layer formation). These boundary conditions are replicated by the FZG test.

The test is primarily used to determine the boundary performance of lubricants. Defined gear wheels turning at a defined speed are either splash- or spray-lubricated with an oil whose initial temperature is recorded. The tooth-flank load is increased in stages and the appearance of the tooth flanks is recorded. This is repeated until the final 12th load stage: load stage 10, Hertzian pressure at the pitch point 1.539 $N\ mm^{-2}$; load stage 11, Hertzian pressure at the pitch point 1.691 $N\ mm^{-2}$; and load stage 12, Hertzian pressure at the pitch point 1.841 $N\ mm^{-2}$. The starting temperature at load stage 4 is 90 °C, the peripheral speed is 8.3 $m\ s^{-1}$, the upper temperature is not defined; and gear geometry A is used.

The damage load stage as defined by DIN 51 524-2 is at least 10. ISO VG 46 hydraulic fluids which do not contain antiwear EP additives normally achieve load stage 6 (ca. 929 N $mm^{-2}$) [11.42] (see Chapter 19). Zinc-containing hydraulic fluids normally achieve damage load stage 10–11 at least. Zinc-free so-called ZAF hydraulic fluids achieve damage load stage 12 or greater.

## 11.5
## Hydraulic System Filters

Hydraulic oils are used for very many sensitive industrial manufacturing machines. Because of the use of these oils, hydraulic systems are reliable and are designed to run for years. The minimum technical requirements of hydraulic fluids according to DIN, ISO and manufacturers' specifications are clearly defined and are generally fulfilled by the fluids presently available on the market. These specifications do not, however, refer to 'good filterability' – requirements are not defined.

In the past most hydraulic and lubricating oil systems in machine tools, presses, stationary and mobile systems were fitted with 25 to 50-μm filters. This mesh size was adequate to satisfy the requirements of critical system elements such as valves. Critical hydraulic system components include those with narrow passages and low flow rates. Table 11.11 summarizes the typical gaps and passage sizes in a selection of hydraulic components [11.51–11.54].

If dirt and contaminants are present in the oil, these critical gaps can influence the function of the system, and wear rates. Experts differ on the size and amount of particles which constitute a critical situation.

### 11.5.1
### Contaminants in Hydraulic Fluids

There are several types and causes of hydraulic fluid contamination. The first major differentiation is between primary and secondary contamination. Primary contamination is that which existed in the hydraulic circuit before it was commissioned. This can include machining residues, assembly residues, and fresh-oil contaminants. The secondary variety is that formed after the system began to operate, e.g. mechanically abraded material, flow-related abrasion, corrosion, wear and dirt which enters the system via cylinder seal materials or via tank de-aerating units. [11.6, 11.51–11.54].

After comprehensive trials by a leading manufacturer into the effect of contaminants on the life of roller bearings great value is now placed on the cleanliness and filtration of oils. Purity and the types of additives used have a significant influence on the life and likelihood of failure of roller bearings and thus a whole system [11.52, 11.53, 11.55]. Trials conducted within the framework of the FVA 179/1 research project 'Influence of Foreign Particles on Roller Bearings and Measures to Avoid Them' also investigated this subject. The causes of premature roller bearing failures are, above all, inadequate lubrication, particle contamination, and overloading.

**Tab. 11.11** Hydraulic component clearance.

| *Component* | *Typical critical component clearance (μm)* |
|---|---|
| Gear pumps (under pressure) | |
| Gear to side plate | 0.5–5 |
| Gear tip to housing | 0.5–5 |
| Vane pumps | |
| Vane tip to stator | 0.5–5 (1) |
| Vane to side plate | 5.0–13 |
| Piston pumps | |
| Piston to cylinder | 5.0–40 |
| Cylinder to valve plate | 1.5 (0.5)–10 (5) |
| Servo valves | |
| Jets | 130.0–450 |
| Splash care | 18.0–63 |
| Piston valve (radial) | 2.5–8 |
| Control valves | |
| Jets | 130.0–10 000 |
| Piston valve (radial) | 2.5–23 |
| Dish valve | 1.5–5 |
| Plug valve | 13.0–40 |
| ***Component*** | ***Film thickness*** |
| Roller bearings | 0.1–1.0 μm |
| Hydrostatic slide bearings | 0.5–100.0 μm |
| Hydrodynamic slide bearings | 1.0–25.0 μm |
| Toothed wheels | 0.1–1.0 μm |
| Seals | 0.05–0.5 μm |

### 11.5.2
### Oil Cleanliness Grades

Several methods can be used to classify oil cleanliness. The best known are ISO 4406 and NAS 1638. Determining oil cleanliness according to ISO 4406 involves examining the number and size of particles in a 100-mL sample of fluid. The number of particles in the categories > 2 μm, > 5 μm, and > 15 μm are recorded. Normally only particles > 5 μm and > 15 μm are reported (old commonly used practice). The new specification ISO 4406 (December 1999) defines the particles in the categories > 4 μm, > 6 μm, and > 14 μm. The particles can be counted with a microscope or by suitable automatic particle counters. ISO 4406 or NAS 1638 defines the maximum permissible contamination according to the type of hydraulic system, how sensitive it is, and which critical components form part of the system. Depending on the operating conditions, the cleanliness categories in Table 11.12 are recommended [11.6, 11.17, 11.22, 11.55].

**Tab. 11.12** Cleanliness categories

| *Type of system / case of application / filter size* | *Cleanliness category in accordance with ISO 4406* | *Cleanliness category in accordance with NAS 1638* |
|---|---|---|
| Against fine soiling and mudding-up of sensitive systems; servo hydraulics | min. 13/10 | 3–4 |
| Heavy duty servo systems, high-pressure systems with long service life | min. 15/11 | 4–6 |
| Proportional valves, industrial hydraulics with high operating safety | min. 16/13 | 7–8 |
| Mobile hydraulics, common mechanical engineering, medium pressure systems | min. 18/14 | 8–10 |
| Heavy industry, low pressure systems, mobile hydraulics | min. 19/15 | 9–11 |

### 11.5.3
### Filtration

Filters intended to remove solid impurities from lubricants have been fitted to hydraulic systems for decades. The filters used are:

- tank vent filters to clean any drawn-in air;
- pressure filters to clean the fluid entering the pump;
- top-up filters which filter the hydraulic fluid as it is being fed into the tank;
- by-pass filters in the tank circuit to improve the cleanliness levels; and
- return filters fitted to fluid return lines.

The filters can be of the cartridge or surface variety. Important data are the mesh and retention size of the filter (the designation $\beta 3 > 200$ describes a filter of 3-μm mesh size and a separation rate of 200, i.e. only one particle of 200 particles will pass the filter). In addition, the initial pressure difference ($\Delta P$ max. 0.1–0.2 bar) and the maximum output pressure difference ($\Delta P$ max. 3–5 bar) in relation to the flow rate, viscosity, and density are of importance. The primary filter materials are microfiberglass, metal meshes, cellulose paper, and some other constructions. Hydraulic filters consist of an element, a housing, a contamination indicator, and other components. In general the fluid flows from the outside to the inside. The selection of mesh size is a matter of experience and depends on the specific requirements of critical components. As a rule, hydraulic systems use filter mesh sizes ranging from 3 to 40 μm [11.55, 11.56]. When using filters with micron ratings of e.g., 1 μm, 3 μm, and 6 μm, attention has to be taken. Especially high molecular components of the fluids (e.g. *VI*-improvers) and contaminations (e.g. grease, corrosion preventives) can block the filters. Filter blockage can occur if additiv systems are incompatible (e.g. mix of incompatible zinc-containing and zinc-free additives).

### 11.5.4
### Requirements of Hydraulic Fluids

High-performance filtering systems make high demands on the filterability of hydraulic fluids. A hydraulic fluid should only generate a small pressure difference across the filter after long-term use. Base oils and additives should be easily filterable with filter mesh sizes of 1, 3, 6, and 10 μm. Nothing in the fresh fluid should cause the filter to block and thus reduce its life (this is examined by special laboratory tests). Naturally, the purity of the fresh fluid should be low.

According to ISO 4406 the cleanliness of drums should be 17/14 (19/15) and experience shows that the cleanliness of road tankers should be 15/12 (18/14), although transport, storage, and environmental influences generally cause the cleanliness factor to deteriorate by 2 to 3 categories. In practice poor filter life often results from contamination of the hydraulic fluid with water, dirt, and other fluids, from inadequate maintenance of the system, or from incorrect filter selection. Determining the exact cause normally requires expensive laboratory tests [11.55, 11.56].

## 11.6
## Machine Tool Lubrication

### 11.6.1
### The Role of Machine Tools

Machine tools are the most important machines in the metalworking industry. With a share of approximately 20 %, Germany is one of the world's leading manufacturers of machine tools. In terms of sales, Germany (DM 14 billion) is second to Japan (ca DM 16 billion) but ahead of the USA (DM 9 billion), Italy (ca DM 6 billion) and Switzerland (ca 4 billion) [11.57]. Machine tools are used for a wide variety of operations including forming, cutting and bending; they are principally for turning, milling, drilling, grinding and machining center. They can combine any of these in a transfer system [11.58]. Machine-tool construction is a major sector in engineering and their share of overall exports for Germany (about 60–70 %) and Japan illustrate the importance of machine tools in national economies [11.59].

### 11.6.2
### Machine Tool Lubrication

This section covers lubricating oils, hydraulic fluids, and greases for machine tools. Apart from cutting fluids, hydraulic oils are volumetrically the most significant group of machine lubricants, followed by slideway oils and gear oils. Neat or water-miscible cutting fluids or metalworking fluids are covered in Chapters 14 and 15.

The lubrication of machine tools is described in DIN 8659-1 and –2 and ISO 5169 and ISO 3498. These standards contain requirements which should be observed when manufacturers and users establishing lube plans. These also satisfy the requirements specified in DIN/ISO 5170 (machine tool lubrication systems) [11.60].

Lubrication plans should cover all the components in a machine tool which need lubrication. These should describe:

- the precise location of all lubrication points;
- the type of lubrication required;
- the lubricant itself according to DIN 8659-1 and –2 and ISO 3498 and the tank volume; and
- the lubrication timetable.

The purpose of a lubricating plan as part of routine servicing is to ensure that a sufficient quantity of the correct lubricant is applied to the right point at the right time (VDI Guideline 3009). Machine manufacturers normally supply lubricant recommendation tables with machines. These list the type of lubricant according to DIN 51 502, ISO 6743, and ISO 3498 for each viscosity grade by its brand name. On the basis of this information, a maintenance plan is created for every machine which shows the type of lubricant and the lubrication interval. For most machines, a maintenance plan and a lubrication chart are included in the service handbook.

Figure 11.17 shows an example of a lubrication plan for a centerless grinding machine. This shows lubricants conforming to DIN 51 502 and ISO 3498, lubrication intervals, tank volume, and the location of all lube points.

Lubricant recommendations should be updated every two years to make use of new lube developments. Technically similar lubricants can often be grouped to enable some lubricant rationalization [11.61].

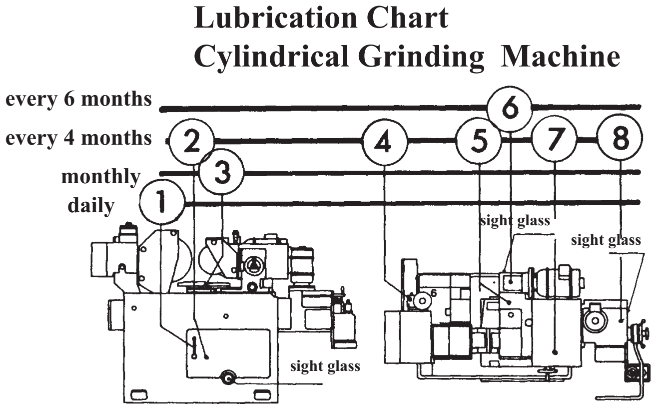
Machine manufacturers often refer to the lubricant recommendations issued by component manufacturers. The recommendations issued by the manufacturers of hydraulic components, gearboxes, slideways, and linear guides must be observed.

For lubrication a machine tool can be divided into a number of major elements: hydraulic unit, gearbox, spindle, slideway, linear system, plain and roller bearings and finally, cutting zone lubrication. In general, a different lubricant is recommended for every component, i.e. at least seven different types and viscosities of lubricant (excluding the cutting fluid) are required.

## 11.6.3
## Machine Tool Components – Lubricants

### 11.6.3.1 Hydraulic Unit

Most hydraulic equipment is designed to use HLP (HM), HLPD (HG) fluids with an ISO viscosity between 32 and 46. Running temperatures range from 40 to 60 °C and peak temperatures of 60 to 80 °C can occur [11.58, 11.59]. Although operating pressures range from 50 to 100 bar (relatively low), pressures up to 400 bar are used in clamping fixtures. Generally low system pressures are used to avoid chatter marks (compressibility of the fluid) which often occur at higher pressures. Moreover, higher pressures lead to more leakage and thus lower overall efficiency [11.58]. Figure 11.18 shows a list of hydraulic oils used in machine tools (survey of 12 Ger-



| | a | b | b | c | d | e | f | g | h |
|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2* | 2** | 3 | 4 | 5 | 6 | 7 | 8 |
| **Lubricant**<br>DIN 51502<br>ISO 3498 | CGLP 68<br>GCC 68 | CLP 10<br>CC 10 | CLP 5<br>CC 5 | K2K<br>XM2 | CGLP 68<br>GCC 68 | HLP 32<br>HM 32 | CLP 150<br>CC 150 | CGLP 68<br>GCC 68 | CGLP 68<br>GCC 68 |
| **Tank Volume**<br>ℓ | 2,7 | 20 | 20 | 0,1 | 1 | 4 | 1 | 1 | 1 |

- **a - centralized lubricating system**
- **b - grinding wheel spindle bearing**
- **c - slideway ( table )**
- **d - slideway ( dressing tool )**
- **e - spindle bearing**
- **f - worm gear**
- **g - slideway ( dressing tool )**
- **h - slideway ( grinding tool )**
- **2* - speed 30-45 m/s**
- **2 ** - speed 45-60 m/s**

**Fig. 11.17** Lubrication chart of a machine tool.

man machine tool manufacturers, 1995) [11.59]. HLPD fluids are often used to solve friction and compatibility problems.

Rotary vane and internal gear pumps are used at pressures between 50 and 100 bar. Higher pressures are generated by radial and axial piston pumps. External gear pumps are seldom used because of the noise they generate. Figure 11.19 shows the types of pump used in machine tools (survey of 12 German machine tool manufacturers, 1995) [11.59].

Figure 11.20 shows the pressures used in machine tools.

Actuator valves, sleeve valves, shut-off valves, and throttle valves are used in machine tools. Many valves have hydrodynamic bearings which make them sensitive to stick–slip effects, contamination, and deposits [11.58–11.62].

Machine tool hydraulic systems are normally equipped with mesh or fiber filters. Approximately 80 % of machine tool manufacturers use filters in the 5 to 10 μm range; the remaining 20 % use filters up to 25 μm [11.59].

Depending on the type of valves used, the pressure, and the importance of the machine, the ISO 4406 cleanliness of the fluids should be between 15/11 and 17/13 or lower according to ISO 4406 [11.28, 11.55, 11.59].

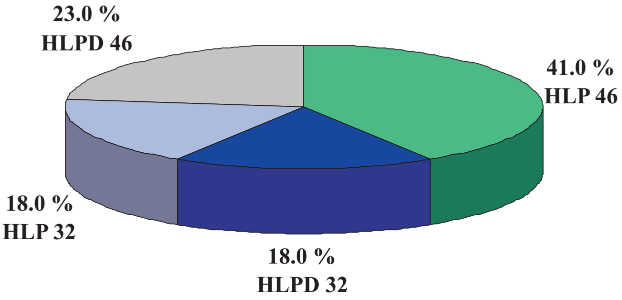


**Fig. 11.18** Hydraulic oils used in machine tools.

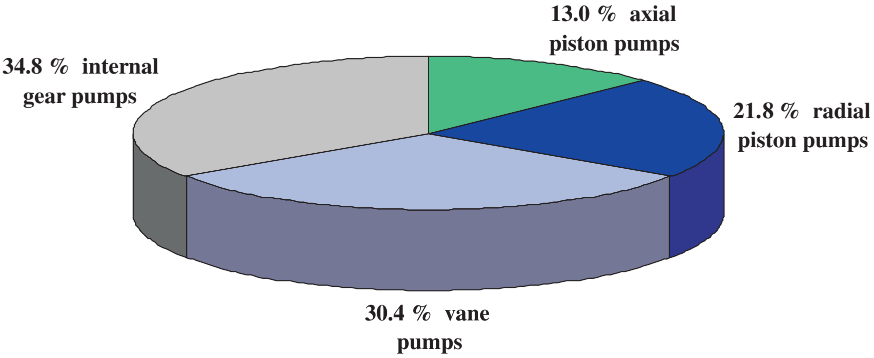


**Fig. 11.19** Hydraulic pumps used in machine tools.

**Pressure range**

80 % of all machine tools are working in a pressure range between 50 and 100 bar
13 % greater than 100 bar
7 % greater than 150 bar

**Minimum viscosity of hydraulic fluids**

**Vane pumps:**
Normally, a viscosity of min. 15 mm$^2$/s at pressures up to 100 bar is required - low viscosity fluids are currently being developed

**Piston pumps:**
Today, they are available for low viscosity fluids - but expensive

**Gear pumps:**
need a lot of assembly volume

**Fig. 11.20** Working conditions used in machine tools.

#### 11.6.3.2 Slideways

Machine tool slideways which guide supports and workpieces are among the most important elements of a machine tool. The special demands made on these slideways include precision, high performance, low manufacturing costs, and low operating costs. The most important features of slideways are:

- low friction, no stick–slip at low feeds and high load-carrying capacity;
- low wear and ultimate reliability against seizures;
- torsional stiffness and minimal play; and
- good damping properties to reduce chatter marks on machined surfaces.

In general, hydrodynamic, hydrostatic, and roller guides are used. Aerostatic and electromagnetic guides are seldom found in machine tools. Hydrostatic guides are losing popularity because of their price but can still be found on many machines. These days, hydrodynamic and linear roller guides (linear systems) are often used. Hydrodynamic slideways are losing market share because they only enable relatively small feed velocities (maximum 0.5 m $s^{-1}$), often suffer from stick–slip, and are more expensive to manufacture than linear roller guides. The most common material pairings used in hydrodynamic slideways are cast iron–cast iron, cast iron–plastic, cast iron–steel, and steel–plastic. Slideway oils should conform to DIN 51 502, ISO 6743-13, and ISO 3498 [11.63]. Horizontal slideways are often lubricated with CGLP 68, HG 68 or G 68 slideway oils. Inclined or vertical slideways are lubricated with CGLP 220, HG 220 or G 220 oils. The oil is applied through central systems and is lost after use. Slideway oils are general lubricating oils with additives to improve oxidation and corrosion protection. They also contain anti-wear agents, EP additives, surface-active substances and often adhesion improvers (tackifyers).

In recent years, roller or linear guides have been fitted increasingly to machine tools. In 1995, nine of twelve German manufacturers surveyed used roller linear guides exclusively and four used hydrodynamic and roller guides. The lubricants used should separate the moving parts in the roller in the contact zone which counter-rotate. The lubricant should also have damping characteristics in the contact zone (especially when the direction of movement changes) and reliably protect against wear and seizures. The lubricant should also form a stable and effective film in a very short time. Such total-loss lubricants are supplied to the linear guide zones via a central system. CGLP 68 and CGLP 220 grades are often used [11.60]. High-viscosity CGLP 220 slideway oils which contain surface-active components are often recommended. Alternatively, K2K or similar greases can also be used (see Chapter 16).

Oils for hydrodynamic slideways and linear guides should have the following properties [11.58, 11.59]:

- chemical compatibility with all cutting fluids used;
- good demulsification of emulsions, no sticky residues on slideways;
- low coefficient of friction (static and dynamic);
- avoidance of stick–slip (sliding and static friction alternates during stick–slip on slideways, which can cause chatter marks);
- good pumpability in central lubrication systems;

- good adhesion to slideways with tacky additives and/or without tacky additives;
- good wear protection (EP and AW additives) FZG ≥ 12;
- good slideway material compatibility;
- good corrosion protection (no black stains on slideways);
- same additive systems as hydraulic oils (i.e. zinc-, ash- and silicone oil-free); and
- meet the specifications of hydraulic oil if hydraulic and slideway oils share a circuit.

#### 11.6.3.3 **Spindles (Main and Working Spindles)**

The function of spindles is to guide the tool and/or workpiece at the cutting zone. In addition, spindles should absorb external forces. The accuracy and surface quality of components made on machine tools depends on the static, dynamic, and thermal behavior of the spindle bearings. These are key elements of machine tools. Tool spindles can be supported on greased roller bearings, oil-lubricated roller bearings, or hydrodynamic plain bearings. Roller bearings have almost completely replaced plain bearings. Oil-lubricated roller bearings are normally fed total-loss oil from a central system or via an oil-mist system. Often, low-viscosity CL/CLP general lubricating oils according to DIN 51 517 or ISO VG 5–22 FC and FD spindle oils according to ISO 6743-2 are used. Spindle oils must lubricate and cool. They have to protect against steel and copper corrosion, and be oxidation-stable. Depending on the application, lubricants with AW/EP additives are used. The spindle speed, defined as the product of rpm ($\text{min}^{-1}$) × average bearing diameter (mm) determines whether a spindle should be lubricated with oil or with grease [11.31, 11.58, 11.64].

#### 11.6.3.4 **Gearboxes and Bearings**

Gearboxes are designed to convert and transfer movement and forces – they are units which transmit energy. Gearboxes in machine tools serve to reduce drive speed to the feed velocity of supports, etc. The gearboxes can have fixed or selectable ratios. Speed adjustments are often made with synchronized or non-synchronized motors. The different gearboxes include spur, worm, crown-wheel and pinion or planetary types [11.58]. The stress on machine tool gearboxes is relatively small and ISO VG 68 to 320 CLP (DIN 51 517 – dated January 2004), CKC or CKD (ISO 6743/6) gear and general lubricating oils are often used. Worm drives are often lubricated with polyglycol-based CLP PG or CKE gear oils. Synthetic, polyalphaolefin-based CLP HC or CKT oils are used for thermally stressed gearboxes [11.31, 11.58, 11.60].

The bearings most often found in gearboxes are plain and roller bearings, although plain bearings are seldom used in machine tools. The most popular types are ball and cylindrical roller bearings. The corresponding lubricants are general lubricating oils or specific gear oils (see Chapter 10).

## 11.6.4
## Machine Tool Lubrication Problems

Many different oils and greases are used in a machine tool. Hydraulic, gear, slideway, and spindle oils, and greases, are the most important groups. An important point is the required compatibility of the lubricants with each other. Leaks can cause large amounts of hydraulic oils to contaminate cutting fluids (every year 3 to 4 times the volume of the hydraulic system enters the cutting fluid circuit [11.59]). At the same time, metalworking fluids can enter the hydraulic oil circuit via cylinders, etc. Slideway oils as total loss lubricants and metalworking fluids are in close contact and must, therefore, be compatible. Developments in the area of universal oils (chemically related lubricants) which eliminate the problem of poor compatibility are currently a high priority. Initial developments of fluid families, i.e. hydraulic, gear, slideway, spindle, and metalworking oils which share common additive packages but which are available in different viscosities have already been tested in practice [11.59, 11.65]. Low-viscosity neat oils have already overtaken water-miscible emulsions (the trend is oil instead of emulsion) [11.5, 11.65]. Future developments will concentrate on Unifluid systems which consist of one low viscosity oil which serves as cutting fluid, slideway oil, and hydraulic fluid. This concept, however, requires the redesign of components such as pumps to handle such low-viscosity fluids. The development of compatible systems could save large amounts of money now spent on the expensive monitoring and maintenance of currently-used water-based cutting emulsions (see Chapter 14).

## 11.6.5
## Hydraulic Fluids – New Trends, New Developments

### 11.6.5.1 Applications

In Germany, 80 to 85 % of hydraulic fluids are mineral oil-based. Of these approximately 40 % are used in mobile hydraulics and 60 % in industrial systems. Approximately 7 % of the hydraulic fluids used are fire-resistant fluids used in underground mining, steel mills, foundries, and power stations. Approximately 7 % of total hydraulic fluids are biodegradable hydraulic fluids for mobile and stationary equipment. Approximately 1 % are formulated for special uses, for example in the food and beverage industries.

### 11.6.5.2 Chemistry

#### Base Fluids

Typical hydraulic fluids are composed of 95–98 % base fluids and 2–5 % additives. As already mentioned above, the largest contributor to base fluids are mineral oils refined from crude oil (mainly paraffinic and naphthenic compounds and hydro cracked base oils). Other base fluids used are, basically, fully and partially saturated esters, polyglycols, polyalphaolefins (PAO), and alkylates.

### Additives

The most important additives for hydraulic fluids are listed in Tab. 11.13

**Tab. 11.13** Most important types of additive in hydraulic fluids, and their chemistry.

| *Type* | *Chemistry* |
|---|---|
| Antioxidants (AO) | Phenolic and aminic AO, zinc dialkyldithiophosphate (ZnDTP) |
| Copper deactivators | Nitrogen compounds (triazoles), dimercaptothiadiazoles |
| Steel/iron corrosion inhibitors | Carboxylic acid derivatives, sulfonates, succinic acid compounds |
| Anti-wear additives (AW) | Esters, ZnDTP |
| Extreme pressure additives (EP) | Phosphorus and sulfurous compounds, thiophosphates, sulfurized hydrocarbons (active and inactive) |
| Friction modifiers | Fatty acids, polar compounds, esters |
| Detergents/dispersants | Ca and Mg phosphates, sulfonates, phenates, polyisobutylensuccinimide |
| Antifoam agents | Silicone oil, silicone oil-free, polymethylsiloxane |
| Viscosity index improvers (VII) | Polymethacrylates |
| Pour-point depressants (PP) | Polymethacrylates |
| Dyes | Azo dyes, fluorescent dyes |
| Tackifiers | Polyisobutylenes, polar compounds |

Approximately 70 % of mineral oil-based hydraulic fluids in Europe are zinc-containing fluids, although an increasing number of zinc-free hydraulic fluids are becoming available. These fluids are formulated without zinc dialkyldithiophosphate (ZnDTP) as multipurpose additive. Although this has a large affect on the performance of hydraulic oils, it is important to state that the element content itself has no decisive effect on the quality of hydraulic fluids. Zinc-free hydraulic fluids usually contain similar or even lower amounts of phosphorus and sulfur than Zn-containing fluids (Tab. 11.14). Traditional Group I mineral base oils have an incorporated sulfur content that contributes positively to anti-wear performance and synergistic functions, e.g. as a radical scavenger for oxidation stability. Additionally a certain aromatic content that is typical for Group I base oils improves solubility of additives and ageing products. New hydraulic fluids using modern base oils (hydrotreated, hydro-cracked or PAO) have a much lower or zero sulfur and aromatic content and therefore need consequently higher additive treat rates to balance the shortfall of base fluid related sulfur and aromatic content.

**Tab. 11.14** Variation of element content of zinc-containing and zinc-free hydraulic fluids.

| *Element* | *Zinc-containing hydraulic fluids* | *Zinc-free hydraulic fluids* |
|---|---|---|
| Zinc | 200–500 ppm | 0 ppm |
| Phosphorus | 200–500 ppm | 60–500 ppm |
| Sulfur | 400–1000 ppm | 0–1000 ppm |
| Sulfur (base oil) | 300–1000 ppm | 300–1000 ppm |

Selected characteristics of hydraulic fluids will be discussed in subsequent sections. The use of Zn-free or Zn-containing additives usually makes a remarkable difference to characteristics and performance and can also crucially affect whether a hydraulic fluid is formulated with demulsifying or detergent/dispersant characteristics.

#### 11.6.5.3 **Extreme Pressure and Anti-wear Properties**

Although the Vickers vane pump test is a traditional and important test for hydraulic fluids, the test results obtained are not sufficiently differentiating for state-of-the-art hydraulic fluids. Other commonly used EP/AW test methods include use of the Shell four ball tester (DIN 51 530-1,2), use of the Brugger test machine (DIN 51 347-2), and use of the FZG test rig (DIN 51 354-2). Tests on hydraulic fluids containing different additives (all ISO VG 46) have been conducted with these methods. The results showed that Zn-free hydraulic oils of the detergent/dispersant type (DD) usually have better EP performance than traditional Zn-containing demulsifying hydraulic oils and other hydraulic oils (Tab. 11.15).

A relatively new test rig for hydraulic fluids is the so-called FE 8 of FAG for the determination of roller bearing wear and friction coefficients. Although this procedure is normally used for greases and gear oils (DIN 51 819-3), some OEMs request a “pass” for their in-house approval of hydraulic oils. This test sequence is passed mainly by Zn-free formulated hydraulic fluids. One can, in general, state that phosphorus/sulfur chemistry without Zn is more suitable for high-EP requirements (e.g. hydraulic systems with incorporated gear drives). Fluids formulated with Zn-based additives perform well in the mixed friction applications where only medium EP but good AW is required. Their multifunctional anti-wear performance is therefore usually better that of than Zn and ash free fluids.

#### 11.6.5.4 **Detergent/Dispersant Properties**

Some OEMs set minimum limits for the dirt-carrying properties of hydraulic oils. A Fuchs in-house test reported by DaimlerChrysler [11.66] entails use of a paper chromatographic method in which a colloidal graphite dispersion is applied to a paper strip that is dipped in the lubricant. Migration of the graphite under specified conditions is reported. The in-house limit of the OEM stipulates a minimum migration distance of 40 mm. Only hydraulic fluids with selected dispersant additives and elevated treat rates pass this test (Fig. 11.21).

**Tab. 11.15** Typical EP/AW test results for different types of hydraulic fluid.

| *Test method* | *Conventional EP/AW* | *Medium EP/AW* | *High EP/AW* |
|---|---|---|---|
| Four-ball EP performance | 1800–2000 N<br>• Zn-containing, DD<br>• Zn-free, demulsifying<br>• Zn-containing, demulsifying | | > 2200 N<br>• Zn-free, DD |
| Brugger EP performance | 20–25 N $\text{mm}^{-2}$<br>• Zn-containing, demulsifying<br>• Zn-containing, DD, synthetic base oil<br>• Zn-free, demulsifying | 30–40 N $\text{mm}^{-2}$<br>• Zn-containing, DD<br>• Zn-free, demulsifying | > 50 N $\text{mm}^{-2}$<br>• Zn-free, DD |
| FZG EP performance | 11–12<br>• Zn-containing, DD<br>• Zn-containing, DD, synthetic base oil<br>• Zn-free, synthetic base oil, demulsifying | >12<br>• Zn-free, DD<br>• Zn-containing, DD | |
| Vickers EP/AW performance | < 120 mg ring<br>< 30 mg vane<br>• all pass (generally) | | |

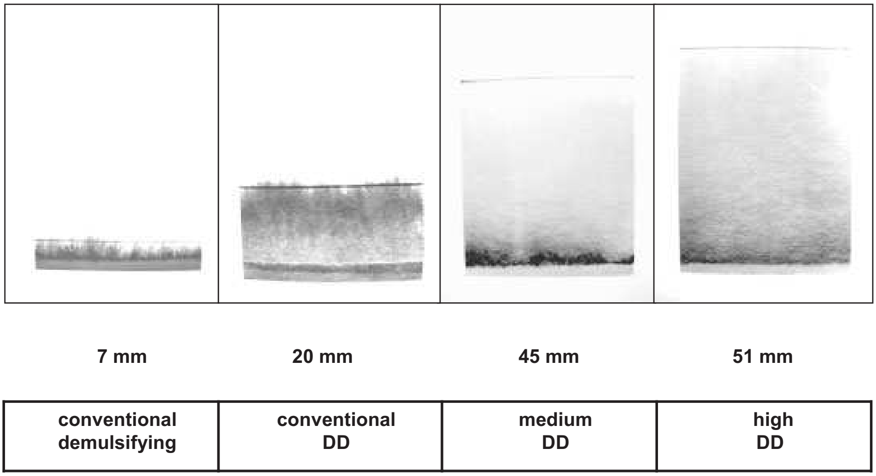


**Fig. 11.21** Detergent/dispersant properties: test results for different types of hydraulic fluid.

### 11.6.5.5 Air Release

It seems logical that hydraulic fluids with demulsifying properties would have much better air-release behavior than fluids with detergent/dispersant characteristics. Despite this, a careful DD formulation with state-of-the-art additive technology can have air-release characteristics similar to or better than those of demulsifying hydraulic fluids. This has been proven using DIN 51 381. All values were less than 10 min and therefore fulfilled DIN 51 524-2, which describes HLP hydraulic oils with limits increased from 10 to 13 min.

#### 11.6.5.6 **Static Coefficient of Friction**

A series of tests using an inclination tribometer have been performed at SKC Gleittechnik in Coburg (Germany). The SKC test procedure was originally developed for slide-way oils and determines the coefficients of friction of the fluids for a combination of plastic and steel. Static coefficients of friction for detergent/dispersant hydraulic fluids were between 0.130 and 0.177, lower than those of demulsifying fluids (0.214). Excellent values of 0.088 to 0.11 where achieved with synthetic esters, because of to their polar characteristics.

#### 11.6.5.7 **Oxidation Stability**

The additives and base oil selected have a substantial effect on oxidation stability. Both hydrolytic and thermal stability are increased by changing from demulsifying to DD formulations. The type and treat rate of DD formulations effect the results as does the type of base oil. Results from the TOST test method according ASTM 943 (DIN 51 587) are shown in Tab. 11.16. Choosing the proper hydraulic fluid can lead to a severalfold increase in fluid lifetime.

**Tab. 11.16** Examples of TOST test results.

| *Hydraulic fluid* | *TAN > 2 mg KOH g$^{-1}$* |
|---|---|
| Minimum test results to fulfill DIN 51524 | 1000 h |
| Mineral oil, demulsifying, low treat rate | 1200 h |
| Mineral oil, DD, medium treat rate | 2300 h |
| Mineral oil, DD, high treat rate | 3900 h |
| Hydro cracked base oil, DD, high treat rate | 4500 h |

#### 11.6.5.8 **Shear Stability**

Improved viscosity–temperature behavior is desirable when hydraulic equipment operates over a wide temperature range. The viscosity index VI is traditionally increased by addition of VI improvers, which are usually polymers. These polymers are, unfortunately, eventually sheared, resulting in a decrease in viscosity. This behavior is now considered in the revised version of DIN 51 524-3, which describes high-VI hydraulic oils (HVLP). Taper roller bearing shear-stability test DIN 51 350-6 is also being introduced. Some OEMs require a maximum shear loss of 15–20 % in this test. Because the viscosity of conventional HVLP oils based on standard VI improvers drops by 30–50 % at 100 °C, conventional HVLP oils must be reformulated by using different additive chemistry, which increases the cost. An alternative method of formulation is to use base oils which already have in-built natural, shear-stable, high VI. Test results can be found in Tab. 11.17.

**Tab. 11.17** Test results of high-VI hydraulic oils: Tapered roller bearing shear-stability test DIN 51 350-6 (20 h, 60 °C, 40 mL)

| | *PAO HVLP 46* | *HEES saturated HVLP 46* | *Mineral oil, conventional HVLP 46* | *Hydro cracked HVLP 68* |
|---|---|---|---|---|
| $V_{-20}$ | 2200 | 1970 | 1700 | 5900 |
| $V_{40}$ | 46 | 46 | 46 | 68 |
| $V_{100}$ | 7.8 | 8.2 | 8.9 | 10.7 |
| $VI$ | 140 | 155 | 180 | 150 |
| Shear loss, $\Delta V_{100}$ | < 1% | < 1% | < 40–50% | < 10% |

In many stationary hydraulic applications multi-grade engine oils are used instead of regular hydraulic fluids. SAE 10W40 and SAE 15W40 are very common. These products have viscosities >95 cSt at 40 °C and are formulated with polymers subject to high shear losses that result in critical decreases in viscosity.

#### 11.6.5.9 **Filtration of Zn and Ash-free Hydraulic Fluids**

The filterability of Zn-free hydraulic oils depends very much on the type of chemistry used. A special multi-pass test rig has been designed for dynamic testing. Different pressures in the presence of 1 % water (for better differentiation) are recorded over time. Depending on the formulation strategy extremely different filtration behavior can be observed. Under the practical conditions used with this test rig long-lasting and filterable hydraulic fluids have been developed. Test results for a variety of Zn and ash-free hydraulic fluid formulations are shown in Fig. 11.22. In practice, the compatibility of Zn-containing and Zn-free hydraulic fluids should usually be tested before they are mixed.

#### 11.6.5.10 **Electrostatic Charges**

Electrostatic phenomena usually depend on the fluids used and on the filter materials used for deep filtration. Because electrostatic charges in fluids depend mainly on the conductivity of the fluids, the risk potential of electrostatic charges in hydraulic fluids depends very much on the additives used (Tab. 11.18). Metal-containing fluids with zinc, calcium, or magnesium compounds as additives are believed to be free from problems, because their conductivity is >300 pS $m^{-1}$. The additives conduct electrostatic charges to the equipment housing. Although Zn-free and metal-free hydraulic fluids behave differently and do not conduct electrostatic charges, proper selection of Zn-free additives and advanced treatment procedures enable adequate conductivity to be achieved. Needless to say, for pure HL oils without any EP/AW additives conductivity is very low and must therefore be regarded as critical. A VDMA working group is actively investigating this phenomenon [11.67].

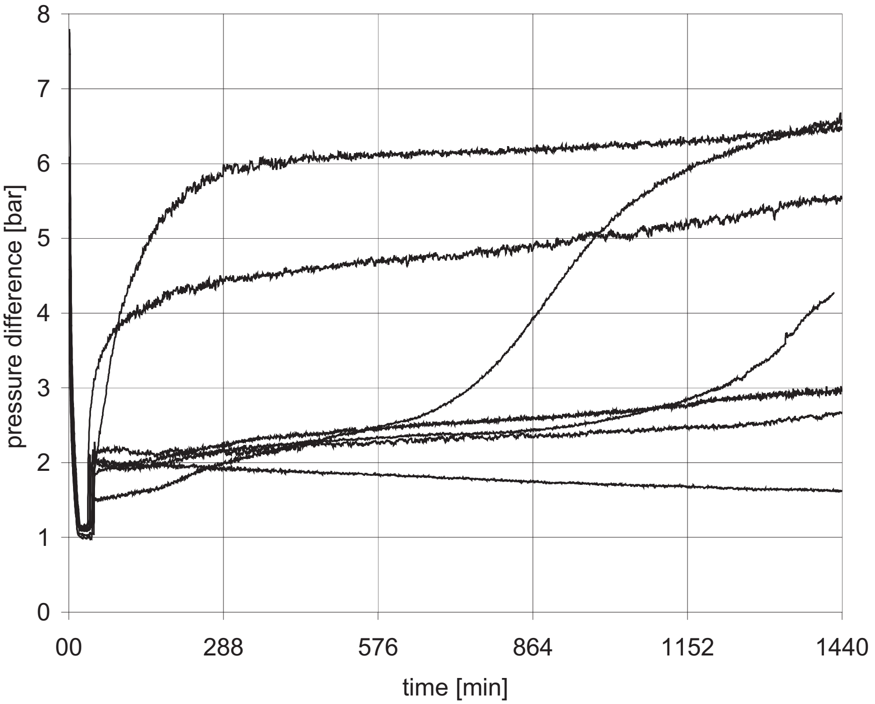


**Fig. 11.22** Multi-pass filterability test results for different Zn-free hydraulic fluids (8.7 L $min^{-1}$, 65 °C, 100 bar, 15 L volume, 3 μm/6 μm).

**Tab. 11.18** Typical conductivity data for different types of hydraulic oil.

| *Type of hydraulic oil* | *23 °C* | *50 °C* |
|---|---|---|
| Mineral oil, demulsifying, Zn-containing | 200 pS $m^{-1}$ | 800 pS $m^{-1}$ |
| Mineral oil, DD, Zn-containing | 470 pS $m^{-1}$ | 2000 pS $m^{-1}$ |
| Mineral oil, DD, Zn-containing | 8.000 pS m | 40 000 pS m |
| Mineral oil, demulsifying, Zn and ash-free | 4 pS $m^{-1}$ | 17 pS $m^{-1}$ |
| Mineral oil, DD, Zn and ash-free | 140 pS $m^{-1}$ | 690 pS $m^{-1}$ |
| Mineral oil, DD, Zn and ash-free | 360 pS $m^{-1}$ | 1100 pS $m^{-1}$ |

### 11.6.5.11 **Micro Scratching**

When low-performance zinc and ash-free hydraulic oils are used instead of zinc-containing fluids a new phenomenon, micro scratching, very small scratches equally distributed all around the piston in the axial direction, is occasionally observed. Some discoloration also occurs and surface roughness reaches peaks of 2 μm compared with 0.1 μm for undamaged surfaces. Examples of damaged and undamaged pistons are shown in Fig. 11.23. The fluid-related reason for this micro scratching is the use of zinc and ash-free hydraulic oils with low EP/AW performance. A special piston test rig at Busak + Shamban in Stuttgart (Germany) has been used to characterize different characteristics of hydraulic oils with regard to micro scratching [11.68]. The conclusion was that Zn-containing hydraulic fluids and Zn-free hydraulic fluids containing high levels of selected EP/AW additives do not usually lead to micro scratching. Film forming properties also affect the behavior positively and detergent/dispersant type hydraulic fluids are therefore recommended in combination with zinc and ash-free chemistry.

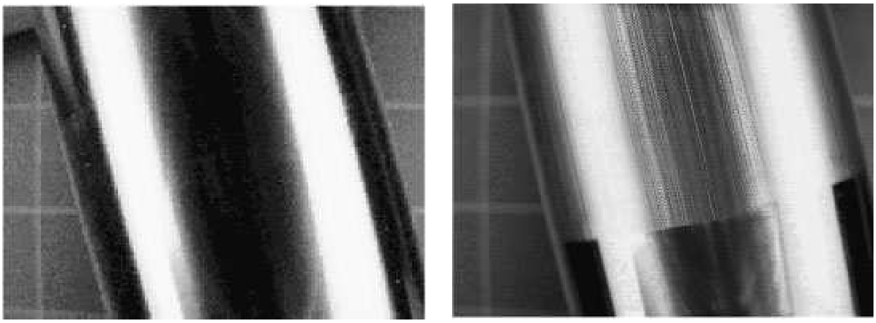


**Fig. 11.23 Pistons without and with micro scratches.**

### 11.6.5.12 Updated Standards

DIN 51 524 for hydraulic fluids was updated in 2006. Major changes were the declaration of purity levels, changes of limits, for example air release from 10 min to 13 min, and introduction of a tapered roller bearing shear-stability test in DIN 51 524-3 (result to be reported only).

European Eco-Label "Margerite" with EU Directive 2005/360/EC was introduced in 2005. For biodegradable hydraulic fluids it is based on technical specification ISO 15 380. Besides requirements for biodegradability, sustainability is also requested – 50 % of the raw materials must be derived from renewable materials.

ISO 6743-4 also was recently updated in respect of technical requirements for mineral oil-based hydraulic fluids

### 11.6.5.13 Conclusion

The variety of available hydraulic fluids are vast, as are their individual technical requirements. Each application, with its ambient conditions, must therefore be considered in detail when selecting the optimum hydraulic fluid. The optimum depends on many different fluid characteristics. It may be the choice between Zn-containing and zinc and ash-free hydraulic fluids, synthetic base oil or mineral base oil, high additive performance or not, demulsifying properties or detergent/dispersant fluid characteristics. The proper choice affects the performance, lifetime, availability, and cost effectiveness of machinery and its hydraulic equipment. Hydraulic fluids are important liquid machine-tools.

## 11.7
## Summary

In technical and economic terms, widely varying specifications and application conditions require different hydraulic fluids. These also have to satisfy a broad performance span. Table 11.19 shows the most important characteristics of hydraulic fluids and the most important fluid groups [11.2, 11.4, 11.6, 11.33].

Hydraulic oils are key elements in machines and machine tools must therefore be included in the planning of plant and equipment to reflect the different properties offered by the various types of fluid.

**Tab. 11.19** Important properties of hydraulic fluids (viscosity class ISO VG 46).

| | ***Mineral oil*** | ***Emulsion/ solution*** | ***Polymer solution*** | ***Synthetic ester*** | ***Synthetic ester*** | ***Triglycerides rapeseed oil*** |
|---|---|---|---|---|---|---|
| DIN 51 502 | HLP | HFA | HFC | HFDR | HFDU/ HEES* | HETG |
| Density at 15 °C [g $cm^{-3}$] | 0.89 | 1.0 | 1.04–1.09 | 1.1–1.2 | 0.93–0.94 | 0.92–0.93 |
| Kinematic viscosity at 40 °C [$mm^2\ s^{-1}$] | 46 | 1–2 | 46 | 46 | 46 | 46 |
| Viscosity index [VI] | 100 (good) | – | 150–200 (very good) | ca. 0–80 (low) | 140–190 (very good) | 200–220 (very good) |
| Average compression modulus, M [N $m^{-2}$] | $2 \times 10^9$ | $2.5 \times 10^9$ | $3.5 \times 10^9$ | $2.3–2.8 \times 10^9$ | $2.3–2.8 \times 10^9$ | $1.8–2.5 \times 10^9$ |
| Specific heat at 20 °C [kJ $kg^{-1}\ K^{-1}$] | 1.9–2.2 | 4.2 | 3.3 | 1.3–1.5 | 1.3–1.5 | 2.1 |
| Thermal conductivity, $\lambda$, at 20 °C [W $m^{-1}\ K^{-1}$] | 0.13–0.14 | 0.6 | 0.3–0.43 | 0.11–0.12 | 0.11–0.13 | 0.15–0.18 |
| Expansion coefficient, $\beta_{Tr}$ [$K^{-1}$] | $7 \times 10^{-4}$ | $1.8 \times 10^{-4}$ | $7 \times 10^{-4}$ | $6.8 \times 10^{-4}$ | $7 \times 10^{-4}$ | $7.5 \times 10^{-4}$ |
| Recommended temperature range/operating temperatures [°C] | –10–100 | 5–< 55 | –20–60 | –10–100 | –10–100* | 0–70 |
| Max. temperature range/operating temperatures [°C] | –35–120 | 0–55 | –35–65 | –20–150 | –35–130* | –20–90 |
| Flash point [°C] | ca. 220 | n.a.** | n.a.** | 240–250 | 250–300 | ca. 315 |
| Ignition temperature [°C] | 310–360 | none | none | 500–550 | 450–500 | 350–500 |

**Tab. 11.19** Continued.

| | ***Mineral oil*** | ***Emulsion/ solution*** | ***Polymer solution*** | ***Synthetic ester*** | ***Synthetic ester*** | ***Triglycerides rapeseed oil*** |
|---|---|---|---|---|---|---|
| DIN 51 502 | HLP | HFA | HFC | HFDR | HFDU/<br>HEES* | HETG |
| Pourpoint [°C] | < –18 | ≈ 0 | < –30 | < –18 | < –30 | ca. –25 |
| Bunsen coefficient, $\alpha_V$, at 20 °C for air | 0.08–0.09 | 0.02 | 0.01–0.02 | 0.012–0.02 | 0.012–0.02 | 0.05–0.06 |
| Acoustic velocity at 20 °C [$m\ s^{-1}$] | 1.300 | 1.400 | ca. 1.400 | – | – | – |
| Vapor pressure at 50 °C [mbar] | $10^{-4}/10^{-5}$ | 120 | ca. 50–80 | $< 10^{-5}$ | $< 10^{-5}$ | $< 10^{-5}$ |
| Risk of caviation | low | very high | middle | low | low | low |
| Relative costs of the fluid [%] | 100 | 10–15 dependant on concentration | 200–300 | 800–900 | 300–600 | 200–250 |
| Market share [%] | ca. 88 | < 1 | ca. 4–5 | < 1 | < 1/2–3 | ca. 1–2 |

Literature:
- Properties according to VDMA 24317, August 1982
- Hydrostatische Pumpen und Motoren, J. u. M. Ivantysyn, Vogel Verlag, Germany
- Grundlagen der Fluidtechnik, H. Murrenholt, IFAS, Aachen, Germany
- TAE: ‘Hydraulikflüssigkeiten’, Nr. 24248/68.479, Esslingen, Germany

* Saturated esters; ** not applicable, no flashpoint

# 12
# Compressor Oils

## 12.1
## Air Compressor Oils

*Wolfgang Bock and Georg Lingg*

Compressors increase the pressure of air or any gaseous medium in one or more stages and thus transfer energy to the medium. As the volume of the medium is reduced, its temperature and density increase.

There are two types of compressor: displacement compressors and dynamic compressors. In displacement compressors, the gaseous medium is drawn into a chamber, compressed and expelled by a reciprocating piston. The principle of dynamic compressors is that turbine wheels accelerate a medium which is then abruptly de-accelerated [12.1]. In the past 25 years, traditional piston compressors have increasingly been replaced by rotary compressors and in particular screw compressors so that the market share of screw and rotary vane compressors is presently about > 60 %. The reasons for this are the light weight and compact dimensions of rotary compressors, there low-noise and vibration-free operation and their reliability. Rotary compressors are generally characterized by the constant, relatively lower-pressurization of larger volumes of air while piston compressors provide pulsating higher-pressurization of smaller volumes.

Compared to piston compressors in which the oil primarily lubricates the bearings, pistons, cylinders and valves, the oil in oil-flooded screw and rotary vane compressors also has the additional function of cooling and sealing. It is possible to differentiate between air and gas compressors, vacuum pumps and refrigerant compressors by analyzing the function of the oil. Figs. 12.1 and 12.2 show a breakdown of compressors according to their construction and to their operative range [12.2, 12.3].

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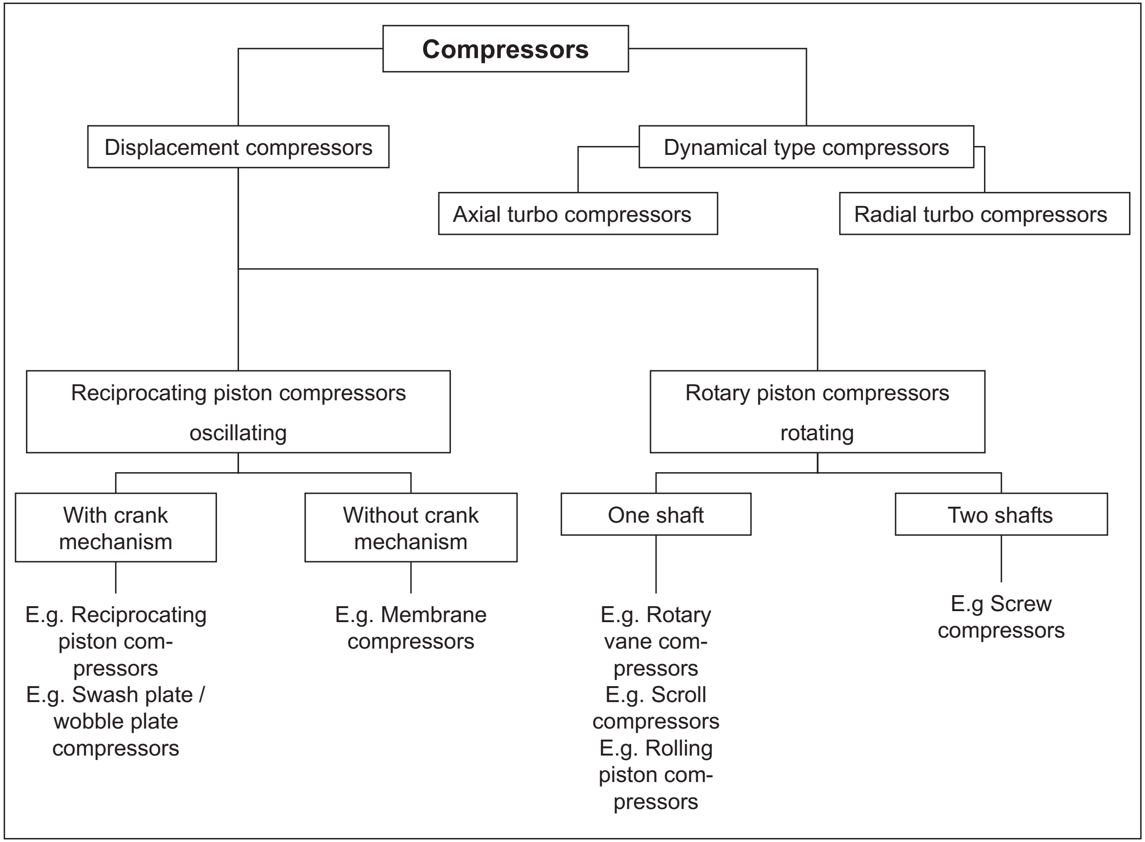


**Fig. 12.1** Classification of compressors according to their construction.

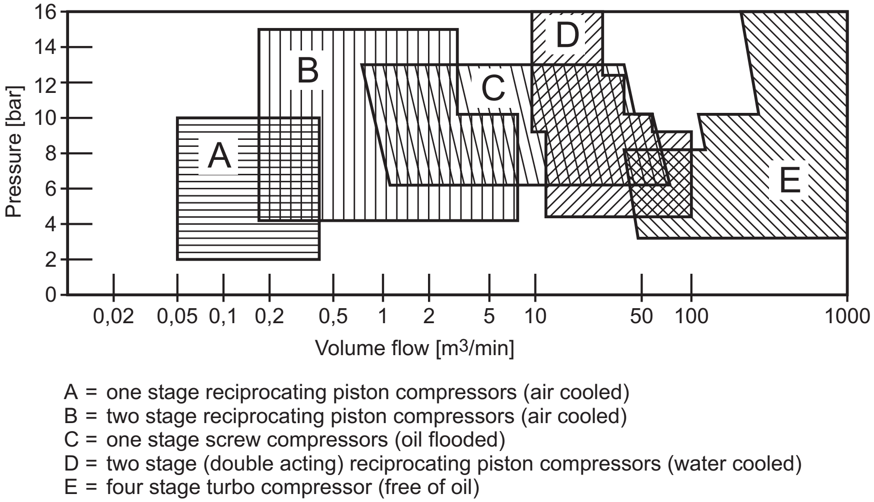


Figure: Mahle Druckluft, Germany

**Fig. 12.2** Classification of compressors according to their operative range.

### 12.1.1
### Displacement Compressors

#### 12.1.1.1 Reciprocating Piston Compressors

Reciprocating piston compressors increase the pressure of the medium by decreasing the volume of its chambers. The air or gas is compressed and displaced by the rising and falling of a piston in a sealed cylinder. The compression and pumping of the medium results from the periodic (oscillating) change in volume. Each stage of the process is controlled by inlet and outlet valves. The compressor is cooled either by air circulating around the ribs attached to the cylinder head or by water which flows through a jacket around the cylinder [12.2, 12.3].

#### 12.1.1.2 Lubrication of Reciprocating Piston Compressors

In reciprocating piston compressors, the piston is connected to the crankshaft by a connecting rod. In general, the cylinder and the drive share the same splash lubrication from oil in the crankcase. In larger compressors, the pistons are driven by cross-head rods (cross-head compressors, single or double action). In such compressors, the drive is lubricated by crankcase splash and separately from the cylinders [12.2].

The cylinders in a piston compressor represent the most difficult task for the lubricant and ultimately decide the choice of lubricants. The lubricant's primary tasks are the reduction of friction and wear, sealing the compression chambers and protection against corrosion. The peak stress occurs at the TDC and the BDC (top and bottom dead center). At these points, there is a danger of the lubricant film tearing and allowing metal-to-metal contact. The oil is also subject to enormous stress resulting from the high temperatures created when the medium is compressed (which can cause oxidation and lead to deposits) and in the case of air, the oxygen enrichment. The cleanest possible air or gas should be compressed because contaminants can accelerate oxidation and wear (especially the water content of the air/gas and other contamination – e.g. aggressive gases from the surroundings can influence the performance of the used lubricant in an extremely negative way). In the case of drive unit lubrication, the lubrication of the bearings is of primary importance.

Piston compressors are available in lubricated-with-oil and oil-free versions. Normally, lubricants based on mineral oil according to DIN 51 506–VCL, VDL (or PAO- or diester-based lubricants) are used with viscosity grades of ISO VG 68 to ISO VG 150. Mobile compressors are often lubricated with monograde engine oil (SAE 20, SAE 30, SAE 40) [12.4, 12.5]. Small to medium-sized piston compressors are used for pressures up to 10 bar.

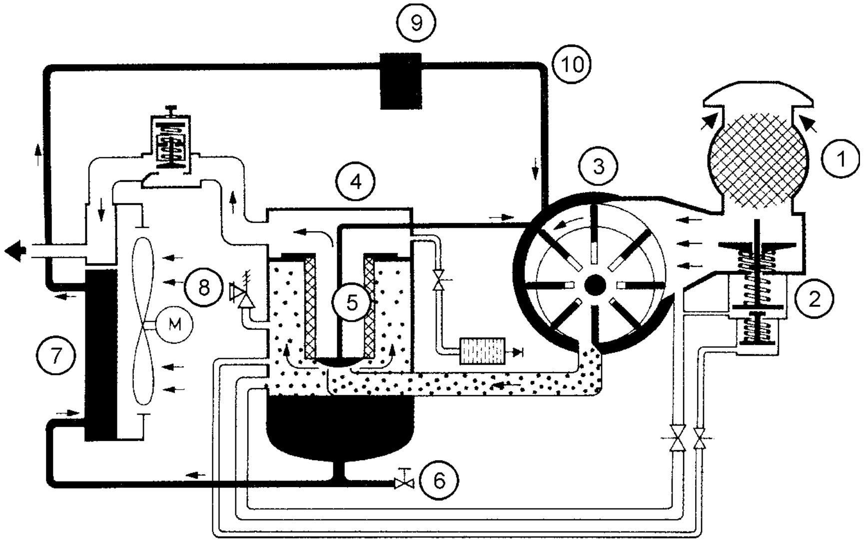
#### 12.1.1.3 **Rotary Piston Compressors (Single Shaft, Rotary Vane Compressors)**

In these compressors, the volume of the pressure chambers varies periodically between two extremes. The eccentric cylindrical rotor which is located in the cylindrical sleeve of the housing has sliders (normally steel or PTFE) fitted in grooves which alter the star-shaped chamber between the rotor and the housing. When the rotor turns, centrifugal force presses the sliders against the wall of the housing. During rotation, the enlargement of the chamber on the inlet side draws air in and as the volume gets smaller the medium is compressed until it is expelled through the outlet. The inlet and compression steps are controlled by slots in the housing.

In single-action operation, pressures of up to 10 bar are possible and up to 16 bar in double-action operation. Volumes can reach 80 $m^3\ min^{-1}$. The advantages of rotary piston compressors are compactness, continuous flow and vibration-free operation compared to piston compressors [12.1, 12.2, 12.6].

#### 12.1.1.4 **Lubrication of Rotary Piston Compressors**

The pressure chambers of rotary piston compressors are cooled and lubricated by total-loss systems or by direct oil injection. The lubrication of rotary piston compressors is similar to the lubrication of the cylinders in reciprocating piston compressors insofar as the lubricant is subject to high outlet temperatures in both cases. In the case of oil-injected and oil cooled rotary piston compressors, a quantity of oil is continuously injected into the compressor chambers. The quantity of the oil is such that the outlet temperature does not exceed 100 °C – 110 °C. At the same time, it seals the pistons against the housing and protects against wear. The cooling of the medium results in an increase in compression performance. Cooling and sealing increase the volumetric efficiency and thus the overall efficiency of the compressor. The oils used are normally VCL or VDL according to DIN 51 506 with an ISO VG between 68 and 150 or monograde SAE 20, SAE 30, SAE 40 engine oils. Rotary piston compressors are mostly used for vehicle and railroad applications (including road tankers). Outlet pressures are mostly less than 10 bar [12.1, 12.6]. Figure 12.3 shows the lubrication circuit of a rotary vane compressor (oil cooled).



| | | | |
|---|---|---|---|
| ① | Inlet filter | ⑥ | Drain value |
| ② | Regulator | ⑦ | Oil cooler |
| ③ | Compression stage | ⑧ | Cooling fan |
| ④ | Oil reservoir | ⑨ | Oil filter |
| ⑤ | Oil seperator | ⑩ | Oil pressure |

Figure: Mannesmann Demag, Wittig

**Fig. 12.3** Lubrication circuit of a rotary vane compressor (oil cooled).

### 12.1.1.5 Screw Compressors

Screw compressors have two counter-rotating axial shafts (screws) and use the displacement principle. One of the two screws is the compressor and the other is an idler and both revolve in the same housing. The inlet area has a large cross-section and volume. As the two shafts revolve, the volume gets smaller and compression takes place. The compressed medium then leaves the housing the outlet. The compressor screw has a helical convex cross section while the idler has a helical concave cross section. The two rotors of oil-free screw compressors are geared to each other so that their surfaces never touch. On the other hand, the rotors in oil-flooded screw compressors contact each other and thus do not require the geared coupling.

The advantages of screw compressors are compact size, minimum vibration and continuous flow [12.1, 12.2, 12.7, 12.8].

### 12.1.1.6 Lubrication of Screw Compressors

In oil-injected screw compressors, the oil has a lubricating, sealing and cooling function. The lubricant is injected into the pressure chamber between the rotors at about 3–4 bar. It then forms a hydrostatic and a hydrodynamic lubricating film. The oil therefore lubricates the meshing rotors and the plain and roller bearings which are part of the geared coupling. Furthermore, it seals the gaps between the rotor and the housing. It also helps absorb heat and dissipate this via radiators. The temperature of the compressed air of about 80 °C to 100 °C is adjusted by the quantity of oil injected. Downstream oil separators (normally cartridge filters) remove the oil from the air. Residual oil quantities of 1–3 mg $m^{-3}$ of air can be achieved. The separated oil de-aerates in tanks, is then filtered and cooled from about 80 °C to 50 °C. As the oil is one the pressure side of the screw compressor (e.g. up to 10 bar), this pressure can be used to re-inject the oil.

As the viscosity of the oil is of primary importance to elasto-hydrodynamic lubrication and thus for the mechanical stability of the film, it must be matched to start-up and normal running conditions. As a rule, ISO VG 46 lubricants cover most manufacturer's recommended viscosity thresholds of about > 10 $mm^2$ $s^{-1}$ at operating temperature to about 500 $mm^2$ $s^{-1}$ when starting-up. This range also satisfies most applications in central Europe. Higher viscosity ISO VG 68 oils or synthetic ester-PAG or PAO-based lubricants are used in countries with high ambient temperatures. In recent years, lubricants based on hydrocracked oils (so called group III oils) have found increasing acceptance. Screw compressor oils have mild EP/AW performance, FZG failure load stage ≥ 10 are typically required. In relation to their size and weight, the volumes achievable with screw compressors are excellent. Screw compressors are primarily used for mobile applications as well as for industrial applications such as in the glass and paper industries and general industry. Pressures of up to about 10 bar and higher are possible with screw compressors [12.4, 12.7, 12.8].

#### 12.1.1.7 **Roots Compressors**

Roots compressors normally consist of two symmetrical, figure-of-eight-shaped rotors in a housing. The counter-rotating rotors are driven by external gears and do not touch. The oil's sole task is to lubricate the rotors' gears and bearings. The benefits of this type of compressor are oil-free air, large volumes and low vibration [12.2].

#### 12.1.1.8 **Lubrication of Roots Compressors**

Recommended lubricants include DIN 51 517 CL and CLP or HD SAE oils in the viscosity grades ISO VG 68 and ISO VG 100 [12.4].

### 12.1.2
### Dynamic Compressors

#### 12.1.2.1 **Turbo Compressors**

Turbo compressors are dynamic machines which convert dynamic energy into compression energy. The medium is accelerated by one or more rotors and the dynamic energy is converted into compression energy at the medium's fixed outlet point. Radial and axial turbo compressors differ in that the inlets to the rotors are either radial or axial.

The advantages of turbo compressors are high volumes, minimum vibration and oil-free air [12.1, 12.2].

#### 12.1.2.2 Lubrication of Turbo Compressors

The oils for this type of compressor lubricates bearings, radial shaft seals and possibly gears via a positive-feed circuit. In some cases, the bearings are lubricated with grease. Ideally, the same lubricant should be used for the compressor and its drive. Most often, DIN 51 515 TDL 32, TDL 46 and TDL 68 turbine oils or TDL-EP grades (EP–Extreme Pressure Additives) are used.

Turbo compressors are principally used for creating compressed air in mines and industrial manufacturing plants [12.3, 12.4].

### 12.1.3

### Preparation of Compressed Air

The oil injected into oil-cooled screw and rotary piston compressors is always removed from the compressed air. The oil which is mixed with the highly compressed air is removed and collected in single or multistage downstream oil separators. Before the oil is re-circulated, it is filtered and cooled. Depending on the specific requirements, the compressed air may then pass a number of subsequent treatment stages such as refrigerant dryers or absorption dryers (to reduce the water content coming from the humidity of the air/gas). Very low residual oil quantities in the air can be achieved by the fitting of a series of in-line oil separators [12.6–12.8].

### 12.1.4

### Lubrication of Gas Compressors

#### 12.1.4.1 Oxygen Compressors

Because of explosion hazards when oxygen is compressed, pressure chambers lubricants must be mineral oil-free. Water and water-based solutions such as glycerin can be used for cylinder lubrication. Mineral oil-based products may be used for compressor drives if it does not come into contact with the pressure chambers [12.4].

Oxygen compressors can be lubricated with inert lubricants based on perfluorether oils (which are extreme expensive).

#### 12.1.4.2 Acid Gas Compressors

Gases often contain acidic components such as $SO_2$ or $NO_x$. If standard compressor oils were used for such applications, the lubricating oil would soon become overacidified. To counter this, lubricants are used for such applications which contain highly alkaline additives. These components can neutralize the acidic components in the gas. In these cases, it is recommended that monograde engine oils (20W-20, 30W, 40W) with high alkaline reserves (high TBN) are used [12.4].

#### 12.1.4.3 Inert Gas Compressors

When inert gases are compressed, the same rules as for air compressors should be used [12.2, 12.4].

#### 12.1.4.4 Hydrocarbon Compressors

Hydrocarbons such as ethane, propane etc. are easily soluble in mineral oil. This causes the viscosity of the lubricating oil to fall if mineral oil-based products are used. For this reason, higher viscosity mineral oils such as ISO VG 100 and ISO VG 150 must be used in piston compressors whose crankcases are subject to low inlet pressures (1–3 bar). In the case of screw compressors (high pressure; 10–15 bar), ISO VG 68, 100, 150, and 220 ester- or polyglycol-based lubricants with lower hydrocarbon solubility are recommended [12.2, 12.4].

#### 12.1.4.5 Vacuum Pump Lubrication

Vacuum pumps are compressors whose inlet is connected to the chamber where the vacuum is created. VDL compressor oils can be used for low vacuums. Greater vacuums require synthetic oils with low vapor pressures (mostly synthetic ester oils). The lubricant selection must consider if the medium to be extracted is not air, but for example a refrigerant. In such cases, a compatible refrigeration oil can be used [12.4].

### 12.1.5
### Characteristics of Compressor Oils

Compressors whose chambers are lubricated pose particular safety problems if air or aggressive gases contact the lubricant. The selection of the most suitable lubricant depends on the type of compressor in question, the pressures involved, the outlet temperatures and the type of air/gas being compressed. Piston compressors which generate the highest pressures are particularly problematic. Turbo compressors which only have lubricated bearings and non-lubricated pressure chambers pose the least problems. Rotary and screw compressors with outlet pressures under 10 bar and correspondingly low outlet temperatures are examples of average compressor lubrication application. Table 12.1 shows an overview of normally used compressor oils.

In general, reciprocating piston compressors need lubricants with higher viscosity (ISO VG 100 or ISO VG 150), extremely low carbon residues, and no or mild EP/AW-performance additives. Screw compressors need lubricants of lower viscosity (ISO VG 46 or 68) with excellent oxidation stability and mild/high AW/EP performance additives.

### 12.1.6
### Standards and Specifications of Compressor Oils

DIN 51 506 describes the classification and requirements of lubricating oils which are used in reciprocating piston compressors with oil-lubricated pressure chambers (also for vacuum pumps). Lubricants for screw and oil-injected rotary vane and screw compressors are not included in DIN 51 506 [12.5]. Table 12.2 shows the classification of air compressor oils according to DIN 51 506. Tables 12.3 and 12.4 contain the minimum requirements of air compressor oils according to DIN 51 506.

**Tab. 12.1** Overview of normally used compressor oils. The viscosity and the quality recommendations of the compressor manufacturers must be taken into consideration.

| *Viscosity classification* | *Type Piston compressors*[a] | *Screw compressors oil-injected* | *Sliding vane compressors*[a,b] | *Turbo compressors (axial and radial)*[c] |
|---|---|---|---|---|
| ISO VG 32 | | MO<br>HC-Oils<br>PAO | | TDL 32<br>TDL 32 EP<br>Synth. Oils |
| ISO VG 46 | | MO<br>HC-Oils<br>PAO<br>POE | | TDL 46<br>TDL 46 EP<br>Synth. Oils |
| ISO VG 68<br>(SAE 20W-20) | MO<br>PAO<br>Diester | MO<br>HC-Oils<br>PAO<br>POE | MO<br>Diester<br>HC-Oils | TDL 68<br>TDL 68 EP<br>Synth. Oils |
| ISO VG 100<br>(SAE 30) | MO<br>PAO<br>Diester | | MO<br>Diester<br>HC-Oils | |
| ISO VG 150<br>(SAE 40) | MO<br>PAO<br>Diester | | | |

MO = mineral oil; PAO = polyalphaolefin;
HC = hydrocrack oil (so-called group III oils);
POE = biologically degradable polyolesters

- Diester, Polyolester and PAO: for very hard working conditions, increase of service intervals is possible
- HC Oils (so-called group III oils): for medium and hard working conditions
- MO: for normal and medium working conditions

- Lubricants for Roots-compressors: HL, CL, CLP; ISO VG 100-150, DIN 51 524, DIN 51 517
- Lubricants for vacuum pumps: ISO VG 68-150

a) Total-loss lubrication: HD-monograde motor oils HD 20W-20, HD 30, HD 40

b)
- For oil-injected compressors in mobile equipment (e.g. railways, buses): multigrade motor oils (e.g. 10 W 40, 15 W 40)
- For oil-injected compressors in stationary units: turbine oils according to DIN 51 515 TDL, air compressor oils according to DIN 51 506 VCL, VDL
- For hard working conditions: monograde motor oils HD 20W-20, HD 30, MIL 2104 D

c) Turbine oils according to DIN 51 515 TDL or TDL-EP with extreme pressure additives

**Tab. 12.2** Classification of air compressor oils according to DIN 51 506–Table 1, September 1985.

| | ***Maximum compressed air temperature (°C)*** | |
|---|---|---|
| ***Lubricating oil category*** | ***For compressors on moving (mobile) equipment for brakes, signals and tippers*** | ***For compressors with storage tanks and pipe network systems*** |
| VDL | Up to 220 | Up to 220 |
| VC | Up to 220 | Up to 160[a)] |
| VCL | | |
| VB | Up to 140 | Up to 140 |
| VBL | | |

a) Rotary multi-vane compressors designed for a once-through lubrication can be operated at compressor end temperatures of up to 180 °C using lubricating oils doped in the same manner as motor lubricants or doped compressor oils, provided that the requirements specified for VCL lubricating oils in table 2 are complied with.

According to this standard, such lubricants are pure mineral oils or mineral oils with additives to increase oxidation stability aging resistance and corrosion protection. The classification of the lubricants depends on the expected outlet temperatures and the general application. DIN 51 506 differentiates between lubricants for mobile applications and stationary applications with reservoirs. Principal differences between the listed groups: VB/VBL, VC/VCL as well as VDL are the use of oxidation and corrosion inhibitors, aging stability and residue formation and the quality of the base oils (cuts). The difference between group VB/VBL and group VC/VCL lies in the aging behavior (formation of residues coke after air-induced aging). Group VDL oils have to pass a more difficult aging test (formation of Conradson coke after air-induced aging in the presence of ferrous oxide). DIN 51 506 VDL oils display the best thermal and oxidation stability and form the least residues. The selection criteria in DIN 51 506 were adopted into the 1983 ISO/DP 6521 draft [12.5, 12.9]. Table 12.5 defines the requirements of air compressor oils for reciprocating piston compressors. Instead of mineral oil-based lubricants, synthetic compressor oils based on hydrotreated (group III base oils) or polyalphaolefin or ester oils have found increasing commercial acceptance (long-life oils).

The selection criteria for screw compressor and piston compressor oils differ greatly. In oil-flooded rotary vane and screw compressors, the injected oil is constantly in contact with the 80–100 °C hot medium gas or air being compressed. The compressed medium and the oil are well mixed and the oil has to be separated by downstream separators and filters. This places special demands on the lubricant. On the whole, these include low foaming, excellent air release and good demulsification (separation) of condensed water. In addition of course, the specifications regarding wear protection (FZG ≥ 10, DIN 51 354), minimum formation of deposits, good corrosion protection etc. also apply. Most manufacturers of oil-injected rotary and screw compressors issue their own lubricant specifications. A draft of ISO/DP 6521 for oil-injected screw compressors has existed since 1983. Table 12.6 describes the requirements of air compressor oils for screw compressors.

**Tab. 12.3** The minimum requirements of air compressor oils according to DIN 51 506 – VB, VBL, VC, VCL.

| ***Grades VB and VC are pure mineral oils, Grades VB-L, VC-L and VD-L contain additives L to increase aging resistance and corrosion protection.*** | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Lube Oil Group*** | ***VB and VB-L*** | | | | | | | | | ***VC and VC-L*** | | | | |
| ***Viscosity Grade*** | ***ISO VG 22*** | ***ISO VG 32*** | ***ISO VG 46*** | ***ISO VG 88*** | ***ISO VG100*** | ***ISO VG150*** | ***ISO VG220*** | ***ISO VG320*** | ***ISO VG460*** | ***ISO VG 32*** | ***ISO VG 46*** | ***ISO VG 68*** | ***ISO VG100*** | ***ISO VG150*** |
| Kinematic Viscosity (DIN 51 561 / 51562 part 1)<br>cST @ 40 °C Min.<br>Max. | 19.8 to 24.2 | 28.8 to 35.2 | 41.4 to 50.6 | 61.2 to 74.8 | 90 to 110 | 135 to 165 | 198 to 242 | 288 to 352 | 414 to 506 | 28.8 to 35.2 | 41.4 to 50.6 | 61.2 to 74.8 | 90 to 110 | 135 to 165 |
| cST @ 100 °C | 4.3 | 5.4 | 6.6 | 8.8 | 11 | 15 | 19 | 23 | 30 | 5.4 | 6.6 | 8.8 | 11 | 15 |
| Flash Point, °C (COC) min. (DIN ISO 2592) | 175 | | 195 | | 205 | 210 | 225 | | 255 | 175 | 195 | | 205 | 210 |
| Pour Point, °C, max. (DIN ISO 3016) | –9 | | | | | –3 | 0 | | | –9 | | | | –3 |
| Ash, % wt. max. (DIN 51 575) | VB & VC: max. 0.02 % oxide ash; VB-L, VC-L: Sulf. ash to be stated by the supplier | | | | | | | | | | | | | |
| Water soluble acids (DIN 51 558 part 1) | Neutral | | | | | | | | | | | | | |
| Neutralization number (acid), mg KOH $g^{-1}$ max. (DIN 51 558 part 1) | VB & VC: max. 0.15 mg KOH/g; VB-L, VC-L: To be stated by the supplier | | | | | | | | | | | | | |
| Water, % (DIN ISO 3733) | 0.1 max. | | | | | | | | | | | | | |

**Tab. 12.3** Continued.

| ***Grades VB and VC are pure mineral oils, Grades VB-L, VC-L and VD-L contain additives L to increase aging resistance and corrosion protection.*** | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Lube Oil Group*** | ***VB and VB-L*** | | | | | | | | | ***VC and VC-L*** | | | | |
| ***Viscosity Grade*** | ***ISO VG 22*** | ***ISO VG 32*** | ***ISO VG 46*** | ***ISO VG 88*** | ***ISO VG100*** | ***ISO VG150*** | ***ISO VG220*** | ***ISO VG320*** | ***ISO VG460*** | ***ISO VG 32*** | ***ISO VG 46*** | ***ISO VG 68*** | ***ISO VG100*** | ***ISO VG150*** |
| Aging characteristics % CRC[a] max. after air aging (DIN 51 352 part 1) | 2.0 | | | | | 2.5 | | | | 1.5 | | | | 2.0 |
| % CRC max. after air/$Fe_2O_3$ aging (DIN 51352 part 2) | Not required | | | | | | | | | | | | | |
| Distillation residue % CRC max. of 20 % distillation residue (DIN 51 356 / 51 551) | Not required | | | | | | | | | 0.3 | | | | 0.75 |
| Kinematic Viscosity at 40 °C, max. of 20 % dist. residue (DIN 51 536 / 51 561 / 51 562 part 1) | Not required | | | | | | | | | | | | | |

[a] CRC – Conradson carbon residue

**Tab. 12.4** Minimum requirements of air compressor oils according to DIN 51 506–VDL.

| ***Lube Oil Group*** | ***VDL*** | | | | |
|---|---|---|---|---|---|
| ***Viscosity Grade*** | ***ISO VG 32*** | ***ISO VG 46*** | ***ISO VG 68*** | ***ISO VG 100*** | ***ISO VG 150*** |
| Kinematic Viscosity (DIN 51 561 / 51 562-1) cST @ 40 °C | 28.8 to 35.2 | 41.4 to 50.6 | 61.2 to 74.8 | 90 to 110 | 135 to 165 |
| cST @ 100 °C | 5.4 | 6.6 | 8.8 | 11 | 15 |
| Flash Point, °C (COC), min. (DIN ISO 2592) | 175 | 195 | | 205 | 210 |
| Pour Point, °C, max. (DIN ISO 3016) | –9 | | | | –3 |
| Ash, % wt., max. (DIN 51 575) | Sulf. ash to be stated by the supplier | | | | |
| Water soluble acids (DIN 51 558 part 1) | Neutral | | | | |
| Neutralization number (acid), mg KOH/g, max. (DIN 51 558 part 1) | To be stated by the supplier | | | | |
| Water, % (DIN ISO 3733) | 0.1 max. | | | | |
| Aging characteristics % CRC max. after air aging (DIN 51 352 part 1) | Not required | | | | |
| % CRC max. after air/$Fe_2O_3$ aging (DIN 51352 part 2) | 2.5 | | | 3.0 | |
| Distillation residue % CRC max. of 20 % distillation residue (DIN 51 356 / 51 551) | 0.3 | | | | 0.6 |
| Kinematic Viscosity at 40 °C max. of 20 % distillation residue (DIN 51 536 / 51 561 / 51 562-1) | maximum of five times the value of the new oil | | | | |

The selection criteria of DIN 51 506 and ISO/DP 6521, along with the recommendation of the compressor manufacturers should be observed when choosing an air compressor oil. At the same time, attention must be paid to the safety procedures issued by industrial and professional associations and to the specifications of the compressor manufacturers.

ISO 6743-3A (1987) divides oil-lubricated air compressors into six mechanical type groups but does not list any physical or chemical characteristics which the lubricants must fulfil [12.5, 12.9].

**Tab. 12.5** The requirements of air compressor oils for reciprocating piston compressors – ISO/DP 6521 – Draft 1983.

| ***Mineral Oil-based Lubricants for Reciprocating Piston Compressors*** | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Category*** | ***ISO-L-DAA*** | | | | | ***ISO-L-DAB*** | | | | | ***Test Method*** |
| Viscosity Grades | 32 | 46 | 68 | 100 | 150 | 32 | 46 | 68 | 100 | 150 | ISO 3448 |
| Viscosity<br>@ 40 °C, cSt ± 10 % | 32 | 46 | 68 | 100 | 150 | 32 | 46 | 68 | 100 | 150 | ISO 3104 (IP71) |
| @ 100 °C cSt | To be stated | | | | | To be stated | | | | | |
| Pour Point*, °C max. | –9 | | | | | –9 | | | | | ISO 3016 (IP15) |
| Copper Corrosion, max. | 1b | | | | | 1b | | | | | ISO 2160 (IP154) |
| Rust | No Rust | | | | | No Rust | | | | | ISO/DP 7120A (IP135A) |
| Emulsion Characteristics Temperature, °C | no requirement | | | | | 54 | | | 82 | | ISO/DP 6614 (ASTM D1401) |
| Time (min) to 3 ml Emulsion, max. | | | | | | 30 | | | | 60 | |
| Oxidation Stability after aging @ 200 °C Evapor. Loss, %, max. | 15 | | | | | no requirement | | | | | ISO/DP 6617 Part 1 (DIN 51 352) |
| Increase in Conradson Carbon Residue, %, max. | 1.5 | | | 2.0 | | | | | | | |
| after aging @ 200 °C Evapor. Loss, %, max. | not applicable | | | | | 20 | | | | | ISO/DP 6617 Part 2 (DIN 51 352) |
| Increase in Conradson Carbon Residue, %, max. | | | | | | 2.5 | | 3.0 | | | |
| Distill. Residue (20 % vol.) Conradson Carbon Residue, %, max. | not applicable | | | | | 0.3 | | | | 0.6 | ISO/DP 6616 with ISO/DP 6615 and ISO 3104 |
| Ratio of Viscosity of residue to that of new oil, max. | | | | | | 5 | | | | | |

* When VG32 or VG46 oils are used in cold climate, pour points lower than –9 °C are required.

**Tab. 12.6** The requirements of air compressor oils for screw compressors – ISO/DP 6521 – Draft 1983.

***Mineral Oil-based Lubricants for Rotary Screw Compressors***

| ***Category*** | ***ISO-L-DAH*** | | | | | ***ISO-L-DAG*** | | | | | ***Test Method*** |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Viscosity Grades | 32 | 46 | 68 | 100 | 150 | 32 | 46 | 68 | 100 | 150 | ISO 3448 |
| Viscosity @ 40 °C, cSt ± 10 % | 32 | 46 | 68 | 100 | 150 | 32 | 46 | 68 | 100 | 150 | ISO 3104 (IP71) |
| Pour Point*, °C max. | –9 | | | | | –9 | | | | | ISO 3016 (IP15) |
| Copper Corrosion, max. | 1b | | | | | 1b | | | | | ISO 2160 (IP154) |
| Rust | No Rust | | | | | No Rust | | | | | ISO/DP 7120A (IP135A) |
| Emulsion Characteristics** Temperature, °C | 54 | | | | 82 | 54 | | | | 82 | ISO/DP 6614 |
| Time (min) to 3 ml Emulsion, max. | 30 | | | | | 30 | | | | | (ASTM D1401) |
| Foaming Characteristics | | | | | | | | | | | ISO/DP 6247 (IP146) |
| Sequence I @ 24 °C Tendency, ml, max. | 300 | | | | | 300 | | | | | |
| Stability, ml, max. | nil | | | | | nil | | | | | |
| Oxidation Stability<br>Evapor. Loss, %, max.<br>Increase in Viscosity, %<br>Increase in Acidity, %<br>Sludge, % weight | To be decided | | | | | To be decided | | | | | To be established |

* When VG32 or VG46 oils are used in cold climate, pour points lower than –9°C are required.

** Required only in those applications where condensation of atmospheric moisture is a problem. Where this does not apply, oils with dispersant additives, which tend to have poor water separating properties, may be used satisfactorily.

## 12.2
## Refrigeration Oils

*Wolfgang Bock*

### 12.2.1
### Introduction

The lubrication of refrigeration compressors occupies a special position in lubrication technology. The longevity expected of refrigeration compressors is closely connected to the high quality which is required of refrigeration oils. The interaction with other substances which the refrigeration oil comes into contact with, and especially the extremely high and low temperatures, makes very specific demands on refrigeration oils.

The principal function of a compressor oil is to lubricate the pistons or rotors and to lubricate and seal the valves and, in some cases, the slip-ring seals. Furthermore, the refrigeration oil must dissipate heat away from hot compressor components and assist in sealing the compression chambers and valves. The refrigeration oil serves as a hydraulic control and functional fluid in refrigeration compressors [12.10, 12.11]. It is vital that refrigeration oil which reaches and collects in colder sections of the circuit in the form of oil vapor or oil mist or as a result of splashing must be returned to the compressor by mechanical means (oil separator) or via the refrigerant flow (refrigerant solubility) in all operating conditions. Figure 12.4 shows the principle of a vapor compression refrigeration cycle and Fig. 12.5 describes the schematic structure of a refrigeration system.

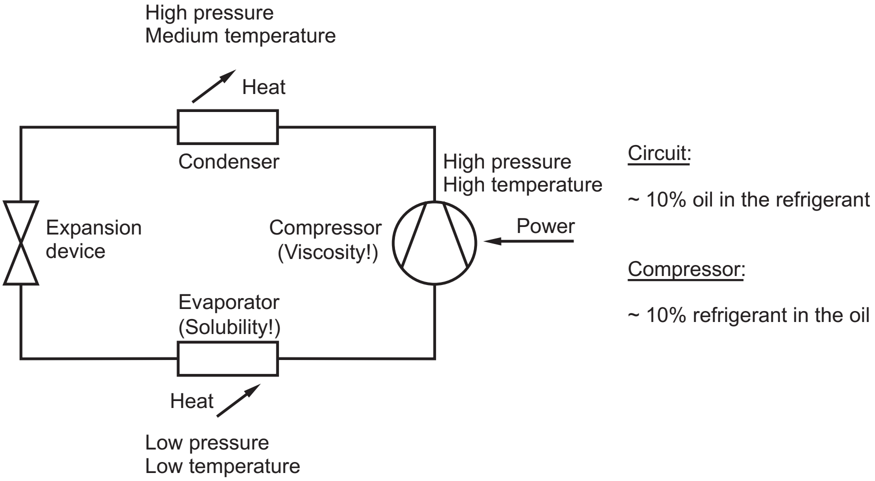


**Fig. 12.4** Principle of a vapor compression refrigeration cycle.

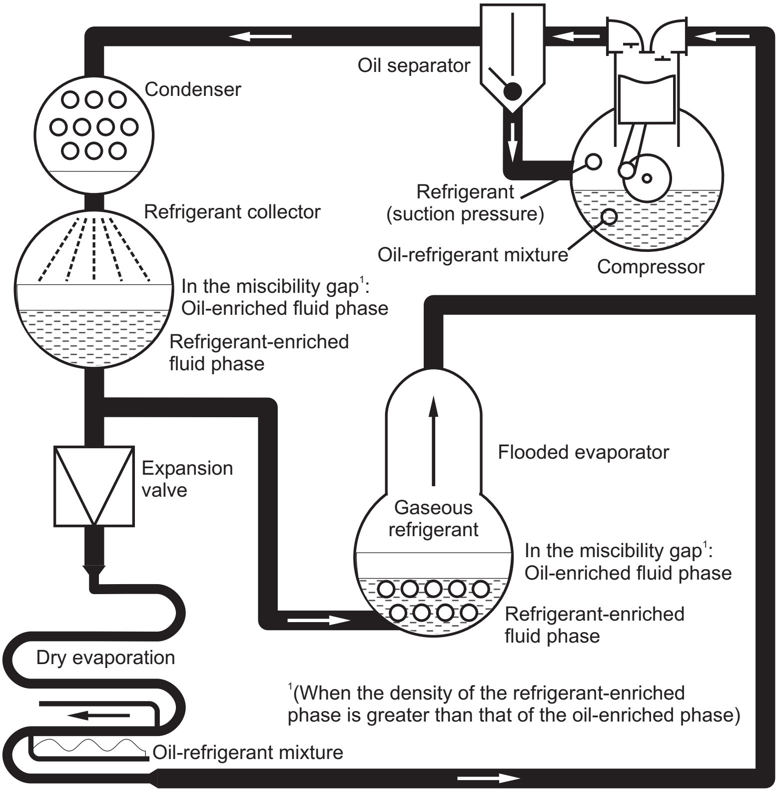


**Fig. 12.5** Schematic structure of a refrigeration system.

### 12.2.2
### Minimum Requirements of Refrigeration Oils

The basic requirements of refrigeration oils are laid down in DIN 51 503-1. This standard defines the basic requirements of refrigeration oils according to the medium being compressed. The introduction of new chlorine-free, polar refrigerants such as HFC R 134a (to replace CFC R 12), necessitated a revision of DIN 51 503 which appeared as DIN 51 503-1 in November 1997.

#### 12.2.2.1 **DIN 51 503-1: Refrigeration oils, Minimum requirements (1997) [12.12]**

Refrigeration oils are classified in alphabetical groups according to the refrigerants being compressed:

| | |
|---|---|
| KAA | Refrigeration oils not soluble in ammonia, ammonia-$NH_3$ oils |
| KAB | Refrigeration oils soluble in ammonia, ammonia-$NH_3$ oils |
| KC | Refrigeration oils for partially- and fully-halogenated fluorinated and chlorinated hydrocarbons (CFC, HCFC) |

| | |
|---|---|
| KD | Refrigeration oils for partially- and fully-fluorinated hydrocarbons (FC, HFC) |
| KE | Refrigeration oils for hydrocarbon refrigerants such as propane or *iso*-butane |

The various refrigerants available are described in DIN 8960 and the ASHRAE standard (ANSI/ASHRAE 34-1992; American Society of Heating, Refrigerating and Air Conditioning Engineers) [12.13].

In addition to appearance, density (ISO 3675) and viscosity (DIN 51 550), several other properties are defined and determined:

| | |
|---|---|
| U-tube flowability | DIN 51 568 |
| Flashpoint | DIN-ISO 2592 |
| Neutralization number | DIN 51 558-3 |
| Saponification number | DIN 51 559 |
| Oxide ash | DIN EN 6245 |
| Water content (K. F.) | DIN 51 777-1 and 51 777-2 |
| Pour point | DIN-ISO 3016 |
| Refrigerant miscibility | E DIN 51 514 (“miscibility gap”) |
| Refrigerant compatibility with R 134a | ASHRAE 97/83 (“Spauchus test”) |

These properties alone do not always provide sufficient information to judge the suitability of a particular refrigeration oil. However, data sheets on oil–refrigerant mixtures “Daniel plots” contain information about the influence of viscosity, density and miscibility on the operational suitability of a refrigeration oil [12.14–12.16]. Table 12.7 gives an overview of the important refrigerants and suitable refrigeration oils in the market.

DIN 51503-2 describes criteria for evaluating used refrigeration oils [12.13].

## 12.2.3
## Classifications of Refrigeration Oils

### 12.2.3.1 Mineral Oils (MO) – Dewaxed Naphthenic Refrigeration Oils

Naphthenic mineral oils are still the most significant group of oils for refrigeration compressors using ammonia ($NH_3$) refrigerants along with CFC and HCFC (e.g. R 22). Naphthenic mineral oils are those which have more than 38 % carbon in naphthenic X(N) bonds. Naphthenic refrigeration oils generally display very low pour points, good cold flowing as well as high thermal and chemical stability. Selected cuts are normally used [12.10, 12.17].

### 12.2.3.2 Mineral Oils (MO) – Paraffinic Refrigeration Oils

Paraffinic mineral oils are those which contain less than about 33 % carbon in naphthenic X(N) bonds.

Paraffinic refrigeration oils are ideal for use in R 11 and R 12 turbo compressors (“old-type”) (ISO VG 68 and 100) because of their good viscosity–temperature behavior. These oils are not recommended for other compressors because of their generally inadequate solubility in refrigerants (miscibility gap, e.g. R22). The boundaries between paraffinic and naphthenic oils are not rigid [12.10, 12.11].

**Tab. 12.7** Classification of important refrigerants and refrigerator oils.

| *Chlorine-free refrigerants and refrigeration oils* | *ASHRAE Name* | *Trade Name* | *Chemical name / formula* | *Refrigeration oil*[a] |
|---|---|---|---|---|
| | R 134a | Diverse | $CH_2FCF_3$ | POE, PAG[c] |
| | R 507 | Solkane 507, AZ 50 | R 125/R 143a | POE |
| | R 404 A | Diverse | R 125/R 143a/ R 134a | POE |
| | R 407 C | Diverse | R 32/R 125/ R 134a | POE |
| | R 410 A | Solkane 410, AZ 20 | R 32/R 125 | POE |
| | R 600a/R 290 | Isobutane/propane | $C_4H_{10}$ / $C_3H_8$ | MO / AB |
| | R 717 | Ammonia | $NH_3$ | MO / PAO / AB |
| | R 744 | Carbon dioxide | $CO_2$ | Synth. Oil[b] |
| | R 723 | 60 % $NH_3$<br>40 % dimethyl ether | $NH_3$/DME | AB/PAO |
| *Drop-in refrigerants and refrigeration oils* | R 22 | Diverse | $CHClF_2$ | MO / AB |
| | R 401 A | MP 39 | R 22/R 152a/ R 124 | MO / AB |
| | R 401 B | MP 66 | R 22/R 152a/ R 124 | MO / AB |
| | R 402 A/B | HP 80/81 | R 22/R 125/ R 290 | MO / AB |
| | R 403 A/B | 69 S/L | R 22/R 218/ R 290 | MO / AB |
| | R 408 A | FX 10 | R 22/R 143a/ R 125 | MO / AB |

a) AB = alkylbenzene oil; MO = mineral oil; PAG = polyalkyleneglycol; PAO = polyalphaolefins; POE = polyolester oil

b) Development product; POE, PAG, ... with AW/EP additives

c) PAG – for A/C R134a-systems

### 12.2.3.3 Semi-Synthetic Refrigeration Oils – Mixtures of alkylbenzenes and mineral oils (MO/AB)

Semi-synthetic refrigeration oils are mixtures of highly-stable alkylbenzenes and highly-refined naphthenic mineral oils. The presence of alkylbenzenes greatly improves the solubility and thermal stability of the naphthenic components. The proportion of synthetic components is usually between 30 and 60 %. Semi-synthetic oils are recommended for CFC/HCFC systems, medium/low-temperature R 22 systems and for drop-in refrigerants such as 401 A/B, 402 A/B and R 22 mixtures [12.14, 12.18].

#### 12.2.3.4 Fully Synthetic Refrigeration Oils – Alkylbenzenes (AB)

Fully synthetic refrigeration oils based on chemically and thermally highly stable alkylbenzenes. Alkylbenzenes have been used as refrigeration oils for a number of years. Carefully selected and specially treated alkylaromatics are used. A number of complex manufacturing stages ensure that the products are free of difficult-to-dissolve waxy substances and other contaminants such as sulfur. Alkylbenzene-based lubricants display excellent solubility in CFC (HCFC) refrigerants (e.g. R 22, R 502) and mixtures thereof at evaporation temperatures down to –80 °C (e.g. R 22).

ISO VG 46 and 68 alkylbenzenes have proved particularly suitable for use in heavy-duty ammonia compressors with very high outlet temperatures. Compared to mineral oil-based refrigeration oils, alkylbenzenes form minimal amounts of coke and slash less when starting-up.

Alkylbenzenes are also being used in hermetically sealed and semi-sealed compressors. Alkylbenzenes are gaining acceptance for use with drop-in refrigerants such as R 401 A/B, R 402 A/B, R 22 mixtures and together with propane/*iso*-butane. Because of changes in the chemical industry the raw materials for alkylbenzene will be scarce in the future [12.17, 12.18].

#### 12.2.3.5 Fully Synthetic Refrigeration Oils – Polyalphaolefins (PAO)

Polyalphaolefins are recommended for ammonia ($NH_3$) compressors because of their good thermal stability. The formation of oxidation products (coke) is avoided even at high compressor outlet temperatures. Compared to mineral oils and particularly in the case of screw compressors, the use of polyalphaolefins can reduce the amount of oil mist and oil vapor which collects in oil separators. The amount of oil in the refrigerant vapor can also be reduced to a minimum. As a result of their chemical structure, polyalphaolefins display good viscosity–temperature behavior (high VI) and thus good cold flowing characteristics. The low pour point and low viscosity of these products guarantee satisfactory oil return even at evaporation temperatures of –50 °C important for newly developed plate-evaporators.

Polyalphaolefins of the viscosity grade ISO VG 68 are generally used in screw and piston ammonia compressors [12.15].

#### 12.2.3.6 Fully Synthetic Refrigeration Oils – Polyol esters (POE)

Previously used refrigerator lubricants such as mineral oils, alkylbenzenes or polyalphaolefins are not or not sufficiently soluble in the new chlorine-free refrigerant mixtures such as R 134a, R 404 a and R 507. This led to the development of synthetic polyol ester-based refrigeration oils which are soluble in FC and HFC refrigerants according to DIN 8960. The chemical and thermal stability of these products is excellent.

Legislation passed in 1991 progressively banned the use of CFCs in all refrigeration units. Since January 1995, new equipment must be completely CFC-free. R 134a and, particularly, R 22 has replaced CFC R 12. These substitute refrigerants are thus steadily gaining acceptance as is the demand for polyol ester-based, synthetic refrigeration oils. Ester oils are suitable for all refrigerant systems which use R 134a, R 404a, and FC and HFC mixtures. Products with the suitable viscosities

(ISO VG 10-320) are available for industrial and household piston and screw compressors. The manufacturers' viscosity recommendations should be observed.

Similarly to all ester oils, saturated and high-purity polyol ester oils can hydrolyze if they come into contact with water in the compressor (hydrolyse: The splitting of esters with water into partial esters and acidic compounds). It is therefore essential that these products are shielded from water, and moisture in general, during storage and use. Ester oils are ultra-dried and are filled into air-tight metal drums with a water content of <30–100 ppm in a nitrogen atmosphere [12.15, 12.19, 12.20].

The special properties of polyol esters are:

- Excellent solubility in FC and HFC refrigerants
- Avoidance of oil build-up in the condenser/evaporator
- Constant thermal conductivity
- High natural Viscosity Index, good viscosity–temperature behavior and thus adequate lubrication at high temperatures
- Very good thermal and chemical stability even in the presence of refrigerants
- Excellent flowing properties at low temperatures
- Long oil life
- Compatibility with all commonly-used sealing materials such as NBR, HNBR, EPDM and others
- The products are ultra-dried.

Polyol esters are hygroscopic lubricants (i.e. they absorb water) which may hydrolyse over longer periods of time when their water content is >200 ppm. Hydrolysis = splitting the ester into its acidic components [12.15, 12.19].

**Limits for interpretation of used refrigeration oils based on polyolester (POE) RENISO TRITON SE/SEZ.**

| | | ***Fresh Oil*** | ***Used Oil*** |
|---|---|---|---|
| Kinematic viscosity @ 40 °C | $mm^2/s$ | ISO VG +/–10 % | ISO VG +/–10 % |
| Neutralization number (with HFC-refrigerants, e.g. R 134a) | mg KOH/g | < 0.05 | > 0.2 = Increased value!<br>> 0.5 = Change oil! |
| Neutralization number (with CFC/HCFC-refrigerants, e.g. R 22) | | | > 0.07 = Increased value!<br>> 0.1 = Change oil! |
| Water content | ppm | < 30 (< 50) | > 100 = Increased value!<br>> 200 = Change oil! |
| Wear-elements (e.g. Fe, Al, Cu) | ppm | 0 | > 20 = Increased value!<br>> 40 = Change oil! |

**Retrofit/Drop-In – Changing CFC systems to other refrigerants**

The terms Retrofit and Drop-In are two methods of changing CFC systems to substitute refrigerants.

*Retrofit*

Retrofit implies the changing of the refrigerant itself (e.g. R 12) as well as the mineral-based refrigeration oil (the residual mineral oil in the system can be minimized by special oil changing procedures). In addition, a number of system components such as expansion valves, drying filters or seals may have to be modified or exchanged. Retrofit uses long-term substitute refrigerants (e.g. R 134a and ester oils). Retrofit is only really feasible for newer systems because of its relatively high cost [12.14].

*Drop-In*

The Drop-In method involves changing just the refrigerant. The characteristics of the new refrigerant must be so similar to the replaced refrigerant in terms of miscibility with mineral oils or alkylbenzenes that no other system components need to be modified or exchanged. It is recommended that the expansion valve is reset and checked for overheating. The substitute refrigerants are normally zeotropic HCFC refrigerant mixtures such as R 22 mixtures. Drop-In is recommended for older systems because of its relatively low cost.

As a rule, Drop-In refrigerants are mixtures of R 22 and HFC refrigerants. As R 22 is an ozone-depleting refrigerant, this substance is no long-term alternative. In Germany for example, the legislation banning CFCs only allows R 22 to be used in new systems up to the year 2000. For this reason, Drop-In refrigerants are only worthwhile for systems which are reaching the end of their operating lives or for systems in which the oil cannot be changed. In many cases, a change to alkylbenzenes or alkylate-mineral oil mixtures is recommended to. (Note: They can remove chlorine contaminants and other residues from the refrigerant circuit) [12.14, 12.18].

#### 12.2.3.7 **Fully Synthetic Refrigeration Oils – Polyglycols (PAG) for R 134a**

Fully-synthetic, polyglycol-based refrigeration oils for vehicle aircon systems use R 134a. Along with the introduction of R 134a as a substitute for R 12 in the area of vehicle aircon systems, most of such compressors are designed to use polyalkylene glycol (PAG) oils. These polyalkylene glycols are not always compatible and miscible with normal lubricants based on mineral oil, alkylbenzenes or esters. Attention must be paid to this point, especially when topping-up or servicing such systems. Polyglycols are naturally polar and thus miscible with R 134a. These polar characteristics make polyglycols very hygroscopic and this fact must be taken into consideration when handling these specialized lubricants. When filled, the water content of polyglycol oils should be <700–1000 ppm (fresh oil according to DIN 51 503-1 – 300 ppm).

PAG-based refrigeration oils are ultra-dried before use [12.15].

#### 12.2.3.8 **Fully Synthetic Refrigeration Oils – Polyglycols for $NH_3$**

Fully-synthetic, polyglycol-based refrigeration oils (ISO VG 68, ISO VG 100) which are soluble and partially-soluble in ammonia.

In the past, ammonia system lubricants used relatively naphthenic mineral oils, alkylbenzenes or PAO. The problems of oil enrichment and oil deposits in such systems are well known. Polyglycols on the other hand, display excellent solubility/miscibility in ammonia refrigerants. Such good solubility will lead to new system designs in the future including very low temperatures and dry evaporation. The carefully selected synthetic components display excellent viscosity-temperature behavior and high thermal stability. The water content of polyglycol oils must be kept low (about 300–500 ppm). Mixing or contamination with mineral oil must be avoided (Reaction products!) [12.21].

#### 12.2.3.9 **Other Synthetic Fluids**

In the past, polysilicic acid, ester-based synthetic fluids were used for evaporation temperatures below –120 °C. Products based on low-viscosity silicone oils (polydimethylsiloxane; PDMS) are also used. Observe manufacturer's recommendations for such applications alternatives can be low-viscosity polyolester oils.

#### 12.2.3.10 **Refrigeration Oils for $CO_2$**

The natural refrigerant $CO_2$ is gaining acceptance among users. The properties of $CO_2$–oil mixtures are not fully known (both the sub- and trans-critical conditions). In principle, one can say that the solubility of $CO_2$ in POE-esteroil is better than PAG-polyglycol based refrigeration oil. Suitable $CO_2$ refrigeration oils are currently being developed. Apart from solubility, thermal stability is also an important factor (outlet temperatures approx. 160–180 °C) [12.22, 12.23].

Normally used refrigeration oils are listed in Table 12.8.

The refrigerant $CO_2$ is increasingly used in industrial applications, in deep-freezing systems (low-temperature cascades), for climate control, in heat-pump systems, and in container refrigeration units. These units can run transcritically and/or subcritically.

Special synthetic polyolester oils (POEs – ISO VG 55, 85, 130, 170) with selected anti-wear extreme pressure additives (to reduce and prevent wear problems) are used in these so-called industrial applications. The solubility of these polyolester oils in $CO_2$ is controlled (reduction of the viscosity of the POE-$CO_2$ mixture must be checked) and the miscibility is excellent (especially important for very-low-temperature systems). This excellent miscibility (no miscibility gap down to –40 °C) guarantee oil flowability and oil return from the evaporator to the compressor. Transcritical air conditioning systems in buses also run with special polyolesters.

Carbon dioxide will be substituted for R134a in car air-conditioning systems in the near future (phasing out begins in 2011/2012). In these subcritical $CO_2$ car air-conditioning systems selected and "endcaped" polyglycols (PAGs) with anti-wear and extreme pressure additives and will be used to guarantee the lifetime of the compressor under the severe conditions of the $CO_2$ transcritical process. Use of special ester oils is also still under discussion [DKV-Tagungsbericht, 32. Jahrgang, 2005 Arbeitsabteilung II.1 – Band II.1].

#### 12.2.3.11 **Copper Plating**

This phenomenon occurs for the most part when R refrigerants are used. Copper from the refrigeration circuit is dissolved in the lubricating oil and transferred around the system where it is primarily deposited on hot metal surfaces. If the mechanical components concerned run at close tolerances, this can lead to failure

**Tab. 12.8** Overview of normally used refrigerator oils. The viscosity recommendations of the compressor manufacturers must be taken into consideration

| *Compressor type* | *Refrigerant Hydrocarbons*[a] *(e.g. R 290 R 600a)* | *Ammonia*[b] *$NH_3$* | *CFC, HCF, (e.g. R 12, R 22)* | *CFC, HCF*[c] *(e.g. R 134a, R 404a)* | *Drop In (e.g. R 402A, R 403A)* |
|---|---|---|---|---|---|
| Hermetic compressors (e.g. piston compressors) | MO<br>AB<br>ISO VG 7-32 | – | MO<br>AB<br>(MO/AB)<br>ISO VG 15-46 | POE<br>ISO VG 10-32 | MO/AB<br>ISO VG 32 |
| Open piston compressors | MO<br>AB<br>PAO<br>ISO VG 46-100 | MO<br>AB<br>PAO, PAG<br>ISO VG 32-68 | MO<br>AB<br>MO/AB<br>ISO VG 32-100 | POE<br>ISO VG 32-68 | MO/AB<br>ISO VG 32-68 |
| Semi-hermetic compressors | MO<br>AB<br>PAO<br>ISO VG 46-100 | – | MO<br>AB<br>MO/AB<br>ISO VG 32-100 | POE<br>ISO VG 32-68 | MO/AB<br>ISO VG 32-68 |
| Scroll compressors | MO<br>AB<br>ISO VG 46-100 | – | MO<br>AB<br>ISO VG 32-100 | POE<br>ISO VG 32-68 | MO/AB<br>ISO VG 32-68 |
| Screw compressors | MO<br>AB<br>PAO, PAG<br>ISO VG 68-220 | MO<br>AB<br>PAO, PAG<br>ISO VG 32-68 | MO<br>AB<br>ISO VG 68-220 | POE<br>ISO VG 100-320 | MO/AB<br>ISO VG 68 |
| Turbo compressors | MO<br>PAO, PAG<br>ISO VG 68-100 | –[d] | MO<br>ISO VG 68-100 | POE<br>ISO VG 68-150 | MO/AB<br>ISO VG 68 |

MO = mineral oil; AB = alkylbenzene;
MO/AB = mineral oil–alkylbenzene mixture;
PAO = polyalphaolefin; PAG = polyalkyleneglycol;
POE = polyol ester.

a) PAG-lubricants are partly soluble with hydrocarbons (low viscosity reduction); MO, AB and PAO are highly soluble with hydrocarbons (high viscosity reduction).
b) MO, AB and PAO are not soluble with ammonia, PAG is (partly) soluble with ammonia, ISO VG 68 is used in piston compressors, up to ISO VG 220 is used in screw compressors, please use PAG compatible components, PAG is hygroscopic
c) PAG-lubricants are used in R 134a air conditioning systems of cars and trucks (PAG 46, PAG 100); POE and PAG lubricants are hygroscopic
d) Normally oil-free

(bearings, slip rings). Copper plating is not a subject specifically connected with the oil, although certain properties of the oil may assist its occurrence. It is reasonable to assume that the oil resin and sulfur contents of a good refrigeration oil are below the threshold above which these values could begin to encourage copper plating.

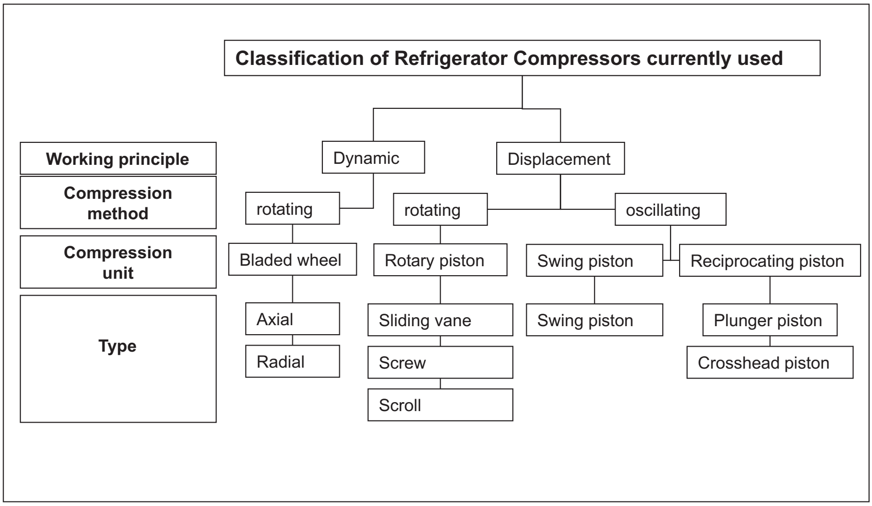


**Fig. 12.6** Classification of refrigeration compressors according to their construction.

Factors likely to promote copper plating are insufficient oil–refrigerant stability, moisture in the system (high water content in the oil), impurities of various kinds, oil acidification by the refrigerant and last by not least, oil aging due to contact with oxygen [12.10, 12.11].

### 12.2.4
### Types of Compressor

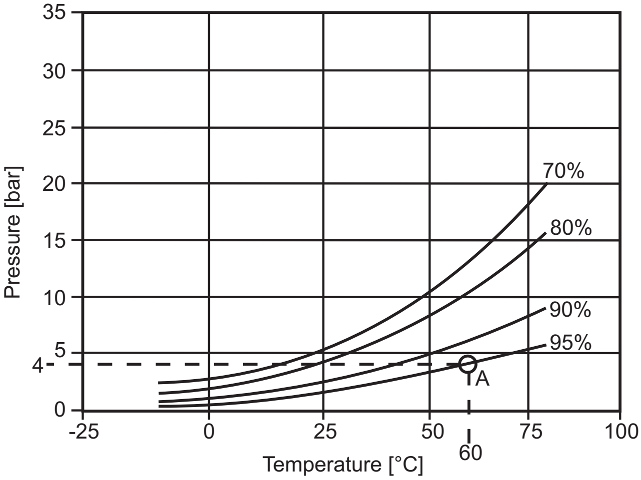
Compressors, as the core element of refrigerant systems, pump gaseous refrigerant around the circuit, and compress the vaporized refrigerant to the liquefaction pressure required to release heat.

Figure 12.6 classifies refrigeration compressors according to their construction. Compressors are divided into two groups: The displacement types which discontinuously compress the refrigerant into increasingly small spaces and the dynamic types which continuously accelerate the refrigerant to increase its pressure [12.24].

### 12.2.5
### Viscosity Selection

#### 12.2.5.1 General Overview

The guidelines for the selection of refrigerator compressor lubricants are much the same as those applied to engineering in general, i.e. fast running machines permit the use of lower viscosities than slow running machinery. High bearing loads require higher viscosities than low bearing loads. In addition, refrigerator compressors require much lower viscosities than those calculated from the hydrodynamic lubrication theory. This fact has been proved by years of practical experience and by the elasto–hydrodynamic theory of lubrication. The influence of refrigerants on the



All %-figures represent the oil mass in the refrigerant.

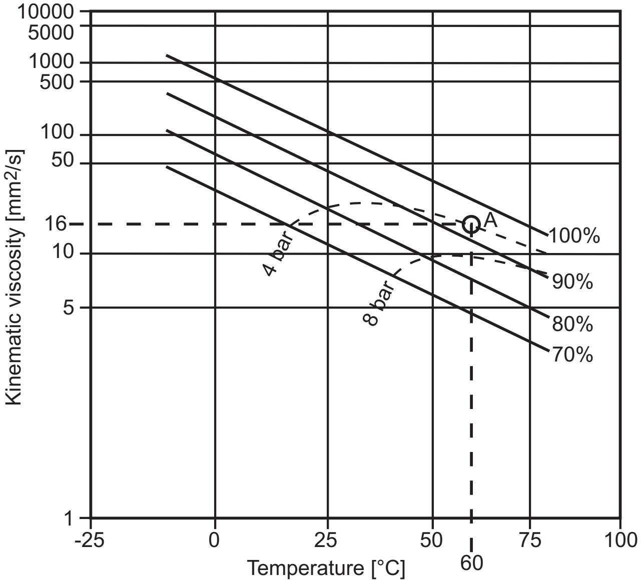
**Fig. 12.7** Mixture concentration in relationship to temperature and pressure (RENSIO Triton SE 55 – R 134a), at 4 bar and 60 °C ~ 5 % R 134a is dissolved in the oil.

operational viscosity of a refrigeration oil must be considered when a selection is made. In the case of piston compressors, the viscosity of the oil depends on the crankcase pressure and in the case of screw compressors, on the outlet pressure (pressure in the oil separator).

In the past, many refrigerator systems were operated with chlorinated refrigerants in order to provide some reliability reserves. Chlorine compounds are excellent EP additives which protect against wear. This is why previously-used refrigeration oils used with CFCs were considered to contain 'anti-wear' agents when the refrigerant was dissolved in the oil. Since the introduction of chlorine-free refrigerants, this function must be performed by the refrigeration oil and/or additives (e.g. isobutane applications).

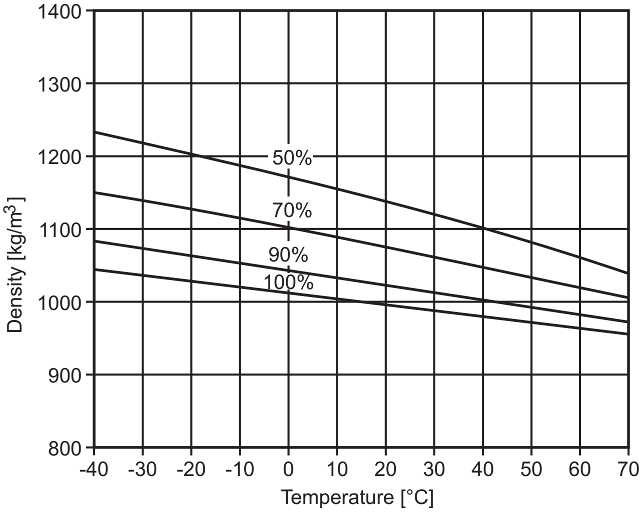
Some of the important inter-relationships to be considered when selecting the correct lubricating oil for a refrigerator compressor are discussed in the following. Reference should also be made to refrigerant–oil data sheets which contain important information for both compressor manufacturers and users [12.25].

The most important parameter for determining the lubricity of oils or oil–refrigerant mixtures is viscosity. The viscosity of oil–refrigerant mixtures should be viewed as the viscosity of a pure oil when bearing load calculations are being made. This applies to the hydrodynamic lubrication of cylindrical plain bearings. In the field of piston and screw lubrication, additional factors are the mixture's boundary friction phenomena. In general, reciprocating piston compressors are lubricated with oils ISO VG 32, 46, and 68 and screw compressors are lubricated with oils ISO VG 150, 170, 220, and 320 – depending on the refrigerant, temperature, pressure and the solubility of the refrigerant in the oil.



All %-figures represent the oil mass in the refrigerant.

**Fig. 12.8** Mixture viscosity in relationship to temperature, pressure, and refrigerant concentration (RENSIO Triton SE 55 – R 134a), at 4 bar and 60 °C ~ 5 % R 14a is dissolved, the viscosity of the POE/R 134a mixture is 16 $mm^2 s^{-1}$.



All %-figures represent the oil mass in the refrigerant.

**Fig. 12.9** Mixture density in relationship to temperature and refrigerant concentration (RENSIO Triton SE 55 – R 134a), POE lower density compared with R 134a.

### 12.2.5.2 Mixture Concentration in Relationship to Temperature and Pressure (RENISO Triton SE 55 – R 134a)

Figure 12.7 shows how much refrigerant is dissolved in a refrigeration oil when saturated at a defined operating condition (pressure, temperature). As saturation is time-related, the refrigerant concentration shown by the diagram is, as a rule, greater than the actual value. It can be viewed as the maximum concentration at any given operating condition. The viscosity which can be taken from the mixture concentration adds a safety margin to any bearing loading calculations.

In this diagram, the refrigerant concentration can be allocated to a point with a defined pressure and a defined temperature [12.15].

### 12.2.5.3 Mixture Viscosity in Relationship to Temperature, Pressure and Refrigerant Concentration (RENISO Triton SE 55 – R 134a)

The precise refrigerant concentration in a system is given at a defined pressure and temperature as shown in Fig. 12.7. Figure 12.8 can be used to read-off the kinematic viscosity of the oil–refrigerant mixture at a defined temperature, defined pressure and defined refrigerant concentration from the left scale (unit of kinematic viscosity is $10^{-6}$ $m^2$ $s^{-1}$ = 1 $mm^2$ $s^{-1}$). The diagram shows the viscosity in relationship to the temperature of the oil–refrigerant mixture at various concentrations.

If the viscosity of the mixture (again only valid when in a state of equilibrium) is required and the refrigerant concentration derived from pressure and temperature is unknown, refer to the diagram. This results from the competing influences of viscosity gains when the temperature of the oil falls and viscosity drops caused by greater quantities of dissolved refrigerant at lower temperatures . This fact is of considerable importance to the design and operation of a refrigerant compressor. Care should therefore be taken that the oil does not reach its maximum viscosity at problematic points in the oil return circuit (e.g. upward flows, evaporators). It is also important that the conditions in the compressor crankcase do not approach those indicated by the falling left-hand part of the viscosity–temperature graph because the smallest temperature fluctuations can have a significant effect on viscosity in such conditions [12.15].

### 12.2.5.4 Mixture Density in Relationship to Temperature and Refrigerant Concentration (RENISO Triton SE 55 – R 134a; Fig. 12.9)

The density of an oil refrigerant mixture depends on the density–temperature behavior of the oil and the refrigerant (Fig. 12.9).

### 12.2.5.5 Miscibility Gap, Solubility Threshold (RENISO Triton Series with R 134a, Fig. 12.10)

R-refrigerants are among the most oil-soluble refrigerants. However, not all are miscible in refrigeration oils at all temperatures and in all concentrations. If, for example, one cools a fully dissolved oil–refrigerant mixture, a point will be reached when the fully dissolved mixture separates into two fluid phases. This area of partial solubility is called the miscibility gap. The miscibility gap span depends on the type of refrigerant and also to a large extent on the type of refrigeration oil. Refrigerant

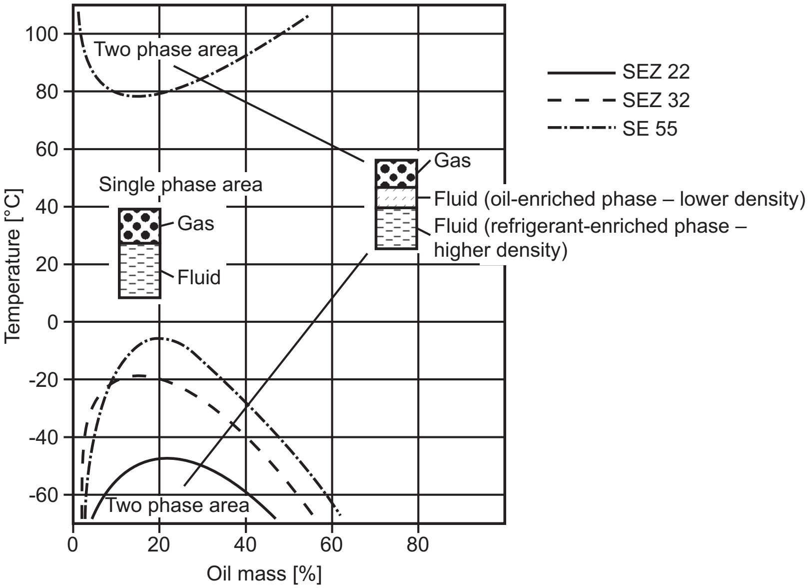


**Fig. 12.10** Miscibility gap, solubility threshold (RENISO Triton series – R 134a).

solubility is defined statically in E DIN 51 514. For normal applications, miscibility gap (with alkylbenzenes) is not a problem for a series of refrigerants such as R 22. Some other refrigerants display pronounced miscibility thresholds. The miscibility gap is of great importance to the refrigerant circuit. If the oil–refrigerant ratio is within the miscibility gap, problems can occur caused by oil-enriched fluid phases depositing in collectors, condensers, evaporators and the crankcase. Flooded evaporators require the largest possible quantity of refrigerant to be dissolved at evaporating temperatures without the phases separating. Figure 12.10 shows various examples of solubility thresholds.

### 12.2.6
### Summary

The selection of an optimum refrigeration oil depends on the specifications of the compressor, the system as a whole and the refrigerant itself. Particularly important factors are the lubricity of the refrigeration oil and any interactions with the refrigerant, evaporation behavior as well as solubility and mixture behavior. Apart from traditional mineral oil-based refrigeration oils, polyol esters for chlorine-free refrigerants represent the next the most important group. Polyglycols are used in R 134a vehicle air-conditioning systems. Polyalphaolefins and partly polyglycol-based soluble oils are increasingly being used for ammonia systems. $CO_2$ refrigerants will gain acceptance in the future for non-stationary applications and will replace R 134a in many areas. In industrial $CO_2$ systems special synthetic AW/EP polyolester oils are in use. In car air conditioning systems special synthetic polyglycols will be used.

# 13

# Turbine Oils

*Wolfgang Bock*

## 13.1

## Introduction

Steam turbines have existed for more than 90 years. Steam turbines are motors with rotating elements which transform the energy of steam into mechanical work in one or more stages. A steam turbine is normally connected to a driven machine, sometimes through a gearbox [13.1, 13.2]. Steam temperatures can reach about 560 °C plus and pressures about 130–240 bar. The greater efficiency made possible by higher steam temperatures and pressures were fundamental factors in the further development of steam turbines. However, higher temperatures and pressures also increased the demands made on the lubricants used. The turbine oils without additives of the early years were unable to cope with these increased demands. Oils with additives have been used in steam turbines for about 50 years now [13.2, 13.3]. These turbine oils which contain aging inhibitors and corrosion protection agents offer outstanding reliability in practice if certain basic rules are observed. Modern turbine oils also contain mild EP/AW additives which protect the component from wear. Steam turbines are used by power stations to drive electrical power generators. In conventional power stations, their output is about 700–1000 MW while in nuclear plants, this figure is about 1300 MW [13.2, 13.3].

## 13.2

## Demands on Turbine Oils – Characteristics

The demands on turbine oils are defined by the turbines themselves and their specific operating conditions. The oil in the lubricating and control circuits of steam and gas turbines has to fulfil the objectives [13.3, 13.4]:

- hydrodynamic lubrication of all bearings and the lubrication of gearboxes;
- heat dissipation
- functional fluid for control and safety circuits
- avoidance of friction and wear on gear tooth flanks in turbine gearboxes and when the turbine is spooled-up.

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Apart from these mechanical–dynamic requirements, the following physical–chemical specifications also have to be fulfilled by turbine oils [13.3, 13.4]:

- aging stability for long operating periods
- hydrolytic stability (especially of the additives used)
- corrosion protection even if water, steam and/or condensation is present
- reliable separation of water (vapor and separation of condensed water)
- rapid air release and low foaming
- good filterability and purity.

These stringent demands on steam and gas turbine oils are met with carefully selected base oils and the inclusion of special additives.

## 13.3
## Formulation of Turbine Oils

Turbine oils used today contain special paraffinic base oils with good viscosity–temperature characteristics as well as antioxidants and corrosion inhibitors. If geared turbines require a degree of load-bearing capacity (e.g. failure load stage FZG 8 min. DIN 51 354-2), some mild EP additives are included.

These days, turbine base oils are created exclusively by extraction and hydration. Refining and subsequent selective high-pressure hydrating significantly determine and influence turbine oil characteristics such as oxidation stability, water separation, air release and foaming. This applies in particular to water separation and air release because these feature cannot be subsequently improved with additives. Steam turbine oils are generally made of special paraffinic base oil cuts [13.2–13.4].

Phenolic antioxidants combined with aminic antioxidants (synergies) are added to turbine oils to improve their oxidation stability [13.3]. To improve corrosion protection, non-emulsifiable corrosion protection agents and nonferrous metal passivators are used. These are not negatively affected by water or steam contamination during operation and remain suspended. If standard turbine oils are used in geared turbines, mild, long life, temperature and oxidation stable EP/AW additives (organo-phosphorus and/or sulfur compounds are included. Furthermore, small quantities of silicone-free anti-foamers and pourpoint depressors are also used in turbine oils [13.3–13.5].

Great care must be taken to ensure that the defoamer used is absolutely free of silicone. Moreover, these additives must not detrimentally affect the air release (very sensitive) of the oil. All additives should be free of ash (e.g. zinc-free).The purity of a turbine oil according to ISO 4406 should be 15/12 in the tank [13.6–13.9]. No circuits, wires, cables or insulation containing silicone should come into contact with turbine oils (observe when manufacturing and during application).

## 13.4
## Turbine Lubricants – Specifications

Special paraffinic mineral oils with additives are normally used for gas and steam turbine lubricants. These serve to protect all shaft bearings in the turbine and generator as well as the gearbox in corresponding configurations. They can also be used as hydraulic fluids in control and safety systems. Fire-resistant, synthetic Type HFD-R fluids [13.4] are normally used in hydraulic systems which operate at pressures around 40 bar, if there are separate lubricating oil and control oil circuits (twin circuit systems).

DIN 51 515 titled Lubricants and Governor Fluids for Turbines is revised in 2001 (Part 1-L-TD formal service, specifications) and new, so-called "high-temperature turbine oils" are defined in DIN 51 515 – Part 2 (Part 2-L-TG lubricants and governor fluids for turbines – for higher temperature service-specifications). Following ISO 6743, Part 5, Family T (Turbines) – Classification of Turbine Oils [13.10], the latest draft of DIN 51 515, dated 2001/2004, classifies turbine oils as in Table 13.1 [13.14, 13.15, 13.16].

**Tab. 13.1** DIN 51 515 classification of turbine oils, Draft 1999

| | *'Normal' turbine oils – Steam turbine oils* | *'High-temperature' turbine oils* |
|---|---|---|
| Without EP | DIN 51 515-1 | DIN 51 515-2 |
| With EP<br>(FZG, Load Stage min. 8) | DIN 51 515-1<br>Annex A | DIN 51 515-2<br>Annex A |

The demands made by DIN 51 515-1 – Steam Turbine Oils [13.10] and DIN 51 515-2 – High-Temperature Turbine Oils [13.10] are shown in Tables 13.2 and 13.3.

ISO 6743-5 classifies turbine lubricants according to whether they are used in steam or gas turbines and if they contain EP agents (Table 13.4) [13.11, 13.12].

The specification according to ISO 6743-5 in conjungtion with ISO CD 8068 "Lubricants, Industrial Oils and related product (class L) – Family T (Turbine oils, ISO-L-T are still under discussion (2003).

**Tab. 13.2** Requirements of steam turbine oils, DIN 51 515 Part 1, June 2001 – LTD – for normal service.

| ***Test*** | ***Limits*** | | | | ***Testing according to[2]*** | ***Comparable ISO* Standards*** |
|---|---|---|---|---|---|---|
| Lubricating oil group | TD 32 | TD 46 | TD 68 | TD 100 | | |
| ISO viscosity grade[1] | ISO VG 32 | ISO VG 46 | ISO VG 68 | ISO VG 100 | DIN 51 519 | ISO 3448 |
| Kinematic viscosity at 40 °C in $mm^2\ s^{-1}$ min.<br>max. | 28.8<br>35.2 | 41.4<br>50.6 | 61.2<br>74.8 | 90.0<br>110 | DIN 51 562-1 or<br>DIN 51 562-2 or<br>DIN EN ISO 3104 | ISO 3104 |
| Flash point (COC), °C min. | 160 | 185 | 205 | 215 | DIN ISO 2592 | ISO 2592 |
| Air release property[4] at 50 °C, minutes max. | 5 | 5 | 6 | no value specified | DIN 51 381 | – |
| Density at 15 °C, g $ml^{-1}$ max. | To be specified by supplier | | | | DIN 51 757 or<br>DIN EN ISO 3675 | ISO 3675 |
| Pour point, °C | $\leq -6$ | $\leq -6$ | $\leq -6$ | $\leq -6$ | DIN ISO 3016 | ISO 3016 |
| Neutralization number mg KOH $g^{-1}$ | To be specified by supplier | | | | DIN 51 558 Part 1 | ISO 6618 |
| Ash (oxide ash) % mass | To be specified by supplier | | | | DIN EN ISO 6245 | ISO 6245 |
| Water content, mg $kg^{-1}$ max. | 150 | | | | DIN 51 777-1 | ISO/DIS 12937 |
| Cleanliness level min. | 20/17/14 | | | | DIN ISO 5884 with<br>DIN ISO 4406 | ISO 5884 with ISO 4406 |
| Water separation ability (after steam treatment), (seconds) max. | 300 | 300 | 300 | 300 | DIN 51 589 Part 1 | – |
| Copper corrosion max. corrosiveness (3 h at 100 °C) | 2-100 A 3 | | | | DIN EN ISO 2160 | ISO 2160 |

**Tab. 13.2** Continued.

| ***Test*** | ***Limits*** | | | | ***Testing according to*** 2) | ***Comparable ISO* Standards*** |
|---|---|---|---|---|---|---|
| Corrosion protection against steel corrosiveness max. | No rust 0 - A | | | | DIN 51 585 | ISO 7120 |
| Aging behavior 3) (TOST) Time in hours to reach delta NZ of 2.0 mg KOH $g^{-1}$ | 2000 | 2000 | 1500 | 1000 | DIN 51 587 | ISO 4263 |
| Foam: | | | | | | |
| Seq. I at 24 °C max. ml | 450/0 | | | | – | ISO 6247 |
| Seq. II at 93 °C max. ml | 100/0 | | | | | |
| Seq. III at 24 °C after 93 °C max. ml | 450/0 | | | | | |

* International Organization for Standardization

1) Middle point viscosity at 40 °C in $mm^2\ s^{-1}$

2) The oil sample has to be stored without any contact to light before doing the test

3) The aging behavior test has to be done as a type test procedure because of the long testing time

4) The test at a temperature of 25 °C has to be specified by the supplier when the customer needs values at low temperatures

**Annex A (normative)** – for EP turbine oils
If the turbine oil supplies also a turbine gear set, the oil has to reach a load stage of min. 8 according to DIN 51 345, part 1 and part 2 (FZG).

**Tab. 13.3** Requirements of high-temperature turbine oils, DIN 51 515 Part 2, November 2004, L-TG for higher temperatur service.

| *Lubricating oil group* | | *Limits* | | *Testing according to*[2] | *Comparable ISO* Standards* |
|---|---|---|---|---|---|
| | | *TG 32* | *TG 46* | | |
| ISO viscosity grade[1] | | ISO VG 32 | ISO VG 46 | DIN 51 519 | ISO 3448 |
| Kinematic viscosity at 40 °C in mm$^2$ s$^{-1}$ | min.<br>max. | 28.8<br>35.2 | 41.4<br>50.6 | DIN 51 550 in conjunction with DIN 51561 or DIN 51562-1 | ISO 3104 |
| Flash point (COC), °C | min. | 160 | 185 | DIN ISO 2592 | ISO 2592 |
| Air release property[4] at 50 °C, minutes | max. | 5 | 5 | DIN 51 381 | – |
| Density at 15 °C, g ml$^{-1}$ | max. | To be specified by supplier | | DIN 51 757 | ISO 3675 |
| Pour point, °C | | ≤–6 | | DIN ISO 3016 | ISO 3016 |
| Neutralization number | mg KOH g$^{-1}$ | To be specified by supplier | | DIN 51 558-1 | ISO/DIS 6618 |
| Ash (oxide ash) | % mass | To be specified by supplier | | DIN EN 7 | ISO 6245 |
| Water content, mg kg$^{-1}$ | max. | 150 | | DIN 51777-1 | ISO/DIS 12937 |
| Cleanliness level | min. | 20/17/14 | | DIN ISO 5884 with ISO 4406 | ISO 5884 with ISO 4406 |
| Foam: | | | | – | ISO 6247 |
| Seq. I at 24 °C | max. ml | 450/0 | | | |
| Seq. II at 93 °C | max. ml | 100/0 | | | |
| Seq. III at 24 °C after 93 °C | max. ml | 450/0 | | | |
| Demulsibility, minutes | | To be specified by supplier | | DIN 51 599 | ASTM-D 1401 |
| Water separation ability (after steam treatment), seconds | max. | 300 | 300 | DIN 51 589 Part 1 | – |

**Tab. 13.3** Continued.

| ***Lubricating oil group*** | ***Limits*** | | ***Testing according to2)*** | ***Comparable ISO* Standards*** |
|---|---|---|---|---|
| | ***TG 32*** | ***TG 46*** | | |
| Copper corrosion corrosiveness max. | 2–125 A 3 | | DIN 51 759 | ISO 2160 |
| Corrosion protection against steel corrosiveness max. | 0–A | | DIN 51 585 | ISO/DIS 7120 |
| Aging behavior3)<br>Time in hours to reach delta NZ of 2.0 mg KOH $g^{-1}$ min. | 3.000 | | DIN 51 587 | ISO/DIS 4263 |
| RBOT, minutes minutes<br>Modified RBOT, minutes % of time in unmodified test | ≥ 800**<br>≥ 85 % | | – | ASTM-D 2272 |

* International Organization for Standardization

** GE recommends only 450 min

1) Middle point viscosity at 40 °C in $mm^2 s^{-1}$

2) The oil sample has to be stored without any contact to light before doing the test

3) The aging behavior test has to be done as a type test procedure because of the long testing time

4) The test at a temperature of 25 °C has to be specified by the supplier when the customer needs values at low temperatures

**Annex A (normative)** – for EP turbine oils
If the turbine oil supplies also a turbine gear set, the oil has to reach a load stage of min. 8 according to DIN 51 345, part 1 and part 2 (FZG).

**Tab. 13.4** ISO 6743-5 classification of turbine lubricants, in conjunctio with ISO/CD 8068.

| | ***Normal turbine oils*** | ***High-temperature turbine oils*** |
|---|---|---|
| Without EP | ISO-L–TSA (steam) | ISO-L–TGB (gas) |
| | ISO-L–TGA (gas) | ISI-L–TGSB (= TGA + TGB performance) |
| With EP | ISO-L–TSE (steam) | ISO-L–TGF |
| | ISO-L–TGE (gas) | ISO-L–TGSE |

Synthetic fluids such as PAO and phosphoric acid ester are also listed in ISO CD 8068 dated 2003 (see Table 13.5) [13.11, 13.12].

Different engineering designs make different demands on the lubricant used. Table 13.6 [13.6–13.9] shows the principal demands made by the world's leading turbine manufacturers. The main differences between these demands relate to the aging and/or oxidation tests and AW/EP performance.

## 13.5
## Turbine Oil Circuits

The oil circuits play an especially important role in the lubrication of power station turbines. Steam turbines are normally fitted with pressurized oil circuits and separate lubricating and control circuits as well as separate lubricating and control oil tanks are used.

In normal operating conditions, the turbine shaft-driven main oil pump draws oil from the tank into the control and bearing supply circuits. Pressure in control circuits is normally between 10 and 40 bar (separate header pump: turbine shaft header pressure: ca. 100–200 bar) [13.1–13.4]. Oil tank temperatures range from 40 to 60 °C. The velocity of the oil in the feed circuit is about 1.5 to 4.5 m $s^{-1}$ (about 0.5 m $s^{-1}$ in return circuits) [13.3]. Cooled and passing through reduction valves, the oil reaches the turbine, generator and possible gearbox bearings at a pressure of ca. 1–3 bar. The individual oil feeds return to the oil tank at atmospheric pressure. The main turbine and generator shaft bearings are mostly white-metal shell bearings. Axial loads are normally absorbed by trunnion bearings. The lubricating oil circuit of a gas turbine is largely similar to that of a steam turbine. However, gas turbines sometimes use roller bearings as well as plain bearings [13.1, 13.2, 13.4].

Larger oil circuits are fitted with centrifugal by-pass filtering systems. These ensure that the finest of contaminants are removed along with aging by-products and sludge. Depending on the size of the turbine, the oil in by-pass systems with separate pumps should pass through the filters about once every one to five hours. The oil should be removed from the lowest point in the tank and be filtered directly before the oil is returned to the tank. If the oil is taken from the main oil stream, the flow rate should be reduced to about 2–3 % of the main pump's flow capacity. The following equipment is often used: Oil centrifuges, fine-paper filters, cellulose cartridge fine-filters and filter units with separators. An additional magnetic filter is also highly recommended. Sometimes by-pass and main flow filters are fitted with

**Tab. 13.5** Classification of lubricants for turbines, ISO 6743-5, Draft 1999 in conjunction with ISO/CD 8068.

| ***Part 5: Family T (Turbines): Table–Classification of lubricants for turbines*** | | | |
|---|---|---|---|
| ***General application*** | ***Composition and properties*** | ***Symbol ISO-L*** | ***Typical application*** |
| 1) Steam turbines, directly coupled or geared to the load, normal service | Refined mineral oils with suitable antioxidants and corrosion inhibitors | TSA | Power generation and industrial drives and their associated control systems, marine drives, where improved load carrying capacity is not required for the gearing |
| 2) Gas turbines, directly coupled or geared to the load, normal service | | TGA | |
| 3) Steam turbines, directly coupled or geared to the load, high load-carrying capacity | Refined mineral oils with suitable antioxidants and corrosion inhibitors with additional extreme-pressure performance to lubricate gear systems. | TSE | Power generation and industrial drives and marine geared drives and their associated control systems where the gearing requires improved load-carrying capacity. |
| 4) Gas turbines, directly coupled or geared to the load, high load-carrying capacity | | TGE | |
| 5) Gas turbines, directly coupled or geared to the load, higher-temperature service | Refined mineral oils with suitable antioxidants and corrosion inhibitors–for higher temperatures. | TGB<br>TGSB (= TSA + TGB) | Power generation and industrial drives and their associated control systems where high temperature resistance is required due to hot spot temperatures |
| 6) Other lubricants (according to ISO 6749-5 and ISO/CD 8068)<br>a) TSC–Synthetic steam turbine fluids with no specific fire-resistant properties (e.g. PAO) (see also TGCH)<br>b) TSD–Synthetic steam turbine fluids based on phosphate esters with fire-resistant properties (alkyl phosphate ester)<br>c) TGC–Synthetic gas turbine fluids with no specific fire-resistant properties (e.g. PAO) (see also TGCH)<br>d) TGD–Synthetic gas turbine fluids based on phosphate esters with fire-resistant properties (alkyl phosphate ester)<br>e) TCD–Synthetic fluids for control systems based on phosphate esters with fire-resistant properties | | | |

**Tab. 13.6** Principal demands of turbine manufacturers.

| | ***Siemens TLV 901304 (July 1999)*** | ***e.g. General Electric GEK 101941 A (Nov. 1999)*** | ***e.g. General Electric GEK 32568 E (May 1999)*** | ***Alstom HTGD 90117 V0001U 7/2005*** | ***Alstom HTGD 90117 V0001U 7/2005*** | ***Testing according to DIN ISO*** | ***Testing according to ASTM*** |
|---|---|---|---|---|---|---|---|
| Lubricating oil group | Steam and gas turbine oils1) | Gas turbine oils with EP/AW additives with bearing ambients above 260 °C2) | Gas turbine oils with bearing ambients above 260 °C3) | Steam and gas turbine oils (with and without EP/AW additives), ISO VG 32/46 4) | Steam and gas turbine oils (with and without EP/AW additives), ISO VG 68 4) | | |
| Kinematic viscosity at 40 °C, (mm$^2$ s$^{-1}$)<br>ISO VG 32<br>ISO VG 46 | <br>28.8–35.2<br>41.4–50.6 | <br>28.8–35.2 | <br>28.8–35.2 | <br>VG 32: ± 10 %<br>VG 46: ± 10 % | <br>VG 68: ± 10 % | DIN 51 562-1 | ASTM-D 445 |
| Gravity (°API) | – | 29–33.5 | 29–33.5 | – | – | | ASTM-D 287 |
| Air release property at 50 °C, (minutes) | ≤ 4 | 5 (max.) | 5 (max.) | ≤ 4 | ≤ 7 | DIN 51 381 | ASTM-D 3427 |
| Neutralization Number, (mg KOH g$^{-1}$)<br>without EP/AW additives<br>with EP/AW additives | <br>≤ 0.2<br>≤ 0.3 | <br>–<br>0.2 max. | <br>0.2 max.<br>– | <br>0.2 max.<br>0.3 max. | <br>0.2 max.<br>0.3 max. | DIN 51 558-1 | ASTM-D 974 |
| Water content, (mg kg$^{-1}$) | ≤ 100 | – | – | – | – | DIN 51 777-1 | ASTM-D 1744 |
| Foaming at 25 °C<br>Tendency, (ml)<br>Stability, (seconds) | <br>≤ 400 (Seq. 1)<br>≤ 450 | 50/0 max.<br>50/0 max.<br>50/0 max. | 50/0 max.<br>50/0 max.<br>50/0 max. | 300/0<br>50/0<br>300/0 | 300/0<br>50/0<br>300/0 | – | ASTM-D 892 |
| Water separability, (seconds) | ≤ 300 | | | ≤ 300 | ≤ 300 | DIN 51 589-1 | – |

**Tab. 13.6** Continued.

| | *Siemens TLV 901304 (July 1999)* | *e.g. General Electric GEK 101941 A (Nov. 1999)* | *e.g. General Electric GEK 32568 E (May 1999)* | *Alstom HTGD 90117 V0001U 7/2005* | *Alstom HTGD 90117 V0001U 7/2005* | *Testing according to DIN ISO* | *Testing according to ASTM* |
|---|---|---|---|---|---|---|---|
| Demulsibility, (minutes) | ≤ 20 | | | ≤ 30 | ≤ 30 | DIN 51 599 | ASTM-D 1401 |
| Density at 15 °C, (kg $m^{-3}$) | ≤ 900 | | | ≤ 880 | ≤ 900 | DIN 51 757 | ASTM-D 1298 |
| Flash point<br>ISO VG 32, (°C)<br>ISO VG 46, (°C) | > 160<br>> 185 | 215 (min.) | 215 (min.) | VG 32 and 46:<br>≥ 200 | VG 68:<br>≥ 205 | DIN ISO 2592 | ASTM-D 92 |
| Pourpoint, (°C) | ≤ –6 | –12 (max.) | –12 (max.) | < –9 | < –6 | ISO 3016 | ASTM-D 97 |
| Particle distribution, (ISO class) | ≤ 17/14 | | | 18/15 | 18/15 | ISO 4406 | – |
| Color | ≤ 2 | max. 2.0 | max. 2.0 | – | – | DIN ISO 2049 | ASTM-D 1500 |
| Copper corrosion, corrosiveness | ≤ 2–100 A 3 | 1 B (max.) | 1 B (max.) | ≤ 2–100 A 3 | ≤ 2–100 A 3 | DIN EN ISO 2160 | ASTM-D 130 |
| Corrosion protection against steel, corrosiveness | ≤ 0–B | 0–B (pass) | 0–B (pass) | O-B (pass) | | DIN 51 585 | ASTM-D 665 |
| Aging behavior, Increase of Neutralization Number in mg KOH $g^{-1}$ after h, TOST test | ≤ 2.0<br>(after 2.500 h) | ≤ 2.0<br>(after 3.000 h) | ≤ 2.0<br>(after min. 3.000 h) | < 1<br>(after 2.000 h)<br>* | < 1<br>(after 2.000 h)<br>* | DIN 51 587 | ASTM-D 943 |
| Additional requirements on turbine oils for use in gearboxes FZG-Test: A/8,3/90 Failure load stage | ≥ 8 | ≥ 8 | – | 8 | 8 | DIN 51 354 | ASTM-D 1947 |
| Carbon residue ramsbottom, (%) | | 0.1 % (max.)<br>(or equivalent) | 0.1 % (max.)<br>(or equivalent) | – | – | – | ASTM-D 524 |
| Oxidation stability by rotating bomb, (minutes) | | 500 (min.) | 500 (min.) | > 300 min. | > 300 min. | – | ASTM-D 2272 |

**Tab. 13.6** Continued.

| | ***Siemens TLV 901304 (July 1999)*** | ***e.g. General Electric GEK 101941 A (Nov. 1999)*** | ***e.g. General Electric GEK 32568 E (May 1999)*** | ***Alstom HTGD 90117 V0001U 7/2005*** | ***Alstom HTGD 90117 V0001U 7/2005*** | ***Testing according to DIN ISO*** | ***Testing according to ASTM*** |
|---|---|---|---|---|---|---|---|
| Oxidation stability by rotating bomb (modified)<br>N2-blown RBOT | | 85 % (min.) of time in unmodified test | 85 % (min.) of time in unmodified test | – | – | – | ASTM-D 2272 |
| Viscosity Index (VI) | | 95 (min.) | 95 (min.) | ≥ 90 | ≥ 90 | – | ASTM-D 2270 |
| Atomic emission spectroscopy<br>Zinc content | | < 5 ppm | | ≤ 5 ppm | ≤ 5 ppm | – | ASTM-D 4951 |
| Filtrability | | | | Stage I min 93 %<br>Stage II min 85 % | | ISO 13 357-2 | |

* Neutralization Number $< 1.8$ mg KOH $g^{-1}$, sludge $< 0.4$ % according to DP 7624

Base Oils:

1) Mineral oils or synthetic oils with additives to improve corrosion protection and aging stability (additionally EP/AW additives in the case of gearbox lubrication).
2) Petroleum lubricating oil
   – Synthetic Hydrocarbons with greater high temperature, oxidation stability and R&O inhibitors, EP/AW additives
3) Petroleum lubricating oil
   – Synthetic Hydrocarbons with greater high temperature, oxidation stability and R&O inhibitors
4) Refined mineral oils with additives mainly aging inhibitors and corrosion inhibitors (no EP/AW additives).

*Other important specifications (examples):*

Westinghouse: I.L. 1250-5312 – Steam Turbines
21 T 0591 – Gas Turbines
Solar: ES 9-224 – Gas Turbines

5) L.S. = Load Stage

coolers to reduce the temperature of the filtered oil. It must also be possible to remove the oil from the tank with a mobile filter unit or centrifuge if water, steam or other contaminants enter the system. For this, the lowest point of the oil tank is normally fitted with a corresponding connector which can also be used to draw oil samples [13.3, 13.4].

Oil aging is also influenced by the frequency with which the oil is pumped through the circuit. If the oil is pumped too fast, excessive amounts of air are either dispersed or dissolved (problem: cavitation in bearings, premature aging etc.). Oil tank foaming can also occur but this generally collapses rapidly. Engineering design measures can positively influence air release and tank foaming. These include oil tanks with larger surface areas and larger return circuit pipe cross-sections. Simple measures such as returning the oil to the tank through an inverted U-tube can produce astonishing benefits. Fitting baffles to the tank also positively influences air release. These have the effect of prolonging the time in which air, water and solid contaminants can be released from the oil [13.2–13.4].

## 13.6
## Flushing Turbine Oil Circuits

Before commissioning, all oil circuits should be mechanically cleaned and finally flushed. Every effort should be made to remove all contaminants such as cleaners and corrosion preventives (oils and/or greases) from the system. The oil can now be added for flushing purposes. About 60–70 % of the total oil volume is required for flushing [13.4, 13.6]. The flushing pump should be operated at full power. It is recommended that the bearings are removed and temporarily replaced with blanks (to avoid the penetration of contaminants into the gap between shaft and bearing shells. The oil should be repeatedly heated to a maximum of 70 °C and then cooled to about 30 °C. The expansion and contraction in the pipework and fittings is designed to dislodge dirt in the circuit [13.4]. The shaft bearing shells should be flushed in sequence to keep the flow rate high. After at least 24 h of flushing, oil filters, oil sieves and bearing oil sieves can be fitted. Mobile filtering units, which may be used, should work with a mesh size of $\leq 5$ µm. All parts of the oil supply chain, including reserve machinery, should be extensively flushed. Finally, the flushing oil should be drained from the oil tank and coolers. All system components should be thoroughly cleaned externally. The flushing oil may be re-used after very fine filtering (by pass filtration). However, a careful oil analysis should first be made and care should be taken that the oil still fulfils DIN 51 515 or the equipment-specific specifications [13.4]. Flushing should be performed until no solid contaminants are found in the filter and/or no measurable increase in pressure is recorded in by-pass filters after 24 h. It is recommended that a few days of flushing and a subsequent oil analysis follows any system modifications or repair work [13.4].

## 13.7
## Monitoring and Maintenance of Turbine Oils

In normal circumstances, oil monitoring intervals of one year are perfectly acceptable. [13.2, 13.4, 13.6]. As a rule, these should be performed in the oil manufacturer's laboratories. In addition, a weekly visual inspection of the oil should be performed to spot contamination and impurities in the oil in good time. Filtering the oil with a centrifuge in a by-pass circuit is a reliable method. The contamination of the air surrounding a turbine with gases and other particles should be considered when operating a turbine. Topping-up lost oil (refreshing of additive levels) is a method worth considering. Filters, sieves as well as oil temperature and oil level should be checked regularly. In cases of longer shut-downs (longer than two months), the oil should be circulated on a daily basis and the water content should be checked regularly [13.4].

The control of used

- fire-resistant fluids in turbines,
- used lubricants in turbines and
- used turbine oils in turbines

is performed by the laboratory of the oil supplier. The analysis and the warning values of the different properties and their following-up are described in the VGB-Kraftwerkstechnik Merkblätter, Germany (VGB = Association of German Power Plants).

## 13.8
## Life of (Steam) Turbine Oils

Oil life of 100 000 h is not uncommon in large steam turbines [13.2, 13.3]. However, the antioxidant level in the oil can fall to about 20–40 % of the fresh oil (oxidation, aging). The life of turbine oil depends heavily on the quality of the turbine base oil, the operating conditions such as temperature and pressure, oil circulation speed, filtering and the quality of maintenance and finally, the amount of oil topped-up (this helps maintain adequate additive levels).

The temperature of the oil in a turbine depends on the bearing loading, speed, bearing dimensions and the oil's flow rate. Radiated heat can also be an important parameter. The oil circulation factor, i.e. the ratio between flow volume $h^{-1}$ and tank volume should be between 8 and 12 $h^{-1}$ [13.3–13.5]. Such relatively low oil circulation factors ensure that gaseous, fluid and solid impurities can be efficiently separated while air and other gases can be released. Furthermore, low oil circulation factors reduce the thermal loads on an oil (with mineral oils, oxidation speed doubles when the temperature increases by 8–10 K). During operation, turbine oils are exposed to considerable oxygen enrichment. Turbine lubricating oils are exposed to air at a number of points around a turbine. The temperatures of bearings can be monitored with thermo-elements. High bearing temperatures can be around 100 °C [13.2, 13.5] and sometimes even more in the lubrication gap. The temperature of bearings can reach up to 200 °C if localized overheating takes place. Such conditions can only be countered by large oil volumes and rapid circulation. The oil draining from plain bearings can be about 70–75 °C [13.3] and the oil in the tank can be about 60–65 °C. Depending on the oil circulation factor. The oil remains in the tank for between 5 and 8 min [13.3, 13.4]. During this time, any trapped air can be released, solid contaminants can settle and water can be separated. If the temperature of the oil in the tank is higher, additive components with high vapor pressures can evaporate. This evaporation problem is worsened by the installation of oil vapor extraction units. The maximum temperature of plain bearings is limited by the threshold temperatures of the white metal bearing shells. These are ca. 120 °C. The development of alternative bearing shell metals which are less heat-sensitive is currently underway [13.1, 13.3, 13.4].

## 13.9
## Gas Turbine Oils – Application and Requirements

Gas turbine oils are used in stationary turbines. These produce either electricity or heat. The compressor fans generate pressures of up to 30 bar which vent into the combustion chambers where gas is injected [13.3]. Depending on the type involved, combustion temperatures of up to 1000 °C are reached (generally 800–900 °C) [13.3, 13.13]. Exhaust gas temperatures can reach about 400–500 °C. Gas turbines with capacities ranging up to about 250 MW are used in urban and suburban steam heating systems, in the paper industry and in the chemical industry. The advantages of gas turbines are compact size, rapid start-ups (< 10 min) as well as small oil and water requirements [13.1, 13.3, 13.4].

Common mineral oil-based steam turbine oils are used for conventional gas turbines. However, it should be remembered that the temperature of some bearings in gas turbines is higher than in steam turbines so that premature oil aging can be expected. Moreover, hot-spots can occur around some turbine bearings and localized temperatures can reach 200–280 °C [13.3] whereby the temperature of the oil in the tank remains at about 70–90 °C (hot air and hot gases can accelerate the aging process). The temperature of the oil reaching a bearing is mostly between 50–55 °C and the exit temperature about 70–75 °C [13.3]. As the volume of gas turbine oils is generally smaller and they circulate more rapidly, their life is somewhat shorter. The volume of oil for a 40–60 MW generator (GE) is about 6000–7000 L and its life is between 20 000 and 30 000 h (in the case of a 40–60 MW Siemens, 14 000 L and 40 000–80 000 h [13.6, 13.9]. Semi-synthetic turbine oils (special hydrotreated base oils (so-called group III oils)) or fully synthetic turbine oils based on synthetic PAO's are recommended for these applications [13.3, 13.4, 13.8].

In civil and military aviation, gas turbines are used for propulsion. Because of the high temperatures encountered, special, low-viscosity (ISO VG 10, 22) synthetic oils based on saturated esters (e.g. polyolester oils) are used in these aircraft engines or turbines [13.13]. These synthetic esters have a high viscosity index, good thermal stability, oxidation resistance as well as excellent low temperature characteristics. Some of these lubricants can contain additives and some not. The pourpoint of these oils are between –50 and < –60 °C. And finally, all relevant civilian and military product specifications must be fulfilled. The lubricants used in aircraft turbines can in some cases also be used in helicopter, ship and stationary, industrial turbines. Aviation turbine oils containing special naphthenic base oils (ISO VG 15–32) with good low-temperature characteristics are also used [13.13].

## 13.10

## Fire-resistant, Water-free Fluids for Power Station Applications

For safety reasons, fire-resistant fluids are used in control and governor circuits which are exposed to ignition and fire hazards. In power stations, this applies in particular to hydraulic systems in high temperature zones such as near to superheated steam pipes. Fire-resistant fluids should not spontaneously ignite when they contact hot surfaces. The fire-resistant hydraulic fluids used in power stations are generally water-free, synthetic fluids based on phosphoric acid esters (type DFD-R according to DIN 51 502 or ISO 6743-0, ISO VG 32–68). These fire-resistant HFD-R fluids based on phosphoric acid esters offer the features [13.4]:

Specifications of triaryl phosphate ester turbine control fluids are defined according to ISO/DIS 10 050–category ISO-L–TCD [13.17]

- fire-resistance
- self-ignition temperature over 500 °C
- auto-oxidation stable at surface temperatures up to 300 °C
- good lubricity
- good protection against corrosion and wear
- good aging stability
- good demulsification
- low foaming
- good air release and low vapor pressure

Additives to improve oxidation stability (possibly foam inhibitors) as well as rust and corrosion inhibitors are sometimes used. According to the 7th Luxembourg Report, the maximum permissible temperature of HFD fluids in hydrodynamic systems is 150 °C. Continuous temperatures of 80 °C should not be exceeded in hydraulic systems. These phosphoric acid ester-based synthetic fluids are generally used for control circuits, but is some special cases, also for the lubrication of plain bearings in turbines as well as other hydraulic circuits in steam and gas turbine installations. However, these systems must be designed for these fluids (HFD-compatible elastomers, paint finishes and coatings). (E) DIN 51 518 lists the minimum requirements which power station control circuit fluids have to fulfil. Further information can be found in guidelines and specifications relating to fire-resistant fluids, e.g. in the VDMA Sheet 24 317 and in the CETOP recommendations R 39 H and RP 97 H.

Information relating to the change of one fluid to another is contained in VDMA Sheet 24 314 and CETOP RP 86 H [13.4].

## 13.11

## Lubricants for Water Turbines and Hydroelectric Plants

Hydroelectric power stations have to pay particular attention to the handling of water-polluting substances, i.e. lubricants. Lubricants with or without additives are used in hydroelectric power stations. The oils are used to lubricate the bearings and gearboxes of principal and ancillary machinery as well as for hydraulic functions in control and governing equipment. The specific operating conditions of the hydroelectric plant need to be considered when selecting lubricants. The lubricants must display good water and air release, low foaming, good corrosion protection, FZG Wear protection > 12 in gearboxes, good aging resistance and compatibility with standard elastomers [13.4]. As there are no established standards for water turbine oils, the existing product specifications for general turbine oils are adopted as basic requirements. The viscosity of water turbine oils depends on the type and design of the turbine as well as its operating temperature and can range from 46 to 460 $mm^2\ s^{-1}$ at 40 °C. Type TD and LTD lubricating and control oils according to DIN 51 515 are used. In most cases, the same oil can be used for bearings, gearboxes and control equipment. In many cases, the viscosity of these turbine and bearing oils is between 68 and 100 $mm^2\ s^{-1}$. When starting up, control and gearbox oil temperatures should not fall below 5 °C and bearing oil temperatures should not fall below 10 °C. In the case of machinery located in cold ambient conditions, the installation of oil heaters is strongly recommended. Water turbine oils are subject to little thermal stress, and as oil tank volumes tend to be high, the life of water turbine oils is very long. In hydroelectric power stations, the oil sampling and analysis intervals can be correspondingly long. Particular care should be taken when sealing the turbine's lubricating oil circuit from possible water ingress. In recent years, rapidly biodegradable water turbine oils based on saturated esters have proven successful in practice. Compared to mineral oils, these products are more rapidly biodegradable and are allocated to a lower Water Pollution Category. In addition, Type HLP 46 hydraulic oils (zinc and ash-free additives), Type HEES 46 rapidly biodegradable fluids and NLGI grade 2 and 3 greases are used in hydroelectric plants [13.4].

# 14
# Metalworking Fluids

*Theo Mang, Carmen Freiler and Dietrich Hörner*

The great flexibility and versatility of the different types of machining method are particularly significant in the metalworking industries. Although for some years now there has been a growing trend towards non-cutting (forming) methods for workpiece quality reasons and to save material and process costs, this has still not had any obvious effects on the volume share of lubricants. This is also apparent from the machine tool statistics. The dramatic change prophesied in the nineteen seventies did not take place in the nineteen eighties and nineties.

Because of the particular significance of cooling for the cutting operation, this process is called cooling lubrication and the fluids used are called coolants. Apart from this term 'coolant' which is commonly used in general practice, there are also numerous other terms for specific applications such as, for example, cutting oils, grinding oils, reaming oils, deep hole drilling oils and honing oils.

There are no exact figures available as to how many machining operations are carried out without using coolants. However, there is no doubt that wet machining is applied very much more frequently than dry machining. Since 1996, efforts have been made to extend dry machining in particular through research projects. The advantages of cutting fluids can be summarized briefly as follows: accelerated heat dissipation with increased tool service life to make higher cutting speeds possible, lubrication between tool, chip and workpiece with reduced tool wear and improvement of the quality of the workpiece surface finish, lubrication of sliding points outside the actual cutting zone between tool, workpiece and chips and improved chip removal. Frequently less attention is paid to lubrication outside the tool–chip contact zone which, nevertheless, can be very important. To be mentioned at this point are, for example, margins of twist drill and reamers as well as the support and guide rails on deep hole drills and honing tools.

In daily practice, after the selection of a suitable coolant, the problem of lubrication and cooling often takes a back seat for a long period. The actual daily work is determined by the correct application and care of these fluids and the numerous secondary demands put on them. As a result the handling of these secondary demands is to be given great attention in the following. Secondary demands and coolant care are also important cost factors in the coolant system, which is why they are also becoming more important when studying the system.

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## 14.1
## Action Mechanism and Cutting Fluid Selection

The simplest geometrical model for a machining operation is demonstrated by the orthogonal cut in a two dimensional view with continuous chip formation (Fig. 14.1). The cutting lip of the tool pushes into the workpiece after overcoming a crowding force and shearing occurs. The chip formed slides over the cutting face of the tool. The main forming work is carried out in the region of the shear zone, the position of which is defined by the idealized shear plane and the shear angle. One also talks about the primary shear zone. However, the friction between chip and tool has particular significance for lubrication and cooling. This causes the shearing action in the contact zone of the tool material (secondary shear zone) and influences the position of the shear plane, the size of the shear angle and the plastic deformation of the material in the shear zone. The magnitude of the plastic deformation, which is also expressed in the chip crowding, becomes greater with increasing tool face friction.

However, here the chip crowding is still influenced by other factors, such as the tool geometry and material properties. As far as this is concerned the direct influence of friction on chip formation can be recognized, which can also cause a change in chip form.

The influence of friction on the plastic deformation when machining by cutting has pushed the traditional explanation using the friction coefficient into the background in view of the latest explanations as to the effects of cutting fluids. New working models have been created and the basics of plasticity principles taken into account.

The decisive result of friction is tool wear. Figure 14.2 shows typical wear phenomena on cutting tools [14.1]. Material particles weld onto the cutting edge of the tool, leading to so-called built-up edges. This makes materials with high formability particularly susceptible to this tool destroying phenomenon (adhesion). Also, certain structure components of steel (austenite, ferrite) and cast iron (ferrite) can promote built-up edge formation. Apart from the influence on the cutting conditions the cutting speed can be especially effected.

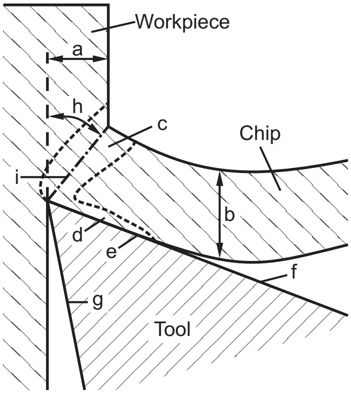


**Fig. 14.1** Chip formation with orthogonal cutting. a, cutting depth; b, chip depth; c, primary shearing zone; d, secondary shearing zone; e, contact zone; f, chip surface; g, front clearance; h, shearing angle; i, shear plane; b/a chip compression.

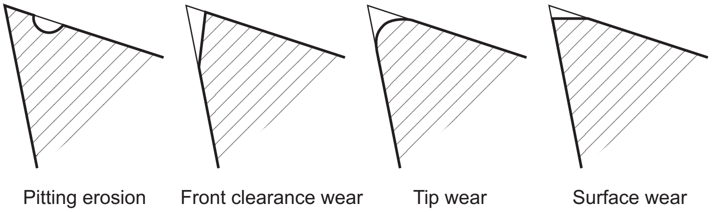


**Fig. 14.2** Significant forms of wear on cutting tools.

### 14.1.1
### Lubrication

There have been very different ideas on the action mechanism of cutting fluids over the last few years. It is mainly assumed that cooling alone reduces wear at high cutting speeds. However, it did not seem possible that the cutting fluid could penetrate into the contact zone between chip and tool at the cutting point in order to have a tribological effect. In general, a tribological significance of cutting fluids has only been determined in the lower cutting speed range. This opinion is still valid today for a number of cutting techniques; nevertheless the tribological effects are becoming more and more important as a result of new findings. For example, more favorable results are achieved with grinding oils for high performance grinding at very high speeds than are achieved with water-miscible products. The special effect of EP additives and the less favorable cooling properties of the oils compared to water-miscible products reflects the overwhelming importance of lubricant properties [14.2].

When machining by cutting, a very reactive 'clean' workpiece material surface is continuously generated which can tend to react adhesively on the cutting surface of the tool in the workpiece material–tool contact zone. It must be assumed that this reactive surface not only tries to saturate the free valences of the tool material but that other available substances are also involved which can be bonded by absorption, chemical absorption or chemical bonding. The oxygen in the air plays a particular role. In numerous tests under vacuum it has been possible to establish that the tool wear is considerably reduced through the saturation of the surface opened up by cutting, compared to where a gas or gas mixture has been available at different partial pressures. This is why it is known today that even in the case of dry cutting changes in the absorption and reaction properties of the material lead to changes in the tribology and, as a direct consequence, wear.

Coolant penetration into the contact surface according to more recent knowledge [14.3] is via a network of capillaries which are linked to each other. The magnitude of the capillary diameters are said to be $10^{-3}$ to $10^{-4}$ mm. Questions of transport kinetics indicate a special role, especially the diffusion speed. It must be assumed that it is frequently not the coolant as a whole but rather the component parts, brought about by evaporation or decomposition (pyrolysis) which impart a tribological effect.

It has been established in laboratory cutting tests on model substances with chlorine that lubrication is improved by the following aspects:

- high reactivity of the effective components with the surfaces;
- low shear strength of the reaction layer (lower than that of the basic metal);
- favorable diffusion properties of the effective components (lower molecular weight, higher vapor pressure).

As far as the saturation process of the newly formed surface is concerned, the surface will never be completely covered by lubrication effective molecules; there will be a clear gradient of surface activity in the direction of the tool tip, with little reaction at the outer range of the tool. High normal pressures will also prevent lubricant transport when machining materials which are difficult to cut. The assessment of cutting tests with model substances has revealed that 30 % saturation of the contact surface between chip and tool phase lead to the friction force being reduced by 75 % [14.3].

Even today in the development of coolants this knowledge is still not taken into sufficient consideration and it is still relegated by other priorities into secondary position.

### 14.1.2
### Cooling

In view of the findings relating to cooling and lubrication, it is impossible to differentiate the two. The changes in material properties caused by temperature are closely associated with the effects of friction. Cooling gains increasing significance for tool wear when maximum cutting temperature approaches the softening point of the tool material. The example shown in Fig. 14.3 [14.1] reveals how temperature can develop on a cutting surface dependent on cutting speed and chip thickness.

On one hand the cooling effect of the cutting fluid and the heat dissipated depends on its thermal properties, especially the heat capacity and the heat transfer

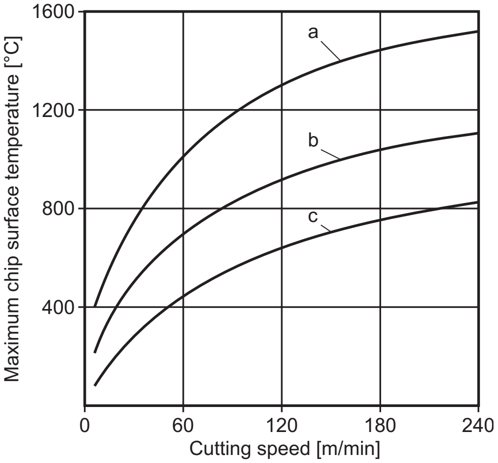


**Fig. 14.3** Dependence of tool surface temperature on chip thickness, $h$, and cutting speed [14.1]. (a) $h$ = 0.062 mm; (b) $h$ = 0.25 mm; (c) $h$ = 1.00 mm.

coefficient; on the other hand the flow conditions and the heat transfer coefficient play a significant role. The heat transfer coefficient can be influenced considerably by the substances active at the interface and by evaporation.

The high specific heat and high heat transfer coefficient of water give water-miscible cutting fluids more favorable cooling properties than non-water-miscible oils.

Through evaporation of water from water-miscible products, the heat of vaporization contributes considerably to the overall cooling. Table 14.1 shows the differences between mineral oil and water as far as thermal properties are concerned; the values for low concentration water-miscible cutting fluids are close to those of water.

**Tab. 14.1** Differences between calorific data of mineral oil and water.

| | *Mineral oil* | *Water* |
|---|---|---|
| Thermal conductivity, W $m^{-1}$ $K^{-1}$ | 0.1 | 0.6 |
| Heat capacity, kW s $kg^{-1}$ $K^{-1}$ | 1.9 | 4.2 |
| Heat of vaporization at 40 °C, kW s $kg^{-1}$ | – | 2400 |

The influence of the cutting fluid on the cutting temperature when wet cutting is shown in an exemplary way in Fig. 14.4 [14.4] in comparison with dry cutting. However, one question is still not answered by such measurements: what share of the reduced temperature is due to the reduction in friction and what share is the result of heat dissipation. Figure 14.5 shows a typical temperature profile in a tool. In this case a close relationship between the geometrical course of the isotherms and the position of the crater wear can often be established. And there are important consequences to be learned for understanding of the role of coolant, especially for the fluid supply.

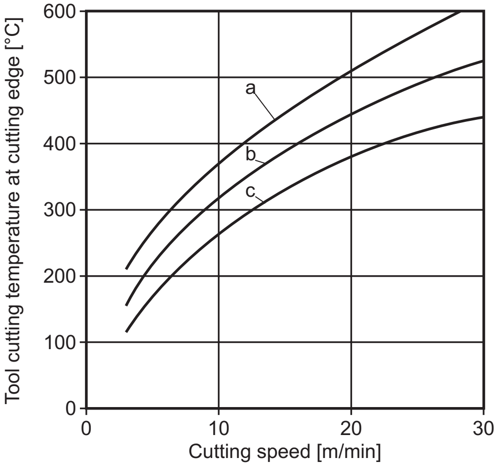


**Fig. 14.4** Cooling effect of three different metalworking fluids [14.4]. (a) dry cutting; (b) neat cutting oil; (c) water-mixed coolant 10 % (v/v).

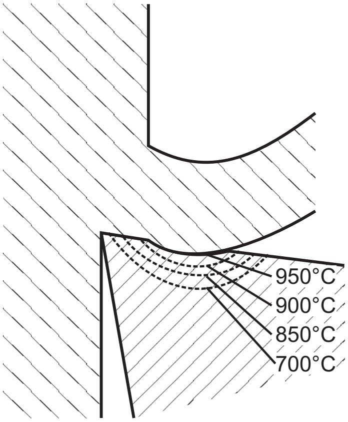


**Fig. 14.5** Relationship between temperature profile (the course of isotherms) and crater wear heat in the rake in workpiece geometry.

### 14.1.3
### Significance of Cutting Fluid with Various Cutting Materials

In his book, K. Sundberg [14.5] provides a good overview as to the significance of various materials and trends in development of tool materials today (Fig. 14.6). Reducing cutting costs and increasing productivity tends to lead more to diversification than to unification. Here the fast developing coating technologies with various substrates are gaining more and more ground.

#### 14.1.3.1 High-speed Steels

These are equivalent to high alloyed, high grade stainless steels based on chromium, nickel, tungsten and vanadium. Advantages are their toughness and the possibility of providing sharp cutting edges as well as good machinability. Their properties can be improved by PVD coatings. They have lost market share to cemented carbides in the last few years because of their comparably low hardness at higher temperatures. This is especially due to the increased cutting speeds.

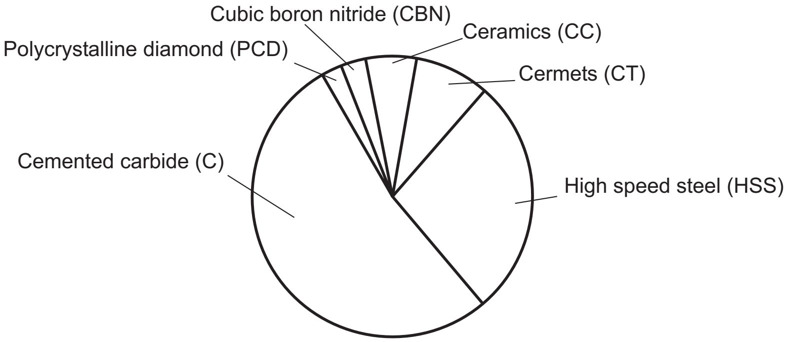


**Fig. 14.6** Distribution according to sales of the six basic cutting tool materials (1999).

Wet machining is normally recommended for these high speed steels. Water-miscible and non-water-miscible cutting fluids are suitable for lower and average cutting speeds while water-miscible products are given preference where very high cutting speeds are concerned.

#### 14.1.3.2 Cemented Carbide Metals

These are metal carbides which are brought together with a metallic binder by powder metallurgy. In this case the metallic phase brings the tenacity, the carbide the hardness. Cemented carbide metals retain their hardness far beyond the area of application of high speed steels. The most significant products for cutting are based on tungsten carbide (WC) and titanium carbide (TiC). Titanium carbide with nickel as the main binder are called cermets (CT) and have gained particular significance in Japan.

Sensitivity to thermal shock has to be considered where cutting fluids are used. Therefore it is expedient to pay special attention to uniform tool cooling and tool flushing with cutting fluid before the actual machining operation begins. When using neat oils, the risk of thermal shock is less than with water-miscible cutting fluids because of the lower cooling effect. This sensitivity is greater with TiC than in the case of WC.

#### 14.1.3.3 Coated Carbide Metals

High resistance to wear of carbide metals is achieved by coating with hard substances. Coating with titanium carbide (TiC) has gained greater importance over the CVD process (Chemical Vapor Deposition). With regards to use of cutting fluids similar conditions are valid as given for cemented carbides.

#### 14.1.3.4 Ceramic Materials

Ceramics is a general name given to this varied material family. Aluminum oxide ($Al_2O_3$) is sintered together with metals and carbides at very high temperature by a method similar to that employed in carbide metal technology. The result is cutting materials which are resistant to wear at even higher temperatures than is the case with cemented carbides. Furthermore, they react even more sensitively to fast changes in temperature so that these are generally dry machined. However, water-miscible coolants are being used more frequently to achieve uniform cooling and to avoid temperature shock.

The ceramics built up on a silicon nitride base have taken over an essential function in the case of high speed machining of cast iron. These have also replaced those ceramic materials based on aluminum oxide. So-called mixed ceramics have significance in turning of hard materials.

#### 14.1.3.5 Cubic Boron Nitride (CBN)

CBN is the second hardest material after diamonds. It is observed that use of CBN inserts is increasing every year to the detriment of uncoated cemented carbides. As a result, growth is also faster than in the case of ceramic materials. One focal point of application is the machining of gray cast iron with very high pearlite content (> 95 %). Hard part machining is an important area of application for CBN and in the case of certain materials also dry machining or the use of reduced volume or minimum quantity lubrication.

#### 14.1.3.6 Polycrystalline Diamond (PCD)

This is the hardest workpiece material and is given preference when used as a sinter material in a metal matrix. Good experience has been gained over many years in the light and medium machining of aluminum alloys. Cutting speeds of more than 1000 m $min^{-1}$ can be achieved with good surface quality finishes. As is the case with ceramic materials, these are sensitive to thermal shock. These are mainly dry machined but coolant is also used.

Synthetic diamonds have been used recently as CVD coating in a cemented carbide substrate.

#### 14.1.3.7 Coatings

Since around 1970 coatings on the various workpiece materials described above have continuously gained greater importance and have led generally to a significant improvement in wear resistance as well as extending the area of application. Initially chemical vapor deposition (CVD) was used (TiC on cemented carbide substrate). Although the earlier coatings were primarily thin mono-layers (3–5 μm) it is possible today to produce 15 μm thick multi-layer coatings.

Through the later introduced physical vapor deposition method (PVD) lower temperatures could be achieved resulting in sharper cutting edges. In this case thin 2–5 μm titanium nitride (TiN) and titanium carbon nitride (TiCN) coatings are applied; today these are the most important coatings and TiAlN is also expected to grow significantly in the future. PVD coatings on high speed steels (HSS) have mainly contributed to these ‘old’ materials still accounting for one third of the used workpiece materials today. CVD and PVD coatings have greatly contributed to reducing tool wear and in this sector have overshadowed the significance of wear reducing properties of EP substances in cutting fluids. They have also enabled dry machining methods to be extended. It is estimated that by the year 2015 up to 20% of today’s wet machining will be replaced by dry machining and minimum quantity lubrication.

### 14.1.4
### Cutting Fluid Selection for Various Cutting Methods and Cutting Conditions

The categorization of the different types of cutting fluids to the various cutting processes and the different cutting conditions (especially material, feed and cutting speed) has been attempted repeatedly in many documents from cutting fluid manufacturers. However, these tables have very little significance in practical application. If there are machining methods which call for a certain focal point specific, relatively good definable cutting fluid, so many aspects have to be considered for product disposition that the majority of the very rough categorizations cannot be utilized. The general product descriptions are not sufficient for an exact categorization of the different types of products. Manufacturer categorization provides only a general rough guide for the range of machining methods to be considered.

Even the essential subdivision of cutting fluids into water-miscible and non-water-miscible products generally provides no indication as to a particular categorization to one type of cutting operation and ease in machining. The use of both water-miscible and non-water-miscible products is applicable for almost all cutting operations, although in the case of reaming, deep hole drilling or honing, for example, non-water-miscible products have the greatest significance. As a result, a non-water-miscible oil is frequently used for a cutting problem because the machine seal does not permit use of water-miscible products.

The choice of the optimum cutting fluid for a particular cutting is most restricted by reasons of economy. The necessity to utilize large central circulation systems and the costs of coolant control and coolant care lead to most extensive rationalization in cutting fluids. Today, for example, especially where large-scale serial production with very varied degrees of difficulty is concerned, very different cutting operations frequently use the same cutting fluid. It may well be that all operations from problematic reaming to difficult grinding are all done with a 3 % emulsion. Such universal cutting fluids have outstanding significance as far as volume is concerned. There is also the increasing use of fluid families and the future use of universal fluid which should be noted (Section 14.10).

The great importance of workpiece material properties is always considered when selecting cutting fluids by selecting for different tensile strength and hardness values. In cutting fluid literature and in many technical product sheets of cutting fluid manufacturers especially in the USA, a machinability index is used [14.6], to provide a simple breakdown by product. In this machinability index, reference is made to a standard material and closely specified cutting conditions. Under these cutting conditions the material is given the value 100. The other materials are allocated a machinability index on the scale in relation to tool service life.

In the most exceptional cases, looking at machinability frequently leads to wrong conclusions being drawn when judging the rating table for cutting fluids. Not taken into consideration is that machinability is a very complex variable dependent on many parameters [14.1]. This becomes clear, for example, in Fig. 14.7, where the suitability of two lubricants is reversed for one material simply by changing the cutting speed. Often, problems of built-up edge formation are not considered because of this very simplistic view; in this case it is possible to make a 'good' coolant out of a 'bad' coolant by increasing the cutting speed. From this example it can be clearly seen that the selection tables of cutting fluid manufacturers – by machining operation and simple material data – can only provide a rough guide. It should also be noted that these tables frequently indicate very different products for one and the same operation.

Despite the complex relationships and the reservations made when defining machinability and the selection of cutting fluids based on this, the material breakdown by machinability groups enables the user to make an initial selection of the cutting fluid. In this case the number of material groups is generally between five and eight; a breakdown by six groups shown in Table 14.2 has proved to be particularly successful [14.7].

**Tab. 14.2** Breakdown of materials by machinability classes for cutting fluid selection [14.7].

| *Material* | *Material (selected examples)* | *Machinability index group (approximate average values)* |
|---|---|---|
| Group 1 | Free cutting steels<br>Unalloyed and low alloyed hardened and annealed steels (C 15, C 35, 16MnCr5)<br>– Automatic machine steels (9S20, 9SMnPb23)<br>– Construction steels (St37, St60) | 80 |
| Group 2 | Difficult to machine steels<br>– High alloyed hardened and annealed steels (24CrMo5, 42CrMo4)<br>– High alloyed chromium steels (X8Cr17, X40Cr13)<br>– High alloyed chromium nickel steels (15CrNi6, 18CrNi8)<br>– Rust and acid resistant chromium nickel steels (X2CrNi189, X10CrNiMoNb1810)<br>– Cast steel (GS-Ck16, GS-37SiMn75) | 50 |
| Group 3 | Most difficult to machine special materials<br>– Nickel and nickel alloys (NiCr10, NiCr1820)<br>– Manganese and silica-manganese steels (40MsCr22, 65SiMn5)<br>– Chromium molybdenum steels (24CrMo5, X6CrMo4)<br>– Silicon steels (38Si6, 55Si7)<br>– Titanium and titanium alloys (TiAI6V4, TiAI7Mo4) | 25 |
| Group 4 | Gray and tempered cast iron (GG-25) GTS-45) | 60 to 110 |
| Group 5 | Non-ferrous metals<br>– Copper and copper alloys (G-Ms65, G-CuSn10Zn) | 100 to 600 |
| Group 6 | Light metals<br>– Aluminum and magnesium alloys (AlMg5, AlMgSil) | 300 to 2000 |

## 14.2
## Friction and Wear Assessment Method for the Use of Cutting Fluids

Until today, no fully satisfactory method has been found to determine friction and wear behavior in the laboratory. The reason for this is that a laboratory test method has to be restricted to a very limited number of cutting conditions because of time and costs. Nevertheless, methods have been developed which are suitable for making a preselection and, in particular, provide important indications for product development. Frequently, the ability to provide useful information is considerably increased by combining values obtained from two or more fast-testing methods. Good assessment criteria can be obtained by combining a drilling test with a turning test for a lubricant, which would be suitable for general machining on automatic screw machines. In this case, it must always be ensured that such a cutting fluid gives a good compromise for a number of cutting operations. This is the only sensible method for testing cutting fluid for a specific process, for example, deep hole drilling.

Even after this fast-testing assessment, final conclusions can only be provided by controlled practical testing.

As a general rule, the mechanical test equipment which is used in many lubricant laboratories does not allow conclusions to be drawn as to tool wear, but provides valuable information on the reaction of additives for development and product control. Only in a few cases has a serviceable relationship been found between the values of a four ball apparatus (VKA), the Almen–Wieland Test (AWT) or the Reichert Wear Test (RVT) and the service-life values of tools in operation [14.8]. This applies also to the methods used more in the English speaking regions, such as the Falex Test [14.8c, d, e] or the Timken Test [14.8f].

### 14.2.1
### Tool Life and Number of Parts Produced by the Tool as Practical Assessment Parameters

The cutting time of a tool between two re-grinds is called the tool life and the distance traveled during the tool life is the tool-life distance. In the case of serial production, the number of parts produced during the tool life can be used instead of the tool service life to assess the cutting oil. In this case, the point in time determined as the limit of the tool life is defined, depending on the type of cutting operation and the workpiece produced. The following aspects are applicable: attaining the wear factor at which tool destruction is to be expected; reduction in acceptable surface quality of the workpiece; exceeding workpiece tolerances; detrimental influence on machine function. In controlled practical tests to assess cutting fluids, the most frequently used method when judging the tool life is the measurement of the wear-mark width.

### 14.2.2
### Measuring Cutting Forces in Screening Tests

The most broadly used rapid testing method applied for many years to assess cutting fluids is the so-called tapping-torque test. To this end, threads are cut in carefully prepared drill holes and the torque measured as an assessment parameter [14.8a]. The 'tapping efficiency' is determined by drawing a comparison with a reference cutting fluid. This calculated value is defined as follows:

$$\text{Tapping efficiency (\%)} = \frac{\text{Torque of standard reference cutting fluid} \times 100}{\text{Torque of the cutting fluid to be assessed}}$$

The values obtained by this method provide good differentiation between various non-water-miscible cutting fluids but the differentiation is not distinct for water-miscible products [14.9]. Nevertheless, the method is also used to assess water mixed products [14.10, 14.11].

Apart from the mechanical recording of the torque during the screening tests, the testing possibilities have been extended through the development of very sensitive drill measuring hubs. Measurements are normally made using a strain gage fitted between drilling spindle and tool.

During drilling and tapping tests, the feed force is also used as an assessment criterion for cutting fluids [14.12].

### 14.2.3
### Feed Rates at Constant Feed Force

The test also offers itself from both a tool and machine point of view. In the case of constant feed force, drilling can be in solid material or in pilot holes with a smaller spread of the measured values. The feed per rotation (feed rate) provides clearly different values for various cutting fluids [14.13, 14.14].

### 14.2.4
### Measuring Tool Life by Fast-screening Methods

Tests to determine tool life are generally expensive both in time and materials. As a result, one tries to reduce the costs for cutting fluid assessment by increasing the cutting speed or the feed. Here again, as a general rule, drilling is selected as the cutting method and the test is continued until a stipulated wear pattern is given or drill failure occurs [14.15]. Apart from providing good differentiation between products, the results of this test reveal the relatively high costs of the test itself: for example, 400 holes are necessary for just one product at a lower cutting speed [14.16]. The tool service life as a deciding factor is described in the Taylor equation:

$$C = V \times T^n$$

In this equation $V$ is the cutting speed, $T$ is the tool service life, and $C$ is a constant dependent on the material and the cutting conditions. The equation can be converted as follows:

$$V = C/T^n$$

or

$$\log V = n \times \log T + \log C$$

Presenting the last equation in a double-logarithmic system give a straight line relationship. Based on this relationship, cutting fluids can be characterized, with regard to their cutting properties, with the help of one point on the straight line and the gradient $n$.

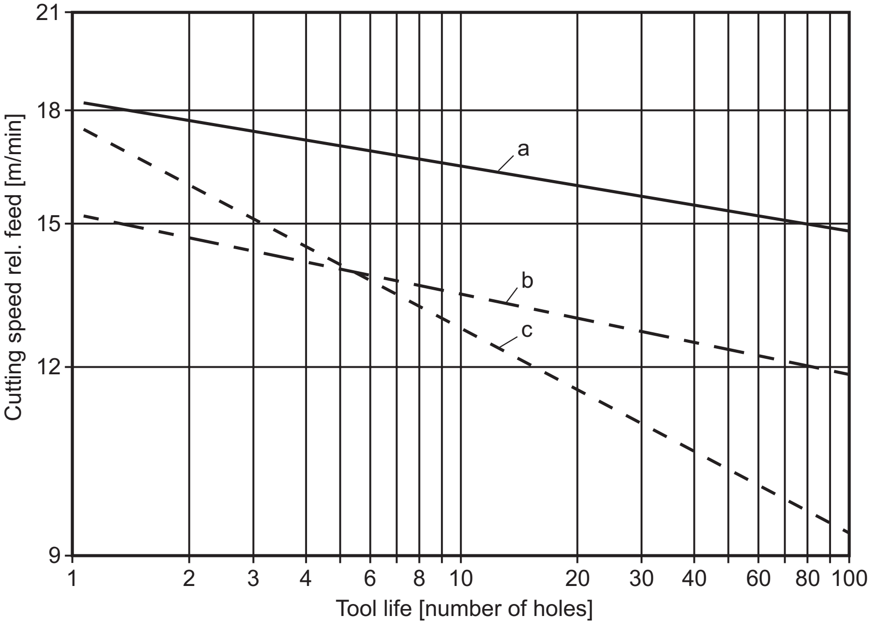


**Fig. 14.7** Assessment of three water-mixed cutting fluids (a, b, c) with straight lines according to Taylor (drilling of austenitic nickel chrome steel [14.16]. (a) mineral oil-free cutting fluid; (b) EP-emulsion (3 % sulfur, 8 % chlorine); (c) conventional emulsion (~ 70 % mineral oil).

The costs are considerably reduced if, in those places which require high drilling effort at a low cutting speed, the drilling feed is increased and, in addition, one uses the product of the feed and the cutting speed in relation to the standard feed as a calculation parameter, instead of the cutting speed alone. Figure 14.7 shows such an assessment for three coolants [14.16].

The higher the cutting fluid line on the graph and the flatter it runs, the more favorable the properties of the cutting fluid being tested. This characterization of the tool-life graph according to Taylor also shows the different suitability under different cutting conditions.

### 14.2.5
### Cutting Geometry and Chip Flow

When the chips run over the cutting face of the tool the friction developed has a considerable effect on the position of the shear zone and, consequently, on the plastic deformation of the chip. The chip length will be shorter than the cutting path and the chip is subject to compression. Since the friction is influenced by the cutting fluid, conclusions can also be drawn as to the influence of chip geometry on the lubricating properties of the cutting fluid. With geometric deviation from the orthogonal cut, the chip cross section (a rectangle in the case of orthogonal cut) experiences a deviation, for example, to a parallelogram, and the chip flow direction is also changed. These criteria are also used in the laboratory to characterize cutting fluids [14.17].

### 14.2.6
### Other Fast Testing Methods

#### 14.2.6.1 Temperature Measurement

An attempt has also been made to assess cutting fluids by measuring temperatures according to the bi-metal method [14.18]. This is a thermal process by which two different cutting materials engage in a workpiece and serve as a thermocouple. The electrical potential can be converted to temperature values. Washing the tools with cutting fluid causes the temperature generated at the point of contact to be reduced, and the fluid also dissipates the heat to a considerable extent. In this situation, the use of the two cutting tools in the form of threading tools is recommended for special assessment of lubricating properties, because friction has a greater significance when thread cutting.

#### 14.2.6.2 Radioactive Tools

A further fast-test method to assess the wear protective properties of cutting fluids can be made with radioactive tools. In this case, the radioactivity of a defined amount of chips, or, depending upon the amount of chips, the radioactivity of the chips suspended in the fluid, is measured. This is a measure of tool wear. Recently, the radio nuclear technology method, and especially the possibility of thin layer activation, has opened up great possibilities. Thin layer activation is clearly less problematic than the earlier activation methods. Since the radiation intensity through the wear of tools tested in this way is clearly reduced, it is also possible to measure radiation intensity directly on the tool.

#### 14.2.6.3 Surface Finish

In some of the described test methods, assessment of the surface of the workpiece is used to differentiate the cutting fluids; in some thread cutting tests this is the most important parameter used to characterize lubricants. For example, in one method [14.13], a barely acceptable surface finish is defined and the cutting speed at which this surface finish is obtained, is determined.

## 14.3
## Water-miscible Cutting Fluids

By volume this is the most significant group of metalworking fluids in the USA and Europe (using as a reference the amount of concentrate from which the users produce the water-mixed finished products). In the Far East, especially in Japan, non-water-miscible products clearly outweigh water-miscible products. Here the trend to increase the share of water-miscible products is even more evident, whereas in Germany, a trend towards non-water-miscible products is more apparent, not least because of the stringent legislation covering storage and handling of lubricants of all kinds.

The question of the cutting performance of water-miscible cutting fluids, which has considerable significance, is not generally of primary importance in their selection, as is the case with non-water-miscible cutting fluids. The secondary demands put on these products are frequently so much the focal point of discussions that their actual main tasks, such as cooling and lubricating, are overshadowed. Of ever-increasing importance are the problems of health protection in the workplace, microbiology, hygiene and the disposal of used fluids. The change of fluids through contamination very frequently makes considerable expenditure necessary for treatment, unlike the case for non-water-miscible cutting fluids. The specific processing costs are normally determined separately for each application, but a general statement as to whether water-miscible cutting fluids are less expensive or not, cannot be made in this way.

Following consideration of these environmental problems, most development work over recent years has centered around improving the resistance to outside influences, especially against microbiological attack.

### 14.3.1
### Nomenclature and Breakdown

In hardly any other sector of lubrication technology are there so many different, and to some extent inaccurate, terms used as is the case with water-miscible cutting fluids. In recent years several attempts have been made to classify the different terms and to orient these either on user-technical criteria or product composition (Table 14.3).

**Tab. 14.3** Classification of cutting fluids.

| *Terms according to DIN 51385* | | *Code letters* |
|---|---|---|
| 0 | Metalworking fluid | S |
| 1 | Non water-miscible metalworking fluid | SN |
| 2 | Water-miscible metalworking concentrate | SE |
| 2.1 | Emulsifiable metalworking concentrate | SEM |
| 2.2 | Water-soluble metalworking concentrate | SES |
| 3 | Diluted metalworking fluid | EW |
| 3.1 | Metalworking emulsion (oil in water) | SEMW |
| 3.2 | Metalworking solution | SESW |

Here, the most significant differences are defined according to concentrates and water-mixed finished products and in general, the terms non-water-miscible coolants and water-miscible coolants are defined. The concentrates, as they are delivered to the user by coolant manufacturers are called water-miscible cutting fluids, the finished products are diluted with water by the user and called diluted cutting fluids. These terms are generally used today in literature and, unlike other conventional terms such as drilling oil, drilling and grinding water or coolant, are already being used by a wide group of users. In contrast, it is still unusual to differentiate between emulsifiable and water-soluble metalworking fluids when describing concentrates which will be used to form the emulsions or solutions. Dividing water-mixed products into metalworking emulsions and metalworking solutions gives an indication as to the composition, whereby emulsions always contain chemical emulsifiers.

The code letters also given in the above standard are used as an abbreviation for the lubricants themselves and are used to mark lubricant drums, lubricating equipment and lubricating points. In this case, the first letter, S, stands for metalworking fluids and the letter E stands for lubricating oils which are used mixed with water. The letter M in the third position stands for a water-miscible coolant with mineral oil content, the letter S for products from a synthetic base. The above-mentioned coding is explained in detail in DIN 51 502, although the additional letters N and W are still not included in this.

Apart from this German terminology, ISO 6743 has become established for use in the international field and is defined under Part 7 of the metalworking group (Table 14.4).

**Tab. 14.4** Classification of lubricants for metalworking (family M) according to ISO 6743/7.

| | |
|---|---|
| L-MHA | Fluids which may have anticorrosion properties |
| L-MHB | Fluids of MHA type with friction-reducing properties |
| L-MHC | Fluids of MHA type with extreme pressure (EP) properties, chemically non-active |
| L-MHD | Fluids of MHA type with extreme pressure (E.P.) properties, chemically active |
| L-MHE | Fluids of MHB type with extreme pressure (E.P.) properties, chemically non-active |
| L-MHF | Fluids of MHB type with extreme pressure (E.P.) properties, chemically active |
| L-MHG | Greases, pastes, waxes, applied pure or diluted with a fluid of MHA type |
| L-MHH | Soaps, powders, solid lubricants, etc., and blends thereof |
| L-MAA | Concentrates giving, when blended with water, milky emulsions having anticorrosion properties |
| L-MAB | Concentrates of MAA type having friction-reducing properties |
| L-MAC | Concentrates of MAA type having extreme pressure (E.P.) properties L-MAI |
| L-MAD | Concentrates of MAB type having extreme pressure (E.P.) properties |
| L-MAE | Concentrates giving, when blended with water, translucent emulsions (micro-emulsion) having anticorrosion properties |
| L-MAF | Concentrates of MAE type having friction-reducing and/or extreme pressure (E.P.) properties |
| L-MAG | Concentrates giving, when blended with water, transparent solutions having anticorrosion properties |
| L-MAH | Concentrates of MAG type having friction-reducing and/or extreme pressure (E.P.) properties |
| L-MAI | Greases and pastes applied blended with water |

In this very detailed list the letter L stands for lubricants. M stands for metalworking but each of the following letters has no particular significance on their own [14.19]. Further subdivisions are to be found, amongst others, under ASTME [14.20] and the Machinability Data Center [14.6].

### 14.3.2
### Composition

Since, worldwide, the proportion of metalworking fluids formulated without mineral oil content (so-called fully synthetic cutting fluids) only amounts to 5 to 15 %, the emulsions are quite clearly the most important products. The main ingredients can be summarized in the following groups (Table 14.5) [14.21].

**Tab. 14.5** The main ingredients of metalworking fluids.

Mineral oil hydrocarbons
Synthetic hydrocarbons, synthetic esters, fatty oils
Emulsifiers
Corrosion inhibitors
Stabilizers, coupling agents
Extreme pressure additives (EP substances)
Anti-wear additives
Lubricating film improvers
Antifoam agents
Microbiocides
Complexing agents

Even if mineral oils are the major constituents, then part or even the total mineral oil content can be replaced by synthetic hydrocarbons such as polyalphaolefins or alkyl benzenes by fatty oils or even synthetic esters. In many cases the use of synthetic hydrocarbon is restricted to specific areas of use but synthetic esters or natural fat oils are in regular use.

Basically, suitable water-miscible cutting fluids can be produced both from paraffinic as well as naphthenic oils; naphthenic oils, however, show more solubility for additives and more favorable emulsifying properties. However, the use of naphthenic oils is declining because of the higher aromatic content compared with paraffinic oils. This is considered critical, particularly with regard to health and safety at work.

#### 14.3.2.1 **Emulsifiers**

Emulsifiers fall under the large group of chemicals which are known as surfactants or surface-active substances. All surface-active substances foam in an aqueous solution; this is an undesirable phenomenon in the entire metalworking field as far as emulsifiers are concerned. The water-soluble surface-active substances, to which emulsifiers are also to be included, are frequently also described by the term ‘detergents’ [14.22]. Apart from emulsifiers, other important material classes are surfactants for cleaners, wetting agents and dispersing agents.

Surfactants have the property of separating at the interface whereby their concentration in the fluid remains lower than at the interface. This property is also termed capillary activity. Apart from the thus achieved lowering of the surface tension, emulsifiers have nevertheless other properties which clearly differentiates them from other surfactants, such as wetting agents. The reduction of surface tension makes the emulsions thermodynamically stable, this means that they reduce the natural tendency of the emulsified phase to reduce interfacial area. However, other conditions must be fulfilled for good or acceptable emulsion stability.

The dissolution of emulsifier molecules, the enrichment at the interface and thus also the entire emulsification process is dependent on time and it is easy to understand that the kinetics of the emulsifier effect have considerable practical significance for the production of emulsions. Apart from the task of reducing interface tension, emulsions have a further important job of keeping the surface of the drops of oil as stable as possible (in the case of oil-in-water emulsions) so that a collision does not cause droplet enlargement. The following mechanism takes place:

The emulsifier molecules, which comprise bipolar substances, arrange themselves on the surface of the drop in a brush structure. If oil droplets are present, the oil-soluble part in the oil phase (lipophilic, hydrophobic) will be adequate, as will the water soluble (hydrophilic) part in the water phase. This is shown in Fig. 14.8. The hydrophilic emulsifier parts orientated to the water phase cause the development of a hydrate layer. Here, to simplify matters, one can imagine an increase of the water surface viscosity and thus a firmer, protecting envelope around the oil droplet. Also, identical polarity on the outside leads to electrical repulsion when two droplets approach each other. Significant energetic importance is also attached to the development of the hydrate layer in the emulsifying process. The energy released when the hydrate layer is formed is available for the surface enlargement. Emulsifiers are used to produce type oil-in-water metalworking emulsions which have a very pronounced hydrophilic effect. To ensure the stability of the concentrates at lower temperature, correct solubility of all components needs to be considered. However, the oil solubility of some emulsifiers can be clearly improved by so-called coupling agents. Alcohol and glycol are used especially as coupling agents. Soaps used as emulsifiers can, for example, be easily dissolved and coupled by Guebert alcohols [14.23].

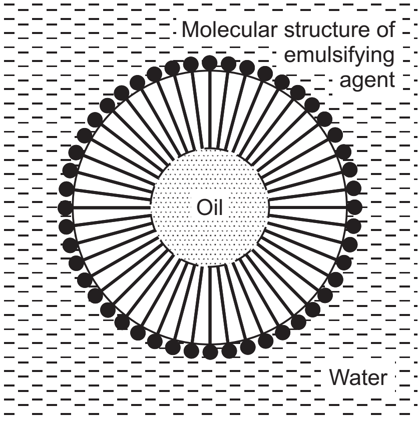


**Fig. 14.8** Orientation of the emulsifier molecules on the OW interface with an OW emulsion.

Surfactants tend to form micelles (molecule knots). In this case the molecular hydrophilic parts in the solution in water are directed to the outside and the lipophilic or hydrophobic parts are directed towards the inside. The equilibrium between the emulsifier molecules oriented of the interface, possibly ionized in the water phase and united in the micelles represents a very complex colloidal picture. The question arises, especially in the case of metalworking emulsions, as to level of excess emulsifiers in the emulsion. This is of special significance in assessment of the ability of emulsion to also emulsify tramp oils.

As is the case with all water soluble surfactants, the emulsifiers are divided into ionic and non-ionic products. The ionic products split (are dissociated) in water to cations with a positive charge and anions with a negative charge. If the anion has surfactant properties, these are anionic emulsifiers. If the cation is bipolar in character, one refers to cationic products. Apart from these two clearly divided groups of ionic emulsifiers, there are also amphoteric substances which can act as cationic or anionic emulsifiers depending upon their environment, especially pH value. Their significance as emulsifiers in cutting lubricants is so small that they do not have to be covered in detail here.

The non-ionic emulsifiers do not dissociate in water and the molecule has complete surfactant properties. Their water solubility is generally caused by oxygen-containing groups in the molecule with a higher affinity to water (hydrogen bonding of the water molecules to the oxygen atoms of the hydrophilic molecule group). The orientation of the water molecules imparts water solubility, and splitting at higher temperatures reduces solubility, which becomes evident through cloudiness.

The most important emulsifier groups for metalworking are described in more detail; Tables 14.6 and 14.7 show the chemical structures of ionic and non-ionic emulsifiers.

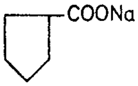
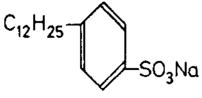
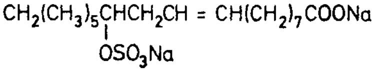
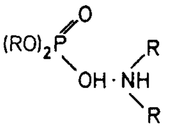
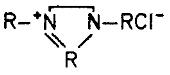
### Anionic Emulsifiers

Here we find the classical and, quantitatively, the most significant emulsifiers for conventional water-miscible cutting fluids and for many emulsions for non-cutting processes.

Soaps have been used for a long time. These are sodium or potassium salt, long-chained carboxylic acids such as, for example, the stearic or oleic acids. The most significant of these alkali soaps for oil-in-water emulsions are the sodium soaps. The limitation to their areas of application is determined primarily in that they are only able to develop their full emulsifying ability with comparably high pH values (~ 10). Such high pH values are not desirable for many machining processes because of the resulting skin problems.

Less dependency on the pH value has been achieved with the introduction of amine soaps. They provide good effectiveness with considerable tolerance and, as a result, also good stability of the emulsions produced by them, in a pH rate of approximately 8. The most important are the mono, di- and triethanolamine soaps (but the use of diethanolamine and morpholine derivatives is declining because of the formation of hazardous nitrosamines (TRGS 611); to produce them the chemicals morpholine, diglycolamine, tri-isopropanolamine and methylaminopropanol [14.24] are used, amongst other chemicals].

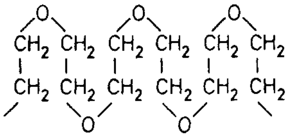
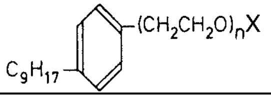
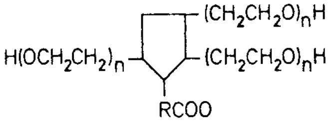
**Tab. 14.6** Chemical structures of ionic emulsifiers.

| | |
|---|---|
| ***Anionic emulsifiers:***<br>Sodium soaps: | $R-COONa$ |
| Amine soaps:<br>(Triethanolamine soaps) | $R-COONH\ (CH_2CH_2OH)_3$ |
| Naphthenic acids: |  |
| Sulfonates:<br>Example of "synthetic sulfonates"<br>Dodecylbenzenesulfonate: | $R-SO_3Na$<br> |
| Sulfates:<br>Special significance:<br>Sulfated castor oil (Turkey red oil) | $R-OSO_3Na$<br><br>$CH_2(CH_3)_5\underset{OSO_3Na}{CH}CH_2CH = CH(CH_2)_7COONa$ |
| Amine salts of phosphoric esters: |  |
| ***Cationic emulsifiers:***<br>Quaternary ammonium salts:<br>Salts of fatty amines<br>Salts of imidazoline | $R-NH_3^+CH_3COO^-$<br> |

The common disadvantage of amine and sodium soaps is that they form non-water-soluble salts with magnesium and calcium ions from the water or with heavy metal ions from the machining operation (for example, in the case of emulsions with copper content when drawing copper wire). As a result a certain proportion of the emulsifier is inactivated and precipitated. This, in turn, can lead to instability of the emulsion. The precipitated, insoluble soaps on the other hand, can also cause considerable trouble during machining or in the circulation systems.

Sodium and amine salts of naphthenic acids also number amongst the soaps. Naphthenic acids, which are produced from acidic naphthenic mineral oil distillates by neutralization and extraction in mineral oil raffination are, by far, the main raw material used. Unlike the previous soaps, they are comparatively insensitive to water hardness agents since the magnesium and calcium salts are more easily soluble in water than those of the carboxylic acid soaps. Frequently, one disadvantage is the stronger tendency of alkali soaps of naphthenic acids to foam [14.24, 14.25].

**Tab. 14.7** Chemical structures of the most important non-ionic emulsifiers (ethoxylates).

| | |
|---|---|
| Polyethylene oxide chain: |  |
| General formula of ethoxylated products:<br>n = number of moles ethylene oxide | $X-(CH_2-CH_2-O)_nH$ |
| Ethoxylated phenols:<br>Special significance:<br>Nonylphenolethoxylates |  |
| Ethoxylated alcohols: | X = RO |
| Ethoxylated fatty acids: | X = RCOO |
| Ethoxylated fatty amines: | X = RNH |
| Ethoxylated fatty acid amides: | X = RCON |
| Polyols,<br>Example ethoxylated sorbitan ester |  |

Sulfonates are very widely used in water-miscible cutting fluids. The most popular are the so-called natural sulfonates or petroleum sulfonates. These were formerly mainly produced as secondary products in the sulfuric acid raffination of lubricant distillates, which had considerable significance especially in the production of white oils. The worldwide trend to produce white oils and even lubricant oil raffinate by the hydrogenation method has reduced the availability of petroleum sulfonates. The favorable properties of these products, especially their good corrosion protection, led to the further development of their manufacturing method. As a result petroleum sulfonates are available today from a special sulfonization process. Chemically, this class of product is primarily a matter of the sodium salts of sulfonic acids of aromatic/aliphatic mineral oil hydrocarbons; this is why they are also termed alkylaryl sulfonates. The molecular weight of the products of interest for metalworking is between 350 and 500. As the molecular weight increases the emulsification properties decline and the corrosion protection properties become more favorable [14.26].

The so-called synthetic sulfonate is gaining more and more in importance. Even if pure aliphatic products are used in individual cases, of dominating significance are the aromatic products of the dodecylbenzene sulfonate type (sodium salt).

The effectiveness of sulfonates is, as in the case of all other anionic emulsifiers, influenced by water hardness salts but in this case they behave more favorably than the soaps. Where the pH values are in a range of over 8.5, the emulsions built up on this basis show good stability [14.23].

Through the sulfonation of natural fats, one gets sulfated fatty oils, a further group of anionic emulsifiers which are used frequently in metalworking emulsions. These are particularly suitable for the emulsification of fatty oils. Particularly important are sulfated castor oil, sulfated fish oil and sulfated colza oil. Unlike the sulfonates, the sulfur in the molecule of the sulfate is not bound directly to the C atom but is linked via an oxygen bridge. The tendency towards acidic hydrolysis calls for particularly careful pH control where emulsions based on sulfated oils are concerned and also calls for special buffer systems [14.24].

Amine salts of phosphoric acid esters offer good corrosion protection and EP properties on top of their action as anionic emulsifiers, which makes their use interesting for a number of applications. However, many of these amine phosphates have unfavorable foaming properties. Countless other special compounds, which are used especially as corrosion inhibitors in water-miscible coolants, have the same effect as anionic emulsifiers. In this context, it is worth mentioning, for example, salts from amido carboxylic acids (sarcosine) [14.27].

### Cationic Emulsifiers

Until now cationic emulsifiers have gained no particular significance for water-miscible cutting fluids and are only used for some special applications. Frequently, incompatibility with anionic products (reciprocal precipitation) is the reason for the restricted use; cationic products adsorb strongly on metal surfaces and thus hydrophobizing is generally a disadvantage. The effectiveness of some cationic emulsifiers in neutral and acidic pH range has favored their use for some special purposes in the processing of aluminum. Also favorable is the frequently good resistance towards microbial activity (bacteriostatic effect) and the stability in hard waters. Quaternary ammonium salts are the most important examples of this group of substances.

### Non-ionic Emulsifiers

The significance of this group of emulsifiers has increased greatly in recent years. Special advantages are the stability in presence of water hardening agents and the electrolyte resistance in general. Their stability in a wide pH range is also more favorable than that of ionic products though the low corrosion protection is often a disadvantage. Principally, these are only the ethoxylated products which have any significance for metalworking. Customized emulsifiers can be produced with very different emulsifier properties from different hydrophobic molecules by attaching a hydrophilic polyethylene oxide chain (polyglycol ether) with an exactly defined length.

The number of attached ethylene oxide molecules defines the degree of ethoxylation. The following substances are preferred in the production of non-ionic emulsifiers: alkyl phenols (nonylphenol used to be the most significant substance but is no longer used in formulas because of the waste water legislation in Europe), alcohols, fatty acids, fatty amines, fatty acid amides and esters of polyols (sorbitan esters are particularly important): In the EU the use of nonylphenolethoxylates is prohibited by Directive 2003/53 EC [14.131]. Table 14.7 shows the chemical structure of the product groups mentioned here. Due to the low corrosion protection of the majority of ethoxylated non-ionic emulsifiers mentioned above, these are combined in metalworking emulsions with anionic products. Special exceptions are some ethoxylated fatty acid amides which can provide considerable corrosion protection [14.24].

#### HLB Value

The HLB value has been created to characterize the emulsifying properties of different emulsifiers. HLB stands for hydrophilic lipophilic balance [14.8, 14.29]. If the reader wishes to go more deeply into this subject their attention is drawn to special literature.

### 14.3.2.2 Viscosity of Emulsions

Although the water-in-oil emulsions used in metalworking are generally pasty in consistency, the oil-in-water emulsions for cutting, wire drawing or rolling are available as thin liquid systems. A number of formulas, the majority of which has empirical origins, have been drawn up to determine the viscosity of oil-in-water emulsions. Interestingly, it has been shown that neither the particle size (degree of dispersion) nor the viscosity of the disperse phase influence the viscosity of the emulsion. The emulsion viscosity $\eta$ has been defined by the Hatschek equation:

$$\eta = \eta_0/(1 - \Phi^{1/2})$$

where $\eta_0$ is the viscosity of the continuous phase (water) and $\Phi$ the volume fraction of the disperse phase. The equation shows that these two parameters alone determine the viscosity of the emulsion. However, when working with metal machining emulsions, microfine systems (small particle size) frequently show a higher viscosity than coarse systems (large particle size) when using the same emulsion concen-

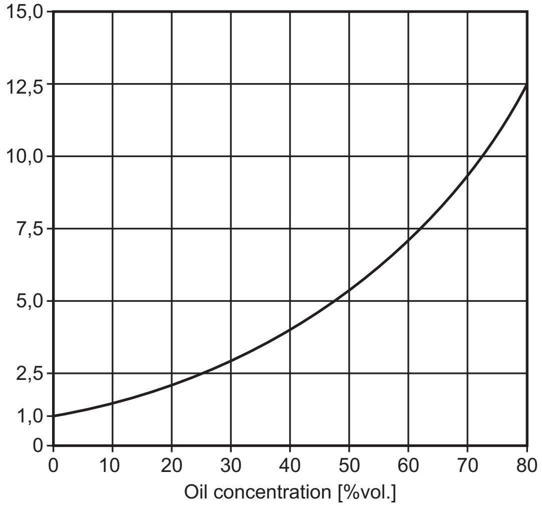


**Fig. 14.9** Dependence of the viscosity of oil-in-water emulsions at 20 °C on the volume proportion of the disperse phase [14.30].

tration. Figure 14.9 shows the dependency of the viscosity on the volume (%) of the disperse phase, determined according to the Hatschek equation [14.30].

### 14.3.2.3 Phase Reversal, Determination of the Type of Emulsion

Phase reversal is defined as the conversion of an oil-in-water emulsion into a water-in-oil emulsion or vice versa. When mixing emulsions for cutting operations a white creamy substance sometimes forms which normally disperses irreversibly. In the majority of cases, this creaming substance represents a water-in-oil emulsion and only presents problems when the reversal (decomposition) in an oil-in-water emulsion is delayed or prevented after stabilization with lime soaps. The water-in-oil emulsion can also occur in the case of a wrong emulsion mix, when, for example, water is added to the concentrate and the process is not reversed. Within certain limits, the phase reversal for many emulsion systems is a function of the concentration relationships. Although it is possible to produce oil-in-water emulsions with 99 % volume as a disperse phase, the oil-in-water to water-in-oil transition range frequently lies between 70 and 80 % oil. This is shown in Fig. 14.10.

Very often it is expedient to determine the type of used emulsion involved, when working with systems which are close to the oil-in-water to water-in-oil transition range.

This is the case, for example, with some drawing greases for sheet metalworking or highly concentrated wire drawing emulsions. Much more specific correction measures can be planned and carried out on such systems if the type of emulsion is known. There are a number of methods which enable these to be determined.

As far as the indicator method is concerned a water-soluble dye (for example, methylene blue) and a fat and oil-soluble dye (for example, a Sudan dye) are used. If the water-soluble dye penetrates into the emulsion, water is the external continuous phase but if the fat soluble dye penetrates into the emulsion this will be a water-in-oil emulsion with oil as the external phase.

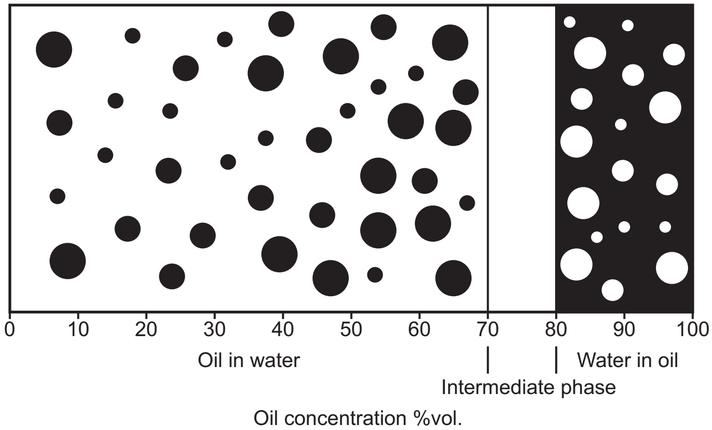


**Fig. 14.10** Dependence of the phase reversal of oil-in-water and water-in-oil disperse systems on oil concentration.

In the case of the drop method, one drop of the emulsion to be tested is placed on water or oil. The oil-in-water emulsion will spread over the water and the water-in-oil emulsion over the oil.

The titration test has been developed for emulsions of firm consistency and, in this case, the absorption of the water (of the oil-in-water emulsion) or the oil (water-in-oil emulsion) is observed.

The change from water-in-oil to oil-in-water to emulsions is always accompanied by a considerable increase in electrical conductivity. This means that measurement of electrical conductivity can also be used to determine the type of emulsion. However, in this case, one needs to know the conductivity of the emulsion type. False results can also occur because of electrolyte enrichment in used emulsions. When the filter paper method is used the emulsion to be tested is spread over a filter paper. In oil-in-water emulsions a moist edge forms around the emulsion stain after some time whereas this does not occur in water-in-oil emulsions, they often produce an oil or fat edge. Distinguishing between the two is sometimes difficult. The method can be improved if the filter paper is soaked in a cobalt chloride solution (pink colored) and is afterwards dried out completely (bright blue coloring). The water given off by oil-in-water emulsions colors the blue paper pink which ensures clear identification [14.31].

#### 14.3.2.4 **Degree of Dispersion**

The particle size of the disperse phase is of decisive importance for different emulsion applications for several reasons. On one hand, size is one of the specific parameters for the stability of an emulsion but on the other hand, it is also one of the decisive points for the lubrication effect and thus has considerable tribological significance. The degree of distribution of the disperse phase characterizes the degree of dispersion. A high degree of dispersion results where very small particle sizes are concerned and a low degree of dispersion with relatively coarse emulsions. The definition of colloidal terms used for the particle size ranges are classified most frequently in Table 14.8. [14.25].

**Tab. 14.8** Particle size terminology.

| Type of colloid | Size (μm) |
|---|---|
| Molecular disperse | < 0.001 |
| Fine colloidal | 0.001–0.01 |
| Medium colloidal | 0.01–0.1 |
| Coarse colloidal | 0.1–1 |
| Colloidal | 0.001–1 |
| Microscopic | 1–1000 |

All the microscopic and colloidal ranges mentioned above are applicable for metalworking if one also includes the soap solutions or other systems containing no oil which are not emulsions. This is shown in the examples in Table 14.9.

**Tab. 14.9** Particle size ranges for metalworking fluids.

| | |
|---|---|
| Unstable emulsions for rolling of fine sheet steel, direct application | > 50 µm |
| Semi-stable emulsions for rolling of fine sheet steel | 10 to 50 µm |
| Typical emulsions for coarse cutting | 0.1 to 10 µm |
| Typical emulsions for grinding (low mineral oil content) | 0.1 to 0.01 µm |
| Soap solutions, different applications | 0.01 to 0.001 µm |

In the case of technical emulsions we are not dealing with monodispersed systems in which all particles are the same size. On the contrary, we are dealing with polydispersed systems with a particle size distribution which is frequently very broad. This can best be demonstrated by presenting the frequency as a percentage divided by the particle diameter. Some metalworking emulsions show clear peaks for one particle size, while others consist of several diameter ranges which becomes apparent by several maxima in the curves (Fig. 14.11).

The droplet size distribution can be determined by microscopic and photographic methods. The techniques used are usually light scattering methods (laser diffraction) and electrical methods (Coulter Counter [14.32]). When working with metalworking emulsions, the droplet size distribution in known systems can be calculated from stability measurements. Modern measuring methods are not nearly as well suited for emulsions for cutting processes with a high percentage of particles with a diameter of less than 1 µm, as they are for emulsions used in aluminum rolling and for the cold rolling of plate and sheet.

### 14.3.2.5 **Stability**

The emulsion stability is closely linked to the droplet size and droplet-size distribution and is a key parameter for practical application. At this point, the physical stability of the emulsions is to be defined, independent of influences foreign to the system.

According to Stokes, the reciprocal value of the rate of climb or descent of the droplet is indirectly proportional to the difference in density between internal and

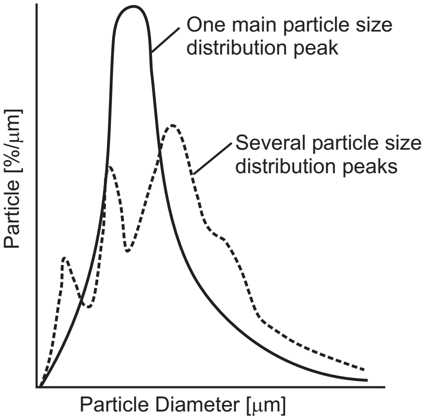


**Fig. 14.11** Particle size distribution for emulsions.

external phase and the square of the particle radius, and directly proportional to the viscosity of the external phase:

$$1/v = K\eta/[r^2 g(d_1 - d_2)]$$

where $v$ is the rate of climb and descent, $\eta$ the viscosity of the external phase, $r$ the drop radius, $g$ the gravity constant, $k$ a constant, $d_1$ the density of the heavy phase, and $d_2$ the density of the light phase.

The creaming, which defines the thickness of the cream layer of unstable emulsions, is given by:

$$v = 2kr^2 g(d_1 - d_2)/9\eta$$

An effort has been made to make the determination of stability for emulsions even more exact by making corrections to the Stoke equation. Nevertheless, this has no significance in practice for metalworking.

From the simple Stoke equation a number of conclusions can be drawn for the development of applications for metalworking emulsions, even if the term stability is very complex in nature. The following statements can be made.

- The stability is indirectly proportional to the difference between the densities of the two phases:

$$1/v \sim 1/(d_1 - d_2)$$

If the oily phase is used for oil-in-water emulsions with a density close to that of water (for example by adding heavy components such as chlorinated paraffins) then there is a trend to higher stability.

- The stability is directly proportional to the viscosity of the external phase:

$$1/v \sim \eta$$

The use of highly viscous oils or emulsions usually leads to more stable emulsions.

- The stability is directly proportional to the effective gravity constant:

$$1/v \sim 1/g$$

Centrifugal processes for separating coarser fractions of, for example, rolling emulsions or cutting emulsions are effective as a result of the centrifugal force, which is many times greater than the gravitation constant. These are successfully used for the separation of tramp oil from central lubrication systems.

- The stability is indirectly proportional to the square of the particle radius:

$$1/v \sim 1/r^2$$

The particle size makes the greatest contribution to emulsion stability which is why the majority of stabilization measures when using metalworking emulsions are directed towards increasing the degree of dispersion by adding emulsifier concentrates.

The methods of testing to establish the physical stability as well as other key stability values (electrolyte stability, thermal stability, microbial stability) will be discussed when dealing with special application methods.

### 14.3.2.6 Corrosion Inhibitors and other Additives

Where, in the case of emulsions, an important part of corrosion protection is provided by the anionic emulsifiers, especially soaps and sulfonates, the inhibitor systems of products free from mineral oil are more extensive and problematic. The great significance of inhibitor systems in cutting fluid substances low or free from mineral oil has also lead to this type of corrosion inhibitors being used for a more accurate classification of products. For example, one refers to the carboxylate type (alkanolamine salts of carboxylic acids), borate type (sodium borate) or PTBB type (*p-tert*-butyl benzoic acid), which, for toxicological reasons, are used only in exceptional cases today. Carboxylic acids, especially the alkaline and alkanolamine salts, have been used in cutting fluids for many years now. The latest inhibitors from the range of technically available carboxylic acid derivatives are being used to an ever increasing degree [14.33]. Table 14.10 shows the most important groups of corrosion inhibitors for water-miscible cutting fluids [14.21, 14.33, 14.34, 14.132].

**Tab. 14.10** Most important groups of corrosion inhibitors for water-miscible cutting fluids [14.21, 14.33, 14.34, 14.132].

| |
|---|
| Alkali and alkanolamine salts of carboxylic acids |
| Sulfonates |
| Alkali and alkanolamine salts of naphthenic acids (naphthenates) |
| Amine |
| Amide |
| Benzoic acid derivative (derivative of *p-tert*-butylbenzoic acid) |
| Amine salts, acidic phosphor acid ester |
| Boron compounds (boric acid amide) |
| Alkali nitrite (sodium nitrite) |
| Alkali carbonate |
| Carboxylic acid derivative |
| Alkylsulfonamide carboxylic acids |
| Arylsulfonamide carboxylic acids |
| Fatty sarkosides |
| Phenoxy derivatives |
| Sodium molybdate |

Five examples of formulas are given in Table 14.11 for coolants with low or no mineral oil content with special reference to the corrosion inhibitors.

**Tab. 14.11** Five examples of formulas for coolants with special reference to corrosion inhibitors. The figures refer to percentages by weight [14.21, 14.35, 14.36].

| | | |
|---|---|---|
| Inhibitor system, sulfonate–naphthenate–nitrite: | 14.0 | Mineral oil |
| | 1.1 | Sodium sulfonate |
| | 1.6 | Sodium naphthenate |
| | 8.0 | Naphthenic acid |
| | 11.8 | Monoisopropanolamine |
| | 2.5 | Sodium nitrite |
| | 61.0 | Water |
| Inhibitor system, borate–amide–carboxylate: | 20.7 | Mineral oil |
| | 15.8 | Water |
| | 5.8 | Sodium borate |
| | 12.6 | Block polymer from polypropylene ethylene oxide |
| | 8.0 | Fatty acid amide |
| | 37.1 | Alkanolamine salt of a carboxylic acid |
| Inhibitor system boramide: | 30.0 | Boric acid amide |
| | 5.0 | Polyolester |
| | 10.0 | Alkanolamine |
| | 1.0 | Biocide |
| | 54.0 | Water |
| Inhibitor system carboxylate: | 30.0 | Alkanolamine salts from carboxylic acids |
| | 10.0 | Alkanolamine |
| | 1.0 | Biocide |
| | 59.0 | Water |
| Inhibitor system carboxylate: | 25.0 | Alkanolamine salt of oleic acids |
| | 5.0 | Hydrocarbons (mineral oil) |
| | 5.0 | Glycol |
| | 1.0 | Biocide |
| | 64.0 | Water |

### Stabilizers, Solubilizers

Poor oil solubility of additives can be overcome by so-called coupling agents (glycols, alcohols). In this way it is also possible to achieve stabilization of concentrates for longer storage at low temperatures. The term stabilizers for the same group of substances also refers to the possibility of improving the emulsion stability of water mixed products.

### Defoamers

Frequently, coolants with high surfactant content tend to foam in use. The formulas for water-miscible coolants therefore sometimes contain defoamers. The most important groups of products used for this purpose are the silicones and, in particular, dimethylsiloxane. This group of antifoam agents tends to persist – they are difficult to remove with common cleaners. Because of this feature, the presence of very small amounts of these products can lead to problems with regard to paint-work, varnishing, and heat treatment of metal parts, for example nitriding or carburizing procedures. To prevent these problems, a second group – organomodified silicones – are very important. These substances have excellent removability, because of their predominantly organic character and greater polarity, so do not have the disadvantages mentioned above. Sometimes even special non-ionic surfactants can be used. The defoamers, also called antifoaming agents, based on silicones, are disproportionately dragged out compared to other coolant components.

#### Complexing Agents

These products are used if non-softened water is used for mixing and producing water-miscible coolants (concentrates). Clouding and precipitation can be avoided by the softening effect. The use of complexing agents in water-mixed products can prevent strong formation of calcium soaps and increase the service life of emulsions, especially in the case of cutting fluids with a high anionic surfactant content. The oxidative aging of coolants can also be reduced through the complexing of catalytic metal ions. It has also been shown that complexing agents in connection with some biocides, especially triazines, have microbiocide effects [14.37].

The most significant group of complexing agents are derivatives of ethylene diamine tetraacetic acid (EDTA). However, complexing agents are particularly undesirable when splitting of the used emulsion takes place by ultrafiltration because then heavy metal ion complexes can migrate to the water phase and lead to an undesirably high heavy metal content. This can have a considerable detrimental influence on disposal because the upper limit of the permissible heavy metal content may be exceeded.

### 14.3.2.7 **Cutting Fluids Containing Emulsifiers**

Figure 14.12 shows the rough composition of 3 types of water-miscible coolants (proportions by weight) with the model of their dispersions [14.23, 14.38]. Product 1 in this figure contains approx. 60 % mineral oil and 24 % emulsifiers, including anionic corrosion inhibitors. The water-mixed products of these concentrates are typical mineral oil emulsions with a white milky, sometimes also very light blue, opal colored appearance. Frequently these are coolants which can be used universally but which are mainly used on rough machining operations. Compared with product 2 the dispersion model shows relatively coarse droplets of emulsion with a low amount of excessive emulsifiers, which take the form of colloidal micelles.

Water-miscible coolants with approx. 70 % mineral oil and 15 to 20 % emulsifiers always provide milky white emulsions with frequently reduced electrolyte stability. Similar in their appearance are products which, apart from the high mineral oil content, also contain a considerable amount of fatty oil. The lubricating properties are improved through the polar components, especially in the case of difficult cutting operations on light and non-ferrous metals. A typical formula for such a product is as follows [14.35]; the figures are percentages by weight: 15 % emulsifiers (carboxylic acid soaps, sulfonates), 15 % lard oil, 1 % biocide, 69 % mineral oil.

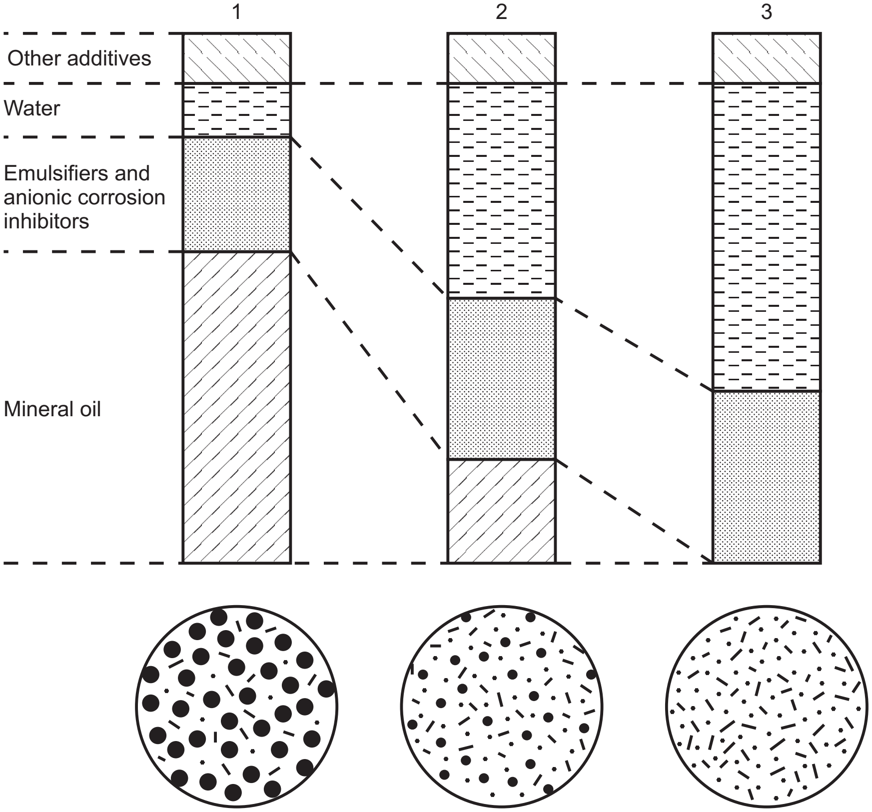


**Fig. 14.12** Composition of water-miscible coolants containing emulsifiers and their dispersion models. 1, conventional soluble oils; 2, emulsion with low mineral oil content (semi synthetic, micro emulsion); 3, mineral oil-free metalworking fluids containing surfactants. The black dots represent the oil droplets, the stars the emulsifier micelles and the lines the molecularly-dissolved surfactants.

Apart from polar substances, many EP additives used for lubricating oils can be incorporated in emulsifying coolants. These are known as EP emulsions. When EP additives containing chlorine are added, care must be taken to ensure suitable stabilization against hydrolysis and the resulting chloride corrosion. The following shows an example of a coolant formula with EP additives on a phosphorus and sulfur basis; the figures are percentages by weight [14.21]: 10 % sulfurized fatty oils, 4 % sulfurized di-isobutene, 3 % zincdialkyldithiophosphate, 16 % emulsifiers (soaps, sulfonates, non-ionic emulsifiers), 1 % other additives, 2 % mineral oil.

The use of EP additives in mineral oil free synthetic coolants is a problem. Many of the water soluble components containing chlorine, sulfur and phosphorous have little or low EP characteristics. The triethanolamine salt of phosphate ester has found particular significance in this respect [14.11].

The coolant 2 in Fig. 14.12 with approx. 30 % emulsifiers and only 20 % mineral oil provides finely dispersed, generally blue, opal colored, lightly transparent emulsions with a high percentage of excessive emulsifiers. It is a typical representative of the so-called semi-synthetic products. In France this type is frequently classified under synthetic coolants and the water mixed product is termed to be a pseudo solution [14.38]. The main areas of application of this type of product are for highly stressed coolant systems with a low drag-out rate.

Type 3 in Fig. 14.12 is a soap solution product with no mineral oil. The high proportion of surfactants, especially in the form of anionic corrosion inhibitors, highlight this coolant and make it clearly different to other mineral-oil free products. The main areas of application are grinding operations. The high surfactant content, which of course is not bound to the mineral oil as an emulsifier, has a good cleaning and flushing effect but frequently with disadvantageous foaming properties. When fresh, the solutions are transparent; cloudiness can be caused by the emulsification of tramp oil. The water-miscible coolants, types 2 and 3, can also be produced with a considerably lower water content. When comparing products, in addition to the routine tests in the laboratory, this must also be taken into account.

### 14.3.2.8 **Coolants Containing Polyglycols**

Although the mineral-oil free coolant described in Fig. 14.12, part 3, is used mainly for grinding, there are many mineral-oil free products containing polyglycol for rough machining. In this respect, the macromolecular polyglycols offer more favorable lubricating properties than mineral oils. The washing properties are generally less favorable than products with a high surfactant content. Polyglycols tend to leave sticky residues as a result of the aging processes and the interaction with salts from hard water, abrasives, machine paints and other foreign substances, which in turn considerably restrict the area of application for these products. Depending upon the structure of the corrosion inhibitor system, these can have a very rapid separating capacity for tramp oils and generally do not tend to emulsify oils. The use of skimmers for tramp oil separation is especially recommended.

A typical composition for a product with a high water content is as follows [14.38]; the figures are percentages by weight: 25 % polyglycols, 26 % corrosion inhibitors, 7 % other additives, 42 % water.

### 14.3.2.9 **Salt Solutions**

As far as their chemical structure is concerned, salt solutions are the simplest coolants. The most important substances are inorganic salts such as sodium nitrite, alkali carbonate, alkali borate and alkali nitrate, frequently in connection with alkanolamines. In earlier years, the combination of triethanolamine and sodium nitrite used to be of major importance [14.34, 14.35] although this product group is no longer accepted by many users because of the toxicological objection (development of nitrosamines). A typical composition of such a product is as follows; The figures are percentages by weight: 17 % sodium nitrite, 13 % triethanolamine, 14 % surfactants as wetting agents, 15 % biocide (formaldehyde depot), 16 % water.

### 14.3.3
### Corrosion Protection and Corrosion Test Methods

Normally the corrosion protection afforded by a water-mixed cutting fluid is reduced as the degree of dilution increases. The quality of a product, as far as its corrosion protection properties are concerned, is, therefore, most frequently defined in laboratory application according to the concentration which still gives no corrosion or achieves a specific degree of corrosion. The most significant test methods are described in DIN 51 360, Part 1 (Herbert corrosion test) or IP 125 as well as in DIN 51 360, part 2 (chips/filter paper method). Similar testing methods are known under ASTM D-4627 and IP 287 whereas the French standard NF T 60-188 is more similar to the Herbert test.

In the Herbert corrosion test steel chips are placed on a ground cast iron plate and wetted with cutting fluid in the concentration to be tested. After a lapse time of 24 h at room temperature in a testing chamber the chips, and more especially the plate, are assessed according to the arising corrosion. The assessment is made based on the black patches (S) and rust (R).

By the chips and filter paper methods the gray cast iron chips (GG 30) are wetted on a circular filter paper with the coolant to be tested and left in a Petri dish at room temperature for 2 h. Afterwards, only the filter is assessed according to the corrosion stains. The assessment is made according to four degrees of corrosion based on the number of corrosion stains and the proportion of the area covered by these stains on the filter surface. In both of the described corrosion protection test methods the materials can deviate from the standard and be adapted to special applications. For example, the Herbert chips may be replaced by cast iron chips, and in the case of the chips/filter paper method, cast iron chips other than those described in the standard may be used. Determined by another group of corrosion test methods is the change in weight of specimens immersed in the fluid at a defined temperature and over a defined period of time. These methods only provide an assessment for special cases of application and, in general, have very little significance; they are sometimes used for the development of new inhibitor systems.

The testing of inhibitor systems for water-miscible coolants can also be carried out by electrochemical methods, such as taking potentiometric and galvanometric measurements [14.33].

When machining light metals and copper materials, slight changes in decorative surfaces sometimes play an essential role. The so-called non-standardized spot methods can be used to determine disadvantageous corrosion on the surface. In this case of few drops of coolant can be applied to carefully prepared test surfaces and the corrosion can be visually assessed after a lapse time of 16 h under fixed conditions (frequently room temperature). The corrosion testing of materials containing copper for water-mixed coolants can take place according to the copper strip test pursuant to DIN 51 759 T1 (ISO 2160, ASTM D-130, IP 154, NF M07-015).

In all corrosion tests with water-mixed coolants, the quality of the water which is used to produce the products has considerable significance. It has been frequently observed that fully demineralized water presents more arduous test conditions than hard water; high chloride contents also lead to more stringent test conditions.

### 14.3.4
### Concentration of Water-mixed Cutting Fluids

The possibility to produce water mixed products from water-miscible coolants with varying concentrations for adaptation to different cutting processes gives great flexibility. Normally, water-mixed coolants with concentrations up to approx. 30 % can be applied without any particular problems. However, if this concentration range is exceeded then the stability has to be assessed, especially in the case of conventional emulsions. When working with hard water, lime soaps can arise when working far below this concentration, which could lead to considerable irregularities in the course of machining; this depends to a great extent on the specific composition of the coolants. Often, the lower concentration limit is determined by the corrosion protection properties. In this respect, attention is drawn to the fact that concentrations below 1.5 % can only be checked with difficulty during operation because measurement of the concentration is a problem. Reducing the concentration by only a few tenths of a percentage point can already lead to a serious corrosion effect. Moreover, considerable problems can arise, especially when using conventional and semi-synthetic emulsions with a high tramp oil content, where an analytical separation between emulsified tramp oils and the true concentration is unclear. In the case of water-miscible coolants, which contain microbiocides, the skin compatibility of these products must be observed when using particularly high concentrations; this also applies for other additives. Normally, cutting fluids are very often used for rough machining with a concentration of between 2 and 5 %, but very frequently at 4 %. In the case of special applications such as deep hole drilling, reaming or generally for particularly difficult cutting operations the use of considerably higher concentrations is possible. Central circulating systems, which have to supply several different cutting operations with cutting fluids, often demand a compromise – also with the concentration selected; in such applications a concentration of 4 % is particular successful.

Frequently a concentration of between 1.5 and 2.5 % is selected for grinding operations which are to be carried out with water-mixed coolants. In the case of high performance grinding (grinding with high chip volume) higher concentrations are also sometimes used (for example, when grinding roller bearing tracks). The level of concentration is often restricted to an upper limit because of the grinding wheel becomes blinded or loaded.

#### 14.3.4.1 Determination of Concentration by DIN 51 368 (IP 137)

In the case of emulsions, the concentration is determined frequently in accordance with DIN 51 368 (IP 137), measurement of the components in water-mixed coolants by acid split. In this method, the emulsion is broken down at a temperature of 95 °C in a special measuring flask using hydrochloric acid. The volume of the separated oil phase multiplied by a product-specific correction factor equals the emulsion concentration. The correction factor is indirectly proportional to the portion of concentrate which can be separated under these conditions. In the case of conventional emulsions (60 to 70 % mineral oil) the separated oil phase is approx. equal to the concentration, and as the mineral oil content of the water-miscible coolant decreases, the correction factor increases and the determination is less accurate.

### 14.3.4.2 Concentration Measurement Using Hand-held Refractometers

A very much simpler method as far as time is concerned, is the refractometric measurement of concentration by means of a hand-held refractometer. The dependency of light refraction on the concentration of a water-dissolved or emulsified substance is exploited using this instrument. Generally, the Abbe refractometer principle is used, so that the measurement is not made directly at the refraction angle but rather at the angle of total reflection, which is also dependent on substance and concentration. This dependency on substance makes it necessary to use product-specific calibration curves, which can be very quickly produced for a series of concentrations. The instruments, which are particularly suitable for use in practical operation because they are very easy to apply in an operational area, are either calibrated in °Brix (distilled water is equal to 0 °Brix, a 50 % saccharose solution 50 °Brix) or are calibrated directly to volume percent related to conventional mineral oil emulsions. In the case of highly contaminated emulsions there are problems when reading the optical measuring fields. Since the measuring accuracy depends upon the measuring range of the instrument it is often expedient to select an instrument whose highest accurate measuring range is within the range of the concentrations in question. Very often, dilution with water at a ratio 1:1, 1:5 or 1:10 is expedient in the case of very high concentrations.

### 14.3.4.3 Concentration Measurement Through Individual Components

There has been no lack of attempts to analyze individual substances in the coolants and use these as an indication for a given concentration. In this case it was always assumed that the concentration of this substance occurred to the same extent as in the total concentration of the coolant or the concentration of all other substances used in the fluid. This leads to very inaccurate conclusions being drawn, particularly when concentration is determined by means of the boron content. The boron can be determined by calorimetric method as well as by AAS or ICP methods. Completely inaccurate values were obtained when determining the concentration of coolants containing boron when the boron acid amide is subject to uncontrolled decomposition in reaction to contaminants and, especially, bacterial mechanisms.

### 14.3.4.4 Determination of Concentration by Titration of Anionic Components

It is only possible to determine the concentration of conventional and low mineral oil content cutting fluids, so-called semi-synthetic cutting fluids, by titration of the anionic active components when a considerable part of the emulsion and inhibitor system is anionic. In practice, reproducible results only occur when there is considerably more than 10 % w/w of anionic substance in the concentrates. In this method, the anionic proportion is converted with a cationic surfactant solution by chemical reaction. An aqueous solution of the quaternary ammonium compound phemerol is, for example, used as a cationic reagent. The titration process is relatively extensive because it has to be carried out as a stage titration process. Recognition of the end point is made difficult by contamination with dark colored emulsions. Until now this method could not be introduced on a broad basis in the lubricant laboratory. However, it offers one of the few possibilities, under the stipulated restrictions, to identify stable emulsified tramp oils in conventional emulsions in conjunction with determination of concentration by hydrochloric acid or by refractometer.

#### 14.3.4.5 Determination of Concentration Through Alkali Reserve

In the case of cutting fluids with no and low mineral oil content with high alkalinity, it is possible to measure concentration by titration with an acid to a determined pH value. This method can only be applied when the alkali reserve of the product is very high compared with the possible introduction of acid by microbial effect or involvement of acidic contaminants.

#### 14.3.4.6 Concentration after Centrifuging

Emulsions can suffer loss of stability in use and tramp oils may be taken into emulsification. If such used-emulsions are subject laboratory centrifuge for a defined period of time (for example 20 min at 8000 g), then the coarse concentrate and tramp oil particles are separated and the stable, finely dispersed phase remains in the emulsion. If the concentration of the emulsions treated in this way is determined without consideration of the separated oil (for example, DIN 51 368), then values are obtained which are frequently considerably lower than the concentration determined before centrifuging. In practice, this concentration value is also termed centrifuge concentration and it sometimes offers a good indication as to the state of the emulsion. The greater the difference between centrifuge concentration and normal concentration, the more carefully the coolant system has to be monitored. However, interpretation of this difference is sometimes difficult because the proportions of concentrate and tramp oils that have been centrifuged can only be determined with great additional analytical expense.

### 14.3.5
### Stability of Coolants

Stability is the most important property for the service life of a coolant. Apart from the general terms for stability, in coolant practice stability is also defined according to the different stresses experienced during service. For example, electrolytic stability, thermal and boiling stability and frequently also stability against microorganisms.

#### 14.3.5.1 Determination of Physical Emulsion Stability

The oiling or creaming of emulsion is frequently assessed visually in a glass beaker by static test after a defined period of time (for example 24 h). A further static test is carried out in a separating funnel by measuring the change in concentration. In this case, the change in concentration of the emulsion found in the bottom part of the separating funnel after a fixed time is also determined and compared with the initial concentration of the emulsion directly after mixing. The stability or stability index (in %) is the quotient of the concentration in the bottom part of the separating funnel, after the appropriate holding time, and the initial concentration multiplied by 100 (see also DIN 51 367, testing the durability of emulsifiable cutting fluids mixed with hard water). In principle, determination of stability can also be carried out by the centrifuge method in the same way as with the static test. After centrifuging an emulsion specimen of known concentration, emulsion can be taken from under the separated oil layer by means of a pipet and the concentration determined.

Sometimes, it can happen with emulsions, whose oil phase is heavier than water, that precipitation or sedimentation occurs instead of oiling or creaming. As a result, values > 100 % can result when determining the stability index in the separating funnel. This can occur with EP emulsions with high additive value, in which, for example, a considerable proportion of the oil phase is chlorinated paraffins.

#### 14.3.5.2 **Electrolyte Stability**

The ions of the salt components of water and also ions from foreign fluids, especially from aqueous degreasing baths, can considerably influence the stability of emulsions. The resistance to accelerated oiling or creaming of a coolant compared to the tramp electrolytes dissolved in this coolant, is termed electrolyte stability. In this context, the quality of the mixing water used to produce the water-mixed cutting fluid is of great significance. Evaporation of water from the coolant during normal use leads to concentration and enrichment of electrolyte salts within the fluid. This increase in salt content also needs to be considered. The enrichment of salts depends not only upon the specific conditions of a circulating system but also on the coolant temperature, the surface area of the circulation system and the circulation factor, but above all on the drag-out value. In the case of high coolant drag-out the increase in salt content is kept low by continuously refilling with fresh emulsion. As a general rule, low drag-out values can lead to dangerous salt concentration levels. This is the reason for recommending the use of partially or fully desalinated water for systems with very low drag-out values. The salt content of the water can be roughly checked through the electrical conductivity or through the evaporation residue. The electrical conductivity is given in µS or mS (Siemens). The conductivity, at which an emulsion becomes unstable is termed the limit conductivity and in conventional emulsions can be as low as 2500 µS, which means that the emulsions oil or cream at higher values, and in the case of products with low mineral oil content these values can exceed 5000 µS.

The evaporation residue which is determined by evaporation of water and gravimetric measurement of the residue, contains, in contrast to the definitions of water hardness, the entire inorganic components of the water. Frequently an evaporation residue of > 3000 mg $l^{-1}$ leads to emulsion instability. This means that with coolant mixing water with an evaporation residue of 750 mg $l^{-1}$, this value is achieved after a fourfold concentration enrichment (see also VDI Directives 3397). Under constant working conditions, taking the origin of the salt into consideration, the salt concentration can also be determined from the chloride or sulfate content.

By water hardness we mean the content of alkaline earth ions, especially calcium and magnesium. The German measuring unit for hardness is °d, but recently mmole has also been used. 1 °d equals 10 mg CaO $l^{-1}$ or 0.179 mmole/l. Water with a high degree of hardness also has correspondingly high evaporation residue and high conductivity, which also leads to a high electrolytic stressing of the coolant. However, it must also be taken into consideration that soft and especially softened waters can have high conductivity and high evaporation residue.

The water hardness is particularly significant for the generally unfavorable development of insoluble alkali earth soaps (mainly lime soaps). Precipitation can occur through a reaction with anionic surfactants which contaminate the coolant systems and reduce the service life of the emulsions by lowering the emulsifier concentration (see also DIN 53905, determination of the resistance of surfactants to hard water).

Frequently a lower limit to the water hardness is dictated by the foaming behavior of the coolant. However, practice has shown that, with the trend towards continuously longer emulsion service lives, the use of partially or completely desalinated water as topping-up volumes has proven very attractive (Section 14.3.6). The determination of the electrolytic stability follows by adding salt (NaCl) and magnesium chloride ($MgCl_2$) or other salts to the emulsion or to the mixing water. Considerable differences can arise, depending upon whether the salt is added to the emulsion after the emulsion has been produced or to the mixing water before mixing the emulsion. Adding 3 g NaCl $l^{-1}$ is the procedure applied in many laboratories to determine the electrolytic stability. The determination of the stability itself is carried out by the same method described to determine the physical stability.

#### 14.3.5.3 **Thermal Stability**

In special cases emulsions must also remain stable under considerable thermal stress. The thermal stability is determined in the same way as the procedure described above but with the emulsion heated. Frequently the test is carried out at 80 °C over 24 h.

If, apart from being used as cutting fluids, emulsions are also used as corrosion protection emulsions or quenching media at high temperatures (60 to 90 °C), then they should be stable to boiling. To test stability during boiling, the emulsions are boiled for a defined period of time under reflux, after which oiling or creaming is assessed visually. Stability to heat can also be determined by a recirculation process, as shown schematically in Fig. 14.13. In the case of the circulating pump apparatus shown, a thin layer of the coolant passes through a stainless steel tube which is heated by a burner. In this way the short term and repeating stress of the coolant by passing over very hot surfaces is reproduced. Thermostatic control of the sump vessel is by means of a cooling and heating facility. The thermal stability can be assessed according to different criteria. Generally the volume of water which is necessary to replace the evaporation losses up to the time when the emulsion breaks is used as a measurement or assessment [14.39].

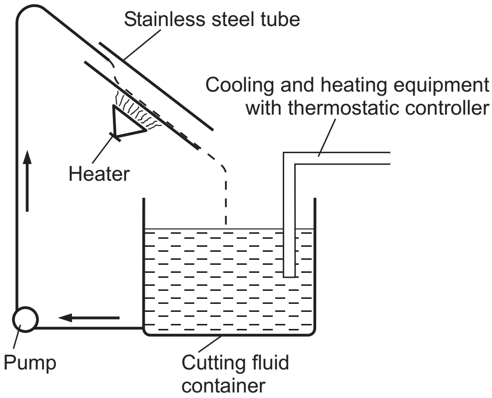


**Fig. 14.13** Schematic diagram of apparatus for determining the thermal stability of emulsions [14.39].

### 14.3.5.4 Stability to Metal Chips

The frequent contact of coolants with large chip surfaces can lead to a chemical reaction because of metal soap formation, and the chip surfaces can also accelerate the catalytic aging of the emulsion. The coolants can also be weakened especially with a high ratio of chip surface to coolant volume. This property can be tested by circulating the fluid through a system of finely packed chips for a longer period (several days or several weeks). Figure 14.14 shows such an arrangement in diagram form. The soap formation, concentration loss or other changes are studied by analyzing the different components after the circulation period.

## 14.3.6
## Foaming Properties

In all metalworking sectors as well as in the metal cutting field, the formation of stable foam on the processing fluids is an undesirable phenomenon. Especially cool-

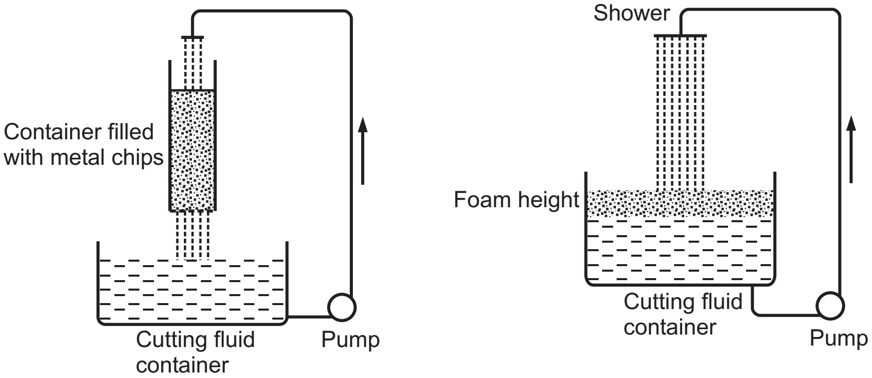


**Fig. 14.14** Pump circulating system for testing the stability of coolants to metal chips, and pump circulation method for determination of foaming characteristics.

ant containing surfactants, and these include all emulsions, tend, to a greater or lesser extent, to develop surface foam, under appropriate agitation.

### 14.3.6.1 **Definition and Origin of Foam**

Foam is a dispersion of a gas in the fluid. The lipophilic (oleophilic) parts of the surfactant molecules orient themselves on the air side of the air–water interface and lead, for example, to the formation of thin fluid lamella when two air bubbles collide with each other in the water or on the surface, which is the actual foam substance of the surface foam. The lamella are occupied on both sides by lipophilic surfactant molecules with the same molecular charge. These are, for example, emulsifier molecules or molecules of surface-active corrosion inhibitors which are molecularly dissolved in the fluid or which form colloidal micelles when a certain concentration level is exceeded (critical micelle concentration). When more and more surfactant molecules bind with the lamella surface the micelles again release molecules which are available to occupy further interfaces.

Depending upon the volume concentration of the enclosed gas either spherical or polyhedric foam is formed. In the case of homodispersed distribution, spherical shaped foam develops below a concentration of 74 % gas volume. Beyond the densest ball pack limit, the bubbles suffer deformation and lamella and polyhedric honeycomb bubbles are developed, which form the actual surface foam. Figure 14.15 shows the schematic formation of spherical and polyhedric foam with the development of a foam lamella.

The thermodynamic presentation of foam development has been proposed by Gibbs and Marangoni. There are a number of complex, generally only empirically determinable phenomena responsible for the actual stabilization of the foam. The formation of viscous surface films is one of the most important causes of foam stabilization [14.39, 14.40].

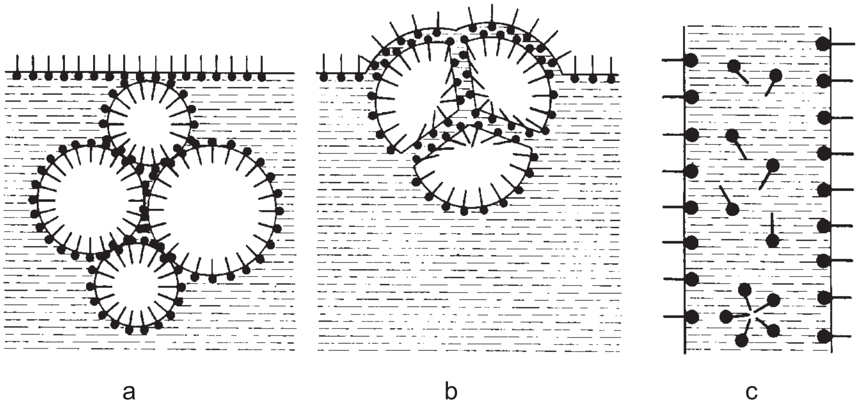


**Fig. 14.15** Schematic diagram of foam development. The hydrophilic part of the tenside molecule extends into to the water phase. (a) spherical foam; (b) polyhedric foam; (c) development of a foam lamella with molecularly dissolved tenside molecules and tenside micelles.

#### 14.3.6.2 **Foam Prevention**

The selection of a lower foaming coolant is the first of the measures to be taken. Very low foaming products are especially available amongst the more conventional emulsions. One reason for this is that the surfactant molecules (in this case essentially emulsifier molecules and anionic corrosion inhibitors) are mainly bound on the oil–water interface, that is to say to the oil drop, and are not available to be sited on the foam lamellae. The inhibition of foam by special foam inhibitors in water-miscible coolants has been common place for a long time. Foam destruction by addition of inhibitors to a water-miscible product during its use in the system is generally only an emergency measure, with very brief effective period.

The effect of the most significant group of defoamers for coolants, the silicones, is based on their particularly low surface tension and their insolubility in both oil and water. If silicones reach a lamella border surface as small droplets these can quickly spread and crowd out the surfactant molecules and start the breakdown of the foam. Performance of such defoamers is considerably enhanced when finely dispersed.

Frequently, foaming problems occur as a result of poorly designed coolant circulating systems. High circulation rates (low coolant volume), excessive drop height in the case of intakes and overflows, incorrectly positioned hydrocyclones for solid particle separation and hydraulic chip transportation via coolant nozzles are particularly important. Some machining processes, especially some grinding processes, are very sensitive to foam and call for particular care when planning the systems.

When foaming problems occur which can not be eliminated by defoamers, particularly in the case of soft water, then an improvement can be achieved by hardening the water. The water can be hardened by adding calcium acetate, i.e., 3 g (anhydrous product) to 100 l water increases the hardness by 1 °d.

#### 14.3.6.3 **Methods of Determining Foam Behavior**

Different kinds of foam generation also lead to different assessment of the foaming properties in coolants.

The method of introducing finely dispersed air through porous stones, which is sometimes used for oils, has not proved particularly successful for water-mixed coolants (ASTM D 892). The generation of foam by an agitation process could also not establish itself in the lubricant laboratory because no relation could be found with generation of foam in practice (for example, with Ultraturrax emulsifying rod or with laboratory agitators to ASTM D 3519-76). Very frequently simple non-standardized shaking processes are used. A defined amount of coolant is shaken in a graduated cylinder with a ground stopper or in other types of vessel for a defined number of strokes. Afterwards the height of the foam and the foam breakdown is determined over time. These methods are very widespread because of the low expenditure in apparatus and time. However, the individual handling of the manual shaking operation only leads to only moderate repeatability and reproducibility. The British norm IP 312, Frothing Characteristics of Water Mixed Metalworking Fluids and the American method ASTM D 3601, Standard Test Method for Foam in Aqueous Media (Bottle Test) are standard shaking methods. The foam height and foam breakdown is determined in both methods.

The pump circulation methods simulate real practice. The defined amount of fluid is circulated by pump, for example, over a shower. The fluid stream from the shower impinges on the surface of the fluid which causes foaming. The foam level after a specific testing period, and the time until the foam overflows the coolant tank and the foam decomposition time after switching off the pump are assessed. Figure 14.14 shows a diagram of the apparatus for such a circulating pump method.

The BAM method is a further development of the Giuglardi method [14.41]. By varying and measuring the spray pressure it is possible to differentiate the coolant over a wide range. The possibility of measuring and recording foam level by means of electric contacts makes it convenient and easy to determine the foaming tendency and the foam resistance (foam decomposition) with satisfactory reproducibility. There have been quite recent studies in Britain to introduce a new IP standard in which different forms of nozzles are also used to determine the foam level over a certain period of time [14.42].

It should be noted in all test methods that the type of coolant mix and especially the time between mixing the coolants and foam testing plays a significant role. It has been determined, for example, that some coolants show very unfavorable foaming behavior 24 h after their production compared to immediately after mixing; on the other hand, other products showed very low foam development after a period of 24 h [14.133].

### 14.3.7
### Metalworking Fluid Microbiology

One of the most significant problems in the development and application of coolants is their microbial susceptibility. In practice, this leads to considerable difficulties through the decomposition of essential compounds such as emulsifiers, corrosion inhibitors and other organic materials [14.43–14.47]:

- Reduced emulsion stability as a result of emulsifier decomposition.
- Reduced emulsion stability through acid development. Acid production by bacteria leads to a drop in pH value and this causes irregularities in the function of anionic emulsifiers and, as a consequence, also leads to emulsion instability.
- Corrosion problems because of pH decrease and corrosion inhibitor decomposition.
- Development of slimy residue which causes considerable contamination in the circulation systems; production breakdowns because intake and return lines become blocked and deposits settle on the machine tools; filter system breakdown through slimy deposits on filter materials.
- Dark staining of the coolants, for example, through metal sulfide; generation of foul-smelling reaction gases with considerable nuisance to the machine personnel.
- Hygienic and toxicological risks for machine personnel.
- Increased tool wear (IWT Bremen, 2006) [14.134].

Microorganisms use the organic material of the coolant as their main energy source. $NO_3^-$ in the mixing water serves as a nitrogen source. Apart from this the necessary nitrogen also comes from amines and amides, generally from corrosion inhibitors. In addition, phosphates and trace elements may be necessary but these are normally available in adequate amounts in the mixing water.

The coolant flora is made up of bacteria and fungi. The fungi are divided into molds and yeasts. Molds, and particularly yeasts, have caused problems in recent years since combating them is significantly more difficult than bacteria.

An important point is the great flexibility of many bacteria and fungi in adapting their nutritional needs in a continuously changing environment. Selectivity under specific conditions can lead to each coolant system being colonized by specific flora. Such conditions include the presence of oxygen, which favors aerobic species, the pH value or even the type of nitrogen sources, which are necessary for decomposition of amino acids. Basically microorganisms can attack the complete organic substance of the coolant and also the hydrocarbon frame of the mineral oils. However the most important point is that different coolant compounds are used by microorganisms, to different degrees and at different rates.

Under favorable conditions bacteria can split every 20 to 30 min, depending upon temperature and nutrients available. Propagation of mold and yeast is similar. Microorganisms therefore find very favorable conditions in coolant systems, since the nutrient solution (substrate) to which it has become adapted is re-supplied again and again as a replacement for the amounts removed.

Coolant concentrates (water-miscible cutting fluids) are sterile as a rule, i.e. they have the a microbial count of less then 100 $ml^{-1}$. In operation, the first inoculation takes place in the mixing water. Even drinking quality tap water can have 99 bacteria $ml^{-1}$. If industrial water from springs or river water is used then the microbial count can be significantly higher. However, microorganisms can get into the coolant through the air, waste and by contact with human beings, but especially through the coolant systems themselves. If, for example, a 100 $m^3$ central system with a microbial count of $10^8$ $ml^{-1}$ is emptied and then refilled, the 1 %, (i.e. 1 $m^3$) of old emulsion remaining in the system already leads to a microbial count of $10^6$ for the new emulsion. Thus system disinfection before refilling is of enormous importance.

Under anaerobic conditions, i.e. under the exclusion of oxygen, the decomposition of groups containing sulfur in sulfates or sulfonates occurs to a great extent. As a result the reduction of sulfur, i.e. by the bacterium *Desulfovibrio desulfuricans* (sulfate reducers) can follow until hydrogen sulfide is produced. This can lead to considerable odour nuisance. For this reason circulation of the coolant during work downtimes, such as weekends and to ventilate the system has been shown to be highly effective. In this way it is possible to avoid the Monday morning smell. Bacteria which adapt to play an anaerobic role, rather than conventional anaerobes, play an important role in coolant flora. They can adapt their metabolism and in the presence of oxygen effect aerobic respiration, while in the absence of oxygen their energy generation is anaerobic.

#### 14.3.7.1 Hygienic and Toxicological Aspects of Microorganisms

In earlier years only technical problems and odour nuisance were noted after microbial infestation, but today there is an ever increasing need to consider the health risks which may occur through highly infected coolant. It has been established that, especially in coolants with a high germ count ($10^7$ to $10^8$), even pathogenic and facultative pathogenic germs can be found [14.43].

Under the German Federal Epidemics Law, pathogens are divided into infectious germs (for example, salmonellae) and non-infectious germs (for example, *Staphylococcus aureus, Pseudomonas aeruginosa*). Infection of human beings can be either by penetration of the bacteria through the skin, by inhaling or swallowing. One area that has still been relatively ignored is the risk of poisoning from microorganisms (toxins).

Since no restrictive directives exist it is recommended [14.43] that the number of bacteria coming out of the human intestine should not exceed 1000 to 10 000 $ml^{-1}$ for water-miscible coolants. The entire bacteria group is termed as lactose positive enterobacteria for identification purposes (for example, *Escherichia coli, Klebsiella, enterobacter, Citrobacter*). The presence of these intestinal bacteria provides an indication of possible existence of the above mentioned infections but allows no conclusions to be drawn about the presence of other pathogens such as *Pseudomonas aeruginosa, Candida albicans* (yeast), *Staphylococcus aureus,* or *Streptococci.*

If in the near future statistics continue to prove that with increasing total microbial counts in coolants the number of pathogenic germs also increases, it will be necessary to consider setting a limit on the total microbial count. In 1978 the limitation of the total microbial count to $10^4$ $ml^{-1}$ was recommended by the UK Health and Safety Executive [14.135, 14.136, 14.137].

#### 14.3.7.2 Methods of Determining Microbial Count

The classic method is the plate casting method. After sterilization, a culture, such as blood agar or malt extract agar is applied in a thin liquid layer to a Petri dish and a small amount of diluted coolant is added. This specimen is incubated in a heated cabinet, frequently for 48 h at approx. 30 °C. The number of easily recognizable colonies multiplied by the degree of dilution, and divided by the amount of diluted specimen in milliliters, equals the microbial count $ml^{-1}$. Depending upon the culture, one can carry out selective measurements. For example, only fungi (mold and yeast and no bacteria) grow on malt extract agar with penicillin and streptomycin.

Easier methods for coolant studies in practical situations have been sought. As a result the membrane filtration method has been introduced in some laboratories, especially because of the good possibility for documentation. This method is preferred especially when routine drinking water tests are carried out in a laboratory.

Particularly suitable for examination of used coolants are the so-called dip slides. After immersing the dip slide in the generally non-diluted specimen to be tested, a thin film of fluid remains on the culture medium. These dip slides are then returned to their sterile, transparent, protective sleeve and often incubated for 24 h at 27 to 30 °C. The colonies formed in this way are compared with sample pictures to estimate the microbial count. This method is less accurate than the plate casting method but is adequate for monitoring the coolant. In the VSI technique (according to the Schweisfurth–Arnold method [14.47]) the dip slides are coated on both sides with culture media, whereby the bacteria count, the number of thread fungi and yeasts and hygienic-relevant bacteria (lactose positive enterobacteria) are determined on three selected areas. This method in particular has become well established in many coolant laboratories and especially also for coolant consumers, because of its easy handling, low expense and the great deal of information that it supplies.

Other methods, especially those that utilize the metabolism reactions of microorganisms by means of color indicators (Redox indicators), have been listed over and over again for coolant monitoring but have been unable to establish themselves because of their inaccuracy.

#### 14.3.7.3 Determination of the Resistance of Water-miscible Coolants Towards Microorganisms

The long, expensive and to some extent uncertain way to differentiate and assess the microbial stability of the coolant in practical use has led to development of appropriate laboratory methods.

Frequently, fresh emulsion is mixed for this purpose with a defined amount of used emulsion with a high microbial count. The microbial count development, and if necessary pH value, corrosion protective properties and stability, are observed over time. Such a testing method is described in ASTM D 3946 Standard Method for Evaluating the Bioresistance of Water-soluble Metalworking Fluids. Since fresh coolant containing biocides still has strong biocidal or biostatic properties, the effect of a one-off addition of germs to a used emulsion is not a particularly good indication of workshop practice. For this reason an inoculation cycle test has been developed by which a coolant flora or, through defined germs over regular intervals (for example, 4 days), a new inoculum is applied and the resistance of the coolant is observed by measuring the microbial count. If after inoculation there is no reduction in count, but rather, there is a further increase, the maximum inoculation cycle count has been achieved and this resistance can be described quantitatively. The Hemmhof test is also used in individual cases for coolants or coolant additives in the same way that it is used in conventional medical microbiology to produce antibiograms for antibiotics.

So-called flour tests are also carried out in which a defined amount of flour is added to the new unused coolant which is then inoculated with a known number of germs from used emulsions and the stability, pH value and especially the corrosion protective properties are observed over time.

In individual cases the bacterial stability of coolants has been defined in accordance with testing in the Warburg apparatus through biological oxygen demand (for example $BSB_5$). High oxygen demand is equivalent to good degradation and poor microbial stability.

#### 14.3.7.4 Reducing or Avoiding Microbial Growth in Coolants

Although the use of microbiocides in coolants is wide spread and has a considerable significance on formulation and monitoring of microorganisms, the selection of components which are very difficult for microorganisms to attack is equally important. In this context, there are considerable differences between emulsion systems and corrosion inhibitors. Table 14.12 demonstrates this for some of the products used in water-miscible coolants. Here the ease with which these substances are attacked by Pseudomonas bacteria, the most significant type of bacteria in coolants by number, is indicated by relative index figures [14.48].

**Tab. 14.12** Attack upon substances in coolant lubricants by Pseudomonas bacteria [14.48].

| *Raw material / component* | *Relative index figure for the ease of attack* |
|---|---|
| Petroleum sulfonates | 0 |
| Naphthenic acid (NZ = 100) | 7 |
| Mineral oil | 11 |
| Sodium naphthenate | 22 |
| Oleic acid | 197 |
| Sodium oleate | 234 |
| Triethanolamine salt of oleic acid | 253 |

Components used in coolants, apart from their normal tasks as corrosion inhibitors or emulsifiers, can also be biocidal in nature. The examination of a large number of alkanolamines has shown, for example, that they have very different biocidal properties [14.49].

The treatment of cutting fluids with ultraviolet radiation during use has not proved successful. A considerable barrier to the use of this method in coolants is the strong absorption in the 240–280 nm radiation range by coolant components. As a result, radiation in the correct wave length can only penetrate the coolant to a very limited extent. In the case of emulsions with medium or high mineral oil content, tests to irradiate in very thin layers entail great expense in energy and have not been successful. However, there have been references in literature from the USA [14.49a] recently which describe the successful use of ultraviolet radiation of one single wave length where the bacteria population can be drastically reduced within 24 to 48 h. It has been reported that ultraviolet radiation effects the DNA of the bacteria and as a result, reduces growth.

The biocide affect was satisfactory during tests with particle radiation [14.50]. However, up to now, no economical way has been found to fulfill the requirements of radiation protection [14.51]

The fact that, as with ultraviolet radiation, ozone treatment (ozonization) is used successfully in the sterilization of drinking water, has also caused ozone tests to be carried out over many years on coolants. This ozone treatment has not become established and is used in Europe in only a few factories. The reasons for this are summarized below:

- No adequate reduction of the microbial count occurred in emulsions despite higher amounts of ozone (for example, 100 g $h^{-1}$ ozone for a 10 $m^3$ central system with ozone introduced through packed contact columns.

- The ozone is not available for an adequate period of time for a biocide effect in emulsions and reacts with coolant raw materials. As a result the coolant is partially open to accelerated biological decomposition during static periods.
- Ozone is a very toxic gas; continuous control of the ozone remaining in the coolant and in the air is necessary. This is why, to some extent, in the case of swimming pool ozonization, the air is re-treated using active carbon [14.52].
- The costs of investments, monitoring and energy for an ozone system are high.

The thermal treatment of coolants for reducing the microbial count has also been extensively tested but deposits of coolant and water components in the heat exchangers, and the high energy costs, make this method appear impracticable.

Since in factories with machining processes such continually high temperatures are not feasible because of the impact on the cooling effect of the fluids and the negative effect on the climate in the workshops, an attempt has been made to kill off the germs by heating up the coolant for a short period to between 60 and 90 °C and cooling it down again afterwards. This process is similar to that used for pasteurization of milk and beer (killing off the pathogens by mild heat treatment, for example, 20 min at 60 °C, or sterilization, where the entire flora is killed by treatment at a higher temperature for a short period of time).

Treatment with specific copper complexes has been used successfully in large central systems in the USA for several years and this method is well-known under the name of Oxcedot. Some products were specially formulated to be treated during use with this method. Due to the strong waste disposal regulations in Europe and other countries concerning heavy metal content in waste water, this method was rejected in some countries especially since the costs for additional treatment before splitting were extremely high.

Irradiation of centralized systems in a German manufacturing facility for several months with X-rays from cobalt 60 clearly showed that it is, in principle, possible to reduce levels of bacteria by use of physical methods. When cost and health-and-safety requirements are taken into consideration, however, and this method is compared with treatment with biocides (chemical method), it must be stated that biocide treatment is more economical and less dangerous [14.138].

### 14.3.8
### Preservation of Coolants with Biocides

The handling of biocides is subject to a variety of regulations on a global basis and especially in Europe, where it is regulated by the EU Biocide Directive, Guideline 98/8/EG [14.139]. The EU Commission has published the Final Lists of Notified and Identified Biocides comprising product category 13 “Biocides for metalworking fluids”. The complete documents for the admission of products according to category 13 must be submitted to the Commission between February 1st and December 7th, 2007. It will only be known after that date which biocide chemistry will be legally permitted for metalworking fluids in the future in the European Community.

Biocides are used to reduce or avoid heavy microbial activity. The addition of such chemicals to the coolants (concentrates) or to water-mixed finished products is called preservation. The aim is to keep the coolants in an unaltered form for the longest possible period of time. Preservation agents should fulfill the following requirements:

- Effectiveness against as many types of microorganisms as possible (broad spectrum effective).
- Fast action: this has particular significance in the case of systems with high drag-out rates; products which act slowly are taken away with the coolant before they become effective.
- Long continuous effect: the biocide should not lose its biocide effect through reaction with other components or contaminants.
- High thermal stability.
- Low influence on pH value of the coolant: here there are different ways of assessing this. In many cases an alkaline biocide is desirable. In the case of coolants in use, which have clearly suffered a drop in pH value through microbial attack, it is expedient to use highly alkaline biocides for correcting purposes. Over and above this, alkaline biocides quickly form an essential part of the reserve alkalinity of coolants. A high alkali reserve at $pH < 9$ is generally favorable.
- Oil solubility: in many cases use in coolant concentrates (water-miscible coolant) is a basic requirement. The biocide is either dissolved or homogeneously dispersed in the concentrate. The use of biocides without solubility in the concentrate is restricted to the treatment of water-mixed coolants.
- No odor nuisance: no odor nuisance in workshops, both with regard to its own odor and by generation of odor by a reaction with microorganisms or components.
- Good skin compatibility: since biocides are generally based on a reaction with the proteins of the microorganisms, skin proteins are frequently subject to the risk of attack from these biocides. However, risk of dermatitis begins, as a general rule, in concentration ranges above the usual concentrations used in cutting fluids.
- Low toxicity: no toxic effects must occur when the biocides penetrate the body, either through the skin or through inhalation and swallowing of the coolant spray.
- No waste water hazard: biocides enter sewage/waste treatment plants through the waste water from the cutting fluids in such low concentrations that they can be biologically decomposed without the sludge flora being destroyed. The ability of biocides to be decomposed is necessary in biological clarification plants in order to avoid burdening public waters, especially through fish toxicity.
- High economy: the great expense of preservation of coolants mentioned at the beginning requires high economic efficiency in these products. Both the recommended concentration and the concentration applied in practice

> should be considered when comparing costs. For better practical handling, the dilution of the active substance should be selected by the biocide manufacturers so that the normal concentration used is between 0.1 and 0.2 % in water-mixed coolants.

Unlike the microbiocidal effect of the biocides, i.e. the germ killing effect, some preservatives also have a microbiostatic effect. In this case the germs are not destroyed, their growth and colonization are only suppressed. The use of microbiostatic properties is gaining more and more in importance when selecting emulsifier systems and corrosion inhibitors; the most common preservatives found in the trade stand out primarily for their biostatic effect.

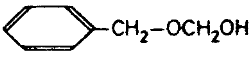
Not all preservatives have the same good microbiocidal effect on both bacteria and fungi. Products effective against bacteria are termed bactericides; products having a particularly good effect against fungi are termed fungicides. As with antibiotics in medicine, resistance can also build up towards preservatives. By this we mean the adaptation of microorganisms to the preservative, whose ineffectiveness then means that higher concentrations have to be used, at the least.

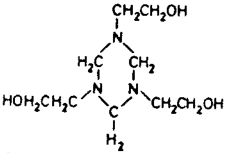
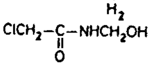
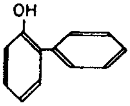
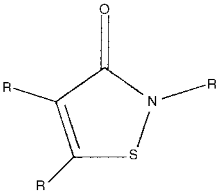
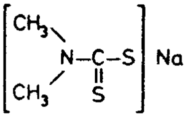
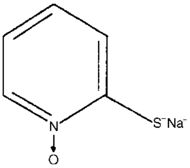
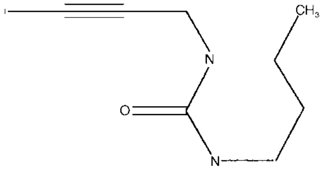
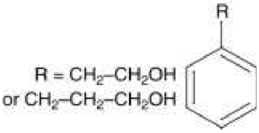
Generally, the coolant manufacturer can provide concentrates with or without preservatives. Technically, the best solution remains for the user to add the preservatives when he needs it. However, this can only be realized in the few cases which have large central circulation systems and good coolant monitoring. In such a case, it must not only be ensured that adequate preservative is added when filling a system for the first time, but also that the, in some cases considerable, top-up volumes are also preserved. For small and medium sized cutting fluid users the use of pre-preserved concentrates is still the best solution. When pre-preserving the concentrates, i.e., when mixing the preservatives in the water-miscible cutting fluids, special care must be taken to ensure compatibility with the other coolant components. In some cases the microbiocidal effect of the pre-preserved concentrate can be considerably reduced after long storage.

A large number of products are offered on the preservative market and selecting the right product is frequently difficult for the consumer

However, these products can be grouped into a comparatively few chemical classes. Table 14.13 summarizes a list of the most significant effective chemical groups with examples of their chemical symbols.

**Tab. 14.13** The most significant groups of substances used as preservatives, with examples of their chemical symbols.

| *Substance class (example)* | *Chemical symbols (examples)* |
|---|---|
| Aldehydes<br>(formaldehydes) | HCHO |
| O-Formals<br>alcohol + formaldehyde<br>(semi formal of benzyl alcohol) |  |

| ***Substance class (example)*** | ***Chemical symbols (examples)*** |
|---|---|
| N-Formals<br>amine + formaldehyde<br>(hexahydrotriazines)<br>methylbisoxazolidines |  |
| amide + formaldehyde<br>(*N*-methylchloroacetamide) |  $ClCH_2-\underset{\underset{O}{\parallel}}{C}-NHCH_2OH$ |
| Phenolic derivatives<br>(*o*-phenylphenol) |  |
| Isothiazoles<br>(benzoisothiazole)<br>chloromethyl, methyl, octylisothiazoles |  |
| Compounds derived from carbon disulfide<br>(sodium-dimethyldithiocarbamate) |  |
| Sodium pyrion |  |
| IPBC<br>(3-iodine-propinylbutylcarbamate) |  |
| Phenoxyethanol<br>Phenoxypropanol |  R = $CH_2-CH_2OH$<br>or $CH_2-CH_2-CH_2OH$ |

### 14.3.8.1 **Aldehydes**

Amongst these, only formaldehyde itself (formalin) has historical significance in cutting fluids; nowadays it is hardly used.

### 14.3.8.2 **Formaldehyde Release Compounds**

Currently this is the most significant group of biocides for coolants. The formaldehyde bearers formed by reacting formaldehyde with other substances are less volatile than formaldehyde itself. The countless reaction possibilities present opportunities of always finding ever new, suitable products for coolants. However, due to the high test costs involved (toxicity, environment etc.), the increasing number of legal requirements in Europe will mean that hardly any new active substances will be developed. The effectiveness of formaldehyde and formaldehyde release agents is based on the effect of the cell wall proteins of the microorganisms. The osmotic balance is destroyed as a result of hardening of cell walls which leads to cell death.

By converting formaldehyde using alcohols or glycols, oxygen formals, also called O-formals, are produced. One differentiates between semi and full formals, whereby semi formals are the most effective because of their low stability. O-formals are pH-neutral products and many of the neutral preservatives on the market are from this group of substances.

The so-called nitrogen or N-formals are produced from the reaction of amines and amides with formaldehyde. Found in this group are the most effective formaldehyde depots; these are hexahydrotriazine, imidazolidine, bis-oxazolidine, aminale and others. By combining other biocide partners with formaldehyde, products can be produced with a wide range of effectiveness. An important example is the result of the reaction between formaldehyde and chloroacetamide, which is also used as a bactericide in coolants. The resulting *N*-hydroxymethylchloroacetamide has a good bactericidal effect, but above all, a good fungicidal effect.

The many discussions concerning a cancer hazard from formaldehyde have not been proved in any of the epidemiological studies yet carried out [14.53].

### 14.3.8.3 **Phenol Derivatives**

These active substances are still seen as important products for coolant preservation. In particular, some examples of this group are well known for their efficiency against yeast and fungi. The toxicological objection to polychlorinated phenols, especially pentachlorophenol, has led to a general negative assessment of the group, although in some cases, there are also examples where there are no objections. So, for example, the LD 50 for *o*-phenylphenol with approx. 3000 mg $kg^{-1}$ and for *p*-chloro-*m*-cresol with approx. 1800 mg $kg^{-1}$ are relatively high.

Another phenolic compound, phenoxyethanol or phenoxypropanol was introduced in coolants in the 90s. Phenoxyethanol or phenoxypropanol has acquired particular significance as a stabilizing primary compound in boron and amine-free coolants and can be recognized by its typical odour.

The detrimental interaction between non-ionic surfactants and the phenolic preservatives is important for technical applications. The incompatibility of some substitution forms of phenolic derivatives with formaldehyde and the formaldehyde release compounds must also be considered. Total loss of biocidal effect can occur by combination of these two preservative groups.

#### 14.3.8.4 Compounds Derived from Carbon Disulfide

Another group of substances is derived from carbon disulfide by reactions with amines and formaldehyde. Amongst others, dithiocarbamate, benzoisothiazole and mercaptobenzothiazole are used in coolants from this group. Products of this group of active substances have both bactericidal and fungicidal effects.

#### 14.3.8.5 Isothiazoles

Very different results can be achieved using this material because of the specific reactivity with some raw materials found in coolant concentrates. To some extent these are very reactive and rapidly responding products as far as their biocide effect is concerned. Special care must be taken when adding the products to the coolants because of the considerable dermatitis risk presented by the highly concentrated substances.

#### 14.3.8.6 Fungicides

The use of sodium pyrion, with a predominately fungicidal character, has proved successful in coolants. One negative result observed is that when used with iron and zinc salts, sodium pyrion can lead to insoluble compounds which occur as black or white precipitation. Iron may be contained in, amongst other things, emulsifiers or other coolant components in low quantities (only a few ppm) due to the production process. A further fungicide compound which originated in wood protection impregnation is IPBC (3-iodo-2-propinylbutylcarbamate) which, because of its low toxicity, is accepted for use by the United States FDA (Food and Drug Administration). IPBC is found primarily in the re-preservation area since its stability in coolant concentrates is not ensured when amine, alkanolamide, etc. compounds are present..

#### 14.3.8.7 Hypochlorites

These salts of hypochlorous acid were sometimes used in the areas of preservation and disinfection because of their strong oxidizing effect. However, they are now rarely used.

#### 14.3.8.8 Hydrogen Peroxide, $H_2O_2$

The strong oxidizing effect, similar to that of hypochlorites, can reduce the microbial count quickly. However, as a result of this, the substances are only available for such a short time as a biocide that we cannot talk of actual preservation. This is why hydrogen peroxide, which is generally available as an aqueous solution with 30 to 60 % concentration, is frequently only used in connection with other biocides. In regular use, the damage to the coolant caused by oxidation of the components must be considered.

#### 14.3.8.9 Quaternary Ammonium Compounds

Because of their cationic character these products are only used occasionally in coolants. Anionic surfactants and these cationic products cancel out each others' effects. Importantly, anionic material not only comes from the coolant itself but is very frequently brought into the system from aqueous degreasing systems, and this possibility must be noted.

## 14.4
## Neat Cutting Fluids

Neat cutting fluids are mainly products such as honing oils, deep hole drilling oils, grinding oils and broaching oils which are not mixed with water prior to use. Neat cutting fluids have the following advantages over water-miscible fluids: they offer longer tool life and better surface finishes for difficult operations performed at low cutting speeds, the maintenance of the oil is much less complicated and the life of the oil is significantly longer. No corrosion problems occur with chlorine-free metalworking oils and such problems stemming from products containing chlorine can be overcome with suitable additives. The contamination of hydraulic and slideway oils with neat oils is less of a problem than with water-miscible products. Moreover, leaks of hydraulic oils and other machine lubricants are easier to cope with if compatible oils are used. New technologies which allow the harmonization or matching of cutting and machine oil additives have made significant contributions to cutting manufacturing costs [14.54].

Their principal disadvantage compared to water-miscible fluids is the poorer cooling offered by oils. This is particularly the case in high speed cutting operations. An exception is high speed grinding where the superior lubricity of oil reduces the amount of heat generated. Another disadvantage is the combustibility of oils as well as the explosion risks of oil mist and vapor. Protection measures against fire and explosion hazards are another cost factor for users. As the viscosity of oils is higher than water-miscible metalworking fluids, drag-out losses on chips and components are also higher and this is mostly felt when tanks have to be topped-up or in the contamination of washing lines. However, this disadvantage can be neutralized by the efficient de-oiling of components, grinding sludge and chips [14.55].

### 14.4.1
### Classification of Neat Metalworking Oils According to Specifications

Neat metalworking oils are classified according to ISO 6743/7, DIN 51 385, or DIN 51 520 on the basis of the additives used (Table 14.14).

**Tab. 14.14** Classification of neat metalworking oils according to ISO 6743/7, DIN 51 385 or DIN 51 520.

| | *DIN 51385 Code letters* | *ISO 6743/7 Code letters* |
|---|---|---|
| Metalworking oils containing mineral oils without anti-friction additives or EP agents | SNO | MHA |
| Metalworking oils containing anti-friction additives | SNP | MHB |
| Metalworking oils containing EP agents, chemically inactive | SNPA | MHC |
| Metalworking oils containing EP agents, chemically active | SNPB | MHD |
| Metalworking oils containing anti-friction additives and EP agents, chemically inactive | SNPC | MHE |
| Metalworking oils containing anti-friction additives and EP agents, chemically active | SNPD | MHF |

### 14.4.2
### Composition of Neat Metalworking Fluids

#### 14.4.2.1 Base Oils and Additives

Most metalworking fluids are based on petroleum hydrocarbons and contain additives to improve lubricity, wear protection and other additives to manipulate secondary characteristics such as foaming, misting, corrosion protection, dispersion, oxidation stability and flushing. Of the petroleum hydrocarbon base oils, low-aromatic, paraffinic solvent extracted or hydrocracked products are preferred. Recent trends in Germany are heavily focused on ester based fluids.

Neat metalworking fluids with no additives are seldom used these days. If at all, they are used for simple machining operations on easy-to-machine materials such as cast iron and nonferrous metals.

Many oil manufacturers use the rough neat oil classification of sulfur-active and sulfur-inactive (copper-active, copper-inactive). This classification is necessary when oils are used for a wide range of applications. Examples include multipurpose oils used for all sorts of materials, and also for hydraulics and other machine lubrication requirements.

Greater amounts of additives normally increase the cutting performance of a metalworking oil. However, this is no truism because the effect and combination of additives have a considerable influence on machining results. Judging cutting and grinding oils according to their sulfur, phosphorus or chlorine content can lead to totally erroneous evaluations. Classifying metalworking oils according to the various additive groups in the structure shown in Section 1 is a highly complex and separate subject. As a result, only the most important EP additives (specified in detail in Chapter 6) will be dealt with here.

Apart from sulfurized fatty oils and polysulfides, sulfur-phosphorus compounds and pure phosphorus compounds are often used as EP additives in advanced cutting

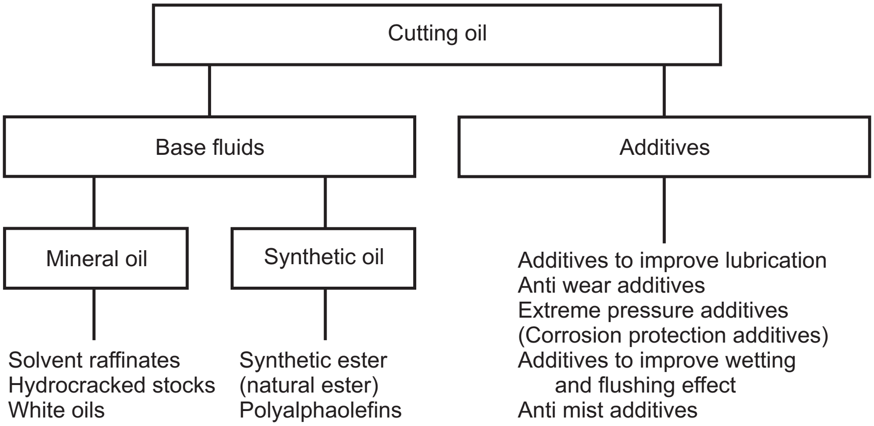


**Fig. 14.16** Composition of neat metalworking oils.

oils. The use of zinc- and other heavy metal-based additives in cutting oils is declining due to environmental and waste water considerations. Chlorinated paraffins are still used as universal EP agents throughout the world but combinations of sulfur additives and ester oils are being increasingly used in Germany and Western Europe as substitutes. The reasons for these developments are the considerably higher disposal costs for products containing chlorine in Germany (Fig. 14.16) [14.140].

#### 14.4.2.2 **Significance of Viscosity on the Selection of Neat Products**

The viscosity of most metalworking oils is between 2 and 46 $mm^2\ s^{-1}$ at 40 °C. The very low viscosities are used for spark erosion applications (EDM) and for honing and super-finishing while the higher viscosities are mostly used for difficult machining work at low cutting speeds such as broaching and gear cutting.

In general, high cutting forces, continuous chips and large chip cross-sections require higher viscosities. These higher viscosities also have a positive effect on the evaporation and misting behavior of the oils. However, the disadvantages of higher viscosity oils are higher drag-out losses on chips and components and the resulting increased pollution of washing lines. For this reason, the current trend is towards low viscosity products. The disadvantages of low viscosities, i.e. lower flashpoints as well as greater evaporation and misting can be compensated for by new hydrocracked base oils or ester oils.

Small components also normally require low viscosities because thicker oils can cause parts to stick together and thus cause transport and positioning problems. Thick oil films can also interfere with monitoring of routine manufacturing tolerance and here again, low viscosity oils are the only solution if component de-oiling is to be avoided. Low viscosity oils offer better heat dissipation and this is a clear benefit in processes which create high heat such as high-performance grinding. Also the better flushing properties of low viscosity oils make then ideal for honing, grinding and lapping operations which require the efficient removal of abraded material from the cutting zone for perfect results. Deep hole drilling also benefits from low-viscosity oils because chips are more rapidly flushed out of the hole. The other advantages of low viscosity oils relate to machinery peripherals. For example, the necessary size of belt filters falls exponentially with viscosity so that both costs and space can be saved.

### 14.4.3
### Oil Mist and Oil Evaporation Behavior

Hygiene and dermatological problems occur in the metalworking industry when machine personnel are subjected to repeated skin contact with metalworking oils. Chip-forming metalworking is particularly affected because of the regular and close contact between personnel and the machines. An overall reduction in the number of dermatological problems and a general reduction in the oil pollution of workshop atmospheres was achieved by mechanical barriers on machines as well as changes to manufacturing technologies, in particular, the automation of machining processes in transfer lines. The pollution of workshop air with oil mists and vapors is still a problem in many companies. Measures such as machine encapsulation, improved machining procedures, automation and above all, extraction at the machine or at a central point in the workshop have certainly improved the situation. However, extraction without de-oiling simply shifts the pollution outdoors and this is in conflict with general environmental protection measures.

Many attempts have been made to reduce not only the misting properties of straight oils but also the misting of water-mixed coolants [14.141, 14.159]. Initial reduction of misting has been achieved but the effect could not be maintained over an 8-h working shift. Two effects must be mentioned – the mist-suppression effect gradually decreases as the molecular weight of the polymer is reduced and the polymer will be filtered out and is, therefore, no longer available for mist suppression. This means, in practice, that permanent addition of a suitable polymer is necessary to reduce the misting of a water-mixed coolant in workshops.

Further studies show that total inhalable particulates can be used to estimate mineral oil mist exposure but cannot be used for water–mix MWF concentrate mist exposure [14.160].

#### 14.4.3.1 Evaporation Behavior

The evaporation of cutting fluids on hot components, tool and chips and from the larger surface area of oil mist droplets leads to vapor pollution of workshops and even to condensed mists. As low-viscosity oils become more popular, the evaporation behavior of base oils is becoming increasingly important. This subject is covered in detail in Chapter 4.

While a number of countries specify an oil mist threshold value in mg m$^{-3}$ (particulates), Germany has in recent years moved over to evaluating the total hydrocarbon concentration in the air. In most metalworking factories, this is 5 to 20 times higher than the oil mist concentration.

#### 14.4.3.2 **Low-Misting Oils**

The development and application of low-misting neat cutting oils have significantly improved the oil mist pollution situation. Development work as well as the general acceptance of these oils in the metalworking industry was significantly influenced by new methods of measuring oil mists and of determining the misting characteristics of oils [14.56–14.58].

#### 14.4.3.3 **The Creation of Oil Mist**

If an oil mist is analyzed, a variety of different factors are involved. lt is soon apparent that the whole process is highly complex in which mechanical, physical and physico-chemical factors are intermixed.

The causes of neat cutting oil mists can be roughly listed as follows:

- As the fluid exits the nozzle, air friction acts on the jet in proportion to its exit velocity. Important factors are the geometry of the nozzle and the exit speed. The result is that droplets of oil are dispersed into the surrounding air.
- If the oil jet rebounds off the machine bed, the component or tool, oil mist can be formed. This is greatly influenced by the oil pressure but also the quantity. The amount of oil mist with droplet sizes $< 5$ μm increases dramatically with increasing pressure [14.59].
- Mechanical stress on the oil during the machining process also creates oil mist. Critical factors here include the machining speed, the geometry of the rotating machine parts and component, chip formation, the quantity of oil and the oil pressure. Oil misting is a major problem in machining processes which use geometrically non-defined cutting edges, i.e. particularly grinding. The porosity of the grinding face allows oil to be thrown-off at high peripheral speeds and form a dispersion.
- When the oil is returned to the tank, air can be trapped in the oil. As the air is released, it carries out oil in the form of a finely-dispersed mist. Important factors here are the geometry of the jet which impacts the surface of the fluid in the tank and the velocity of the jet.
- Particularly if high oil pressures are used and depending on the pressure-related solubility equilibrium, larger volumes of air can be dissolved in the oil. When the pressure falls, the air is released from the oil. This escaping air can transport oil droplets to the workshop air. The decrease in the solubility of air in oil as the temperature of the oil increases causes air to be released from the oil as the temperature of the oil increases in the cutting zone.
- Apart from the above-mentioned aerosols, metalworking and other processes can cause condensation aerosols to form. A large part of the energy consumed by the machining process is converted into heat and this can lead to very high tool and component temperatures. This, in turn, can cause a partial evaporation of the oil. This evaporation process can continue beyond the cutting zone insofar as oil can still evaporate on the hot machining chips. Shortly after this evaporation, the vapor cools somewhat and condenses. This sequence can create condensation aerosols with very fine droplet sizes. Apart from the surface temperature of

the wetted components, the surface itself, the thickness of the oil film and last but not least, oil-specific factors such as its vapor pressure also effect the creation of oil mist. Outside influences such as the amount of dust and moisture in the air influence condensation. In a simple laboratory misting test, an oil mist with a pronounced maximum droplet distribution at 1.2 μm was examined [14.60].

### 14.4.3.4 Sedimentation and Separation of Oil Mists

All the above-mentioned possible causes of oil mist can create mists with a very large spectrum of droplet size distributions. Immediately after creation, oil mists begin to collapse. While droplets which are much larger than those in the mist itself normally precipitate in the immediate vicinity of the machine, smaller droplets can spread much farther if they have enough kinetic energy. A major factor in the precipitation of an oil mist is the droplet size. Suspended oil droplets agglomerate until they reach a maximum diameter of 3 μm when they begin to sink slowly (0.5 $m\ h^{-1}$ at 2 μm diameter) [14.61]. Sedimentation can also be accelerated by coagulation. Air flow conditions and Brownian movement of smaller droplets can cause droplets to collide and thus grow by coagulation. And finally, small and large oil mist droplets can undergo an interchange caused by surface-activated evaporation and condensation.

In plants in which chip-forming machining is performed with neat cutting oils, the oil mist in the workshop atmosphere can have droplet sizes up to a maximum of 3 μm [14.61]. The maximum numeric distribution is about 1 μm. The maximum mass distribution is naturally near to the upper droplet size limit if one assumes that the mass of a droplet increases by the third power of its radius. The average droplet size measured at various points on a centerless external grinding machine was about 95 % < 3 μm [14.61].

### 14.4.3.5 Toxicity of Oil Mist

An oil mist is a dispersed system with droplet sizes between 0.01 and 10 μm. This very wide range of sizes justifies the term, polydispersed system. The toxicological evaluation of oil in the air in the form of gas should be viewed totally differently to the evaluation of oil mists.

Research on aerosols and dusts has shown that only particles or droplets smaller than 5 μm can reach the lung's alveoles. Larger droplets are filtered out by the nose or are trapped in the bronchial tubes and made relatively harmless for the body's organism [14.62]. Medical research on silicosis has shown that (as far as dust is concerned), particle sizes of 0.5 to 1.5 μm are efficiently retained, i.e. these particle sizes are effectively trapped by the alveoles in the respiratory system. Hydrocarbon oil mists tests on animals [14.63] have shown that this droplet size range is particularly important. Most oil mist tests have examined mineral oil hydrocarbons without additives. These are viewed chemically as relatively inert substances and this is mostly the case if one disregards the above-mentioned discussion about aromatic hydrocarbons or the tests performed on highly condensed aromatics. Nevertheless, toxicological evaluation of pure hydrocarbon mists is still extremely complicated. If lubricant additives are included in the evaluation, which are a much more reactive group of substances than hydrocarbons alone, generalized statements on the toxicity of neat cutting oils would be almost impossible. This proviso must however form the focus of the health hazards of oil mist.

According to Reiter's retention charts [14.64], the lung's alveoles only retain about 10 % of oil droplets with a diameter of 0.1 µm but 70 % with a diameter of 1 µm. This should be noted when oil mists are evaluated even if the toxicological details are not available.

As was the case with dust contamination, these medical considerations led to the creation of oil mist thresholds. The last time oil mists were mentioned in the German MAK (Maximum Workplace Concentration) list [14.65] was 1966. However, uncertainties regarding the toxicological evaluation of oil mists was the reason why MAK values were not established for oil mist.

Some countries set the limit at 3 mg $m^{-3}$ (and 5 mg $m^{-3}$ for longer exposure). The American TLV list (Threshold Limit Values) contains a threshold of 5 mg $m^{-3}$ oil mist (particulates) suggested by the 1973 Conference of Government Industrial Hygienists. This threshold was based on animal tests with a naphthenic white oil (molecular weight: 350 to 410) with no additives or aromatics [14.66]. lt is therefore particularly significant that the animal tests were oriented to oil mist droplet sizes found in practice. The maximum droplet size distribution was about 1.3 µm (90 % of the droplets were < 1.6 µm). This droplet size distribution roughly corresponds to that of mists found in metalworking shops and also to the type of mists which are toxicologically important. Without doubt, aromatic-free white oils are much less problematic than the mineral oil cuts normally used in cutting and grinding oils. Above all, the effect of additives was excluded.

Two OSHA air contaminant permissible exposure limits currently apply to metalworking fluids. These are 5 mg $m^{-3}$ for an 8-h time-weighted average (TWA) for mineral oil mist and 15 mg $m^{-3}$ (8-h TWA) for particulates not otherwise classified (PNOC) (applicable to all other metalworking fluids) [14.142]. There are no other requirements.

There are also other recommended exposure limits. In 1998 the National Institute for Occupational Safety and Health (NIOSH) published a criteria document which recommended an exposure limit (REL) for metalworking fluid aerosols of 0.4 mg $m^{-3}$ for thoracic particulate mass as a time-weighted average (TWA) concentration for up to 10 h per day during a 40-h working week. Because of the limited availability of thoracic samplers, measurement of total particulate mass is an acceptable substitute. The 0.4 mg $m^{-3}$ concentration of thoracic particulate mass approximately corresponds to 0.5 mg $m^{-3}$ total particulate mass. The NIOSH REL is intended to prevent, or greatly reduce, respiratory disorders causally associated with exposure to metalworking fluid. It is NIOSH's belief that in most metal-removal operations it is technologically feasible to limit metalworking fluid aerosol exposure to 0.4 mg $m^{-3}$ or less [14.143].

The American Conference of Governmental Hygienists (ACGIH) threshold limit value (TLV) for mineral oils is 5 mg $m^{-3}$ for an 8-h TWA and 10 mg $m^{-3}$ for a 15-min short-term exposure limit (STEL). In 1999 the OSHA Metalworking Fluids Standards Advisory Committee also recommended a new 8-h time-weighted average permissible exposure limit (PEL) of 0.4 mg $m^{-3}$ for thoracic particulates (0.5 mg $m^{-3}$ total particulates). The committee based the recommended PEL on studies of asthma and reduced lung function.

In Germany, the toxicological evaluation has been based in recent years on the total hydrocarbon concentration in the atmosphere (which means the total of oil mists and vapor). The threshold value currently used (regulation officially not valid, February 2006) is set at 10 mg $m^{-3}$, which applies both to water-miscible and neat cutting fluids. Because of the diversity in measurement methods, many TLV (threshold limit values; MAK values) have been established; all are based on the same dimension of mg $m^{-3}$, but the results can never be compared with each other. As an example, if the method used in the USA gives a result of 0.5 mg $m^{-3}$ the method applied in Germany under identical conditions leads to a result which is sometimes more than 30 mg $m^{-3}$. This is because the method used in the USA measures particulates in a specific size range only whereas the German method detects aerosols and hydrocarbon vapor of different droplet size [14.144].

### 14.4.3.6 **Oil Mist Measurement**

Determining the total hydrocarbon content of air is possible with good accuracy using carbon tetrachloride solvent washing with subsequent infrared spectroscopic examination of the CH-valence shifts [14.67]. These measurements do not analyze oil mist concentration because the total hydrocarbon content in workshop atmospheres in the metalworking industry can be many times greater. Most suitable of all is a scattered light spectrometer which can be adjusted to measure the droplet sizes which are relevant to the retention characteristics of the lung's alveoles. This method ensures that the toxicologically relevant oil mist is measured [14.56, 14.161]. This measuring method is simple and fast and can be used in all areas of a workshop. For comparative measurements of an oil's misting behavior, a scattered light spectrometer is installed in a unit which is schematically described in Fig. 14.17 (Fuchs procedure) [14.68].

Air, at a given pressure, volume and temperature, is blown into the oil. This creates an oil mist which is measured by the scattered light spectrometer over time. Figure 14.18 illustrates the influence of viscosity on the oil mist characteristics of a series of neat cutting oils.

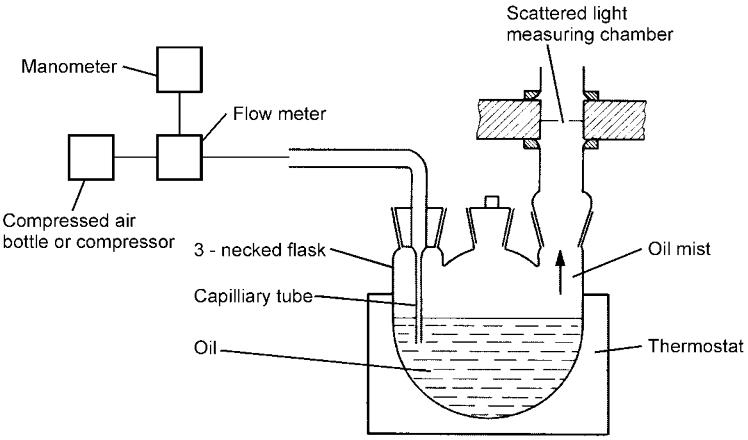


**Fig. 14.17** Measuring apparatus for determining the misting characteristics of cutting and grinding oils.

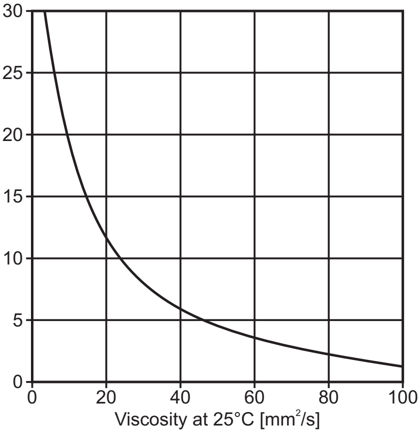


**Fig. 14.18** Influence of viscosity on the oil mist characteristics of a series of neat cutting oils.

### 14.4.3.7 Oil Mist Index

To give the oil misting characteristics of metalworking oils a figure, the oil mist concentration in mg $m^{-3}$ is determined in line with defined machining conditions and this oil mist concentration set against that of a reference fluid. This ratio has no dimension but is multiplied by 100 to provide the Oil Mist Index. If the reference fluid is di-*n*-octyl phthalate or di-*iso*-octyl phthalate, whose misting behavior is similar to that of a standard cutting fluid at a given viscosity, an Oil Mist Index as in Fig. 14.19 results. Figure 14.19 shows values for standard and low-misting oils and the viscosity of the oils. The oil mist index of medium viscosity standard oils (40 $mm^2\ s^{-1}$ at 40 °C) is between 80 and 120 but between about 4 and 6 for low-misting versions. This provides a possible definition of low-misting oils. For low-misting oils with a viscosity > 30 $mm^2\ s^{-1}$ at 40 °C, the index is < 10. According to Fig. 14.19, greater differentiation must be made for oils with lower viscosities [14.57].

### 14.4.3.8 Oil Mist Concentration in Practice

Back in 1978, Fuchs [14.69] performed a far-reaching study in Germany to determine the oil mist concentrations near to machine tools using neat cutting oils. Table 14.15 shows values from 350 measuring points in 65 companies for standard and low-misting oils. The large deviation in values was mainly due to varying machine-specific counter-measures (encapsulation, extraction etc.). Figure 14.20 shows the oil-mist concentration in a machining shop over a period of 12 h (only neat cutting oils via a central system). Apart from the positive effect of low-misting oils, the extraction system's effect on reducing the oil mist concentration can be seen. Fig. 14.21 shows the high sensitivity of the scattered light spectrometer and the effect of low-misting oils on a Gleason gear cutting machine. Each tooth cut by the machine can be identified by the pattern of oil mist concentration and the time scale. Such measurements also allow the cutting oil feed to be optimized in terms of oil mist pollution. Such measurements and mist concentration profiles are also of great assistance when extraction equipment is installed and/or monitored.

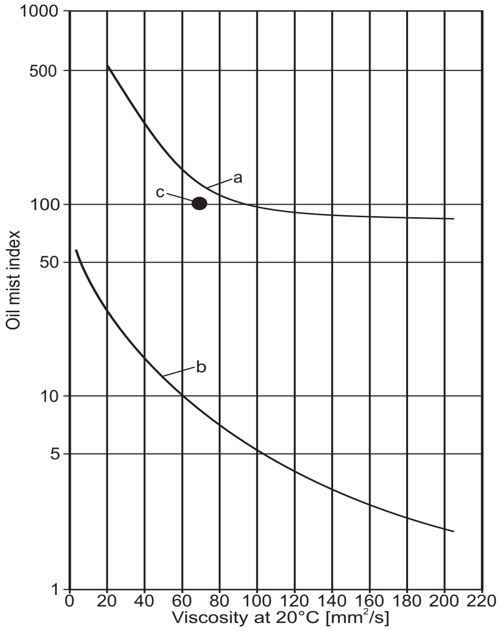


**Fig. 14.19** Dependence of the oil mist index (*NI*) on viscosity. (a) standard oils (not low-misting); (b) low-misting oils; (c) value for dioctyl phthalate (DOP, according to definition). $NI = k_p/k_r \times 100$; $k_p$ = mist concentration of the oil under test, mg $m^{-3}$; $k_r$ = mist concentration of the reference oil (DOP), mg $m^{-3}$.

**Tab. 14.15** Values from 350 measuring points in 65 companies for standard and low-misting oils.

| *Type of oil* | *Standard oils, mg $m^{-3}$* | | *Low-misting oils, mg $m^{-3}$* | |
|---|---|---|---|---|
| ***Measuring point*** | ***Range*** | ***Average*** | ***Range*** | ***Average*** |
| Workshop locations, various machining ops; points at some distance from machines; walkways between machines (> 1.50 m) | 1.6 to 0.3 | 0.95 | 0.9 to 0.2 | 0.31 |
| Hobbing; head-hight, < 1.5 m from the machinig point | 40 to 3 | 18.1 | 21 to 0.2 | 7.9 |
| Grinding; head-hight, < 1.5 m from the grinding point | 99 to 2 | 19.4 | 21 to 0.2 | 6.9 |
| Automatic lathe; Type I, head-hight, < 1.5 m from the machining point | 95 to 2 | 13.3 | 24 to 0.8 | 5.2 |
| Automatic lathe: Type II | 72 to 1.5 | 6.3 | 7 to 1.2 | 3.1 |
| Automatic lathe: Type III: head-hight, < 1.5 m from the machining point | 34 to 10.8 | 10.1 | 9 to 0.3 | 2.9 |

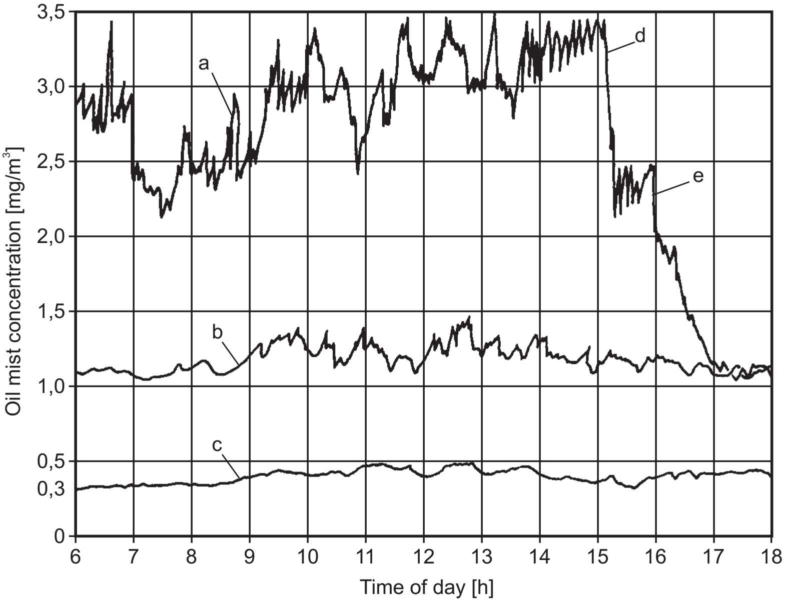


**Fig. 14.20** Oil mist concentration in a machining shop over a period of 12 h. (a) conventional neat cutting oil without extraction; (b) conventional neat cutting oil with extraction; (c) anti-mist cutting oil with extraction; (d) basic extractor switched on; (e) complete extraction system switched on.

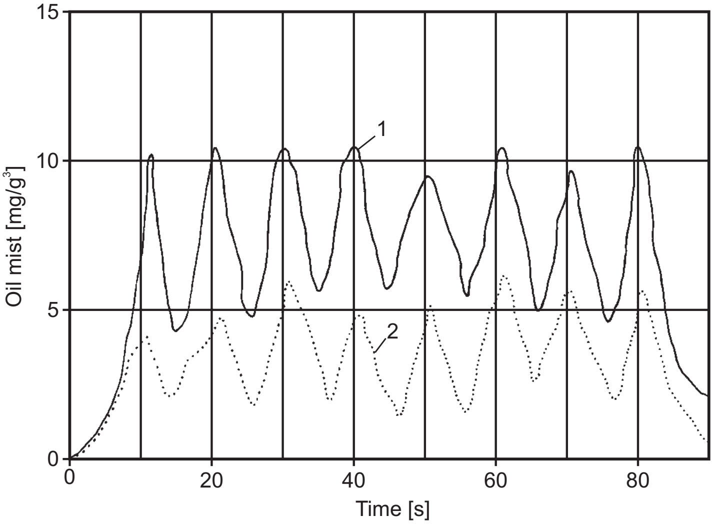


**Fig. 14.21** Gear cutting on Gleason machines. 1, conventional neat cutting oil; 2, equiviscous anti-mist neat cutting oil.

## 14.5
## Machining with Geometrically Defined Cutting Edges

### 14.5.1
### Turning

With a turning operation it is possible to produce parts which are round in shape. Cylindrical and conical surfaces are generated from a rough cylindrical blank which rotates about its longitudinal axis and against a cutting tool.

Most turning operations use single-point tools. Normally, a tool holder contains an indexable insert with multiple cutting edges.

The speed of the workpiece, the feed rate of the tool, and the depth of cut are main specifications for turning operations. Roughing cuts, which remove metal at the maximum rate, are followed by finishing cuts at a higher cutting speed, a lower feed rate, and a smaller depth of cut [14.70]. These machining conditions depend on the workpiece and tool materials, the surface finish, the dimensional accuracy, and the machine tool capacity. Each combination of workpiece and tool materials has an optimal set of tool angles. Tool geometry will affect the direction of chip flow. The objective is to avoid long continuous chips which can interfere with the machine tool operation or damage the part surface. Chips will break when they hit the workpiece or tool holder.

- The major turning processes performed on a lathe are straight or cylindrical turning, taper turning, facing, and boring.
- Turning operations occur on the external surface of a part whereas facing is used to produce a flat surface.
- Boring is an internal turning process and may be done to enlarge a hole made by a previous process, to enlarge the inside diameter of a hollow tube, or to machine internal grooves.
- For turning operations, water-mixed metalworking fluids are used only when running automatic lathes; neat oils are often preferred.

### 14.5.2
### Drilling

The most common shape found in a manufactured part is a circular hole, and many of these holes are produced by drilling. Since the chips are formed within the part, the flutes or grooves normally serve two purposes. In addition to providing a conduit for the removal of chips, they also allow the cutting fluid to reach the tool–workpiece interface. Most drills are made from HSS which demands the application of a cutting fluid [14.71].

The coolant pressure recommended in drilling depends on several factors. The most important factors include the following: workpiece hardness, feed rate, hole diameter, depth, tolerance, and finish. As the coolant pressure is increased, recirculating the coolant could become a problem [14.72].

Drilling times can be reduced by using internally cooled drills with inserts. Tungsten carbide inserts and inserts with PVD or CVD coatings are commonly used in these applications. An efficient coolant system supplies that coolant at the proper pressure and flow rate. An inefficient coolant system can lead to poor surface finish inside the drilled holes. The drill diameter is the most important factor in determining the coolant pressure and flow rate [14.73]. Chips can block the channels that deliver the coolant to the inserts if the coolant system is inefficient. This condition can cause tool breakage.

Water-mixed metalworking fluids are used where high cooling properties are required. Recent developments show that this operation may be run also with MQL (Minimum Quantity Lubrication) or by using dry cutting technology. Both techniques depend on the material machined and the machine tool designed for this application.

### 14.5.3
### Milling

A variety of milling processes is available where end milling, slab milling, and face milling are included. High metal removal rates are possible since the tools have multiple teeth and each tooth produces a chip. In most applications, the workpiece is fed into a rotating tool. The feed motion usually is perpendicular to the tool axis, and cutting occurs on the circumference of the tool.

In end milling, the axis of cutter rotation is perpendicular to the workpiece surface to be milled. End mills commonly have two, three, or four flutes.

Flat surfaces, recess cuts for making dies, grooves and profiles around thin parts are common operations for end mills.

Hollow end mills are used on automatic screw machines. The internal cutting teeth will machine a cylindrical surface to an accurate diameter from round bar stock.

Face milling is similar to end milling. However, a face-milling cutter is relatively large in diameter compared to its length. Face-milling cutters are designed to machine flat surfaces. The geometry of the cutter and the major application are appropriate for carbide inserts which can be indexed. Due to the relative motion between the cutting tool and the workpiece, tool marks, such as those produced in turning, are found on face-milled surfaces also.

The direction of cutter rotation in milling can produce different effects. In the traditional method, called up milling or conventional milling, the tool rotates against the direction in which the workpiece is fed. In down milling or climb milling, the cutter rotates with the feed direction. Up milling produces a chip that gets thicker, but the chip gets thinner in down milling.

The major advantage of up milling is that tool wear is not affected by the surface condition of the workpiece. A smoother surface is possible if the cutting edges are sharp.

When down milling materials with a hard surface, such as castings and hot-worked metals, the teeth will wear faster and can be damaged. Advantages of down milling include a reduced tendency to have tool marks on the part and a surface finish that is not affected by a built-up edge if one is created on the cutting edge.

When machining cast iron, dry milling has been state-of-the-art for many years. High tensile steels and aluminum alloys are lubricated with water-mixed metalworking fluids but recent developments show that MQL can in some cases provide further advantages from a cost perspective.

### 14.5.4
### Gear Cutting

The most important machining operations are gear hobbing, gear shaping and gear shaving. The different forms of teeth and various types of machines with different tool kinematics very frequently lead to different oil recommendations by the machine tool maker. Nevertheless, in this case the users very clearly want to have a unique product for some gear cutting applications. Today in large serial automobile gearbox production this has already been generally achieved and all hobbing and generating shaping operations are carried out with just one oil. Broaching is a competitive application to run with hobbing cutters and shapers in the production of external toothing and should be mentioned here; uniform metalworking fluid generally presents no problems.

As far as tool cooling is concerned, the tool cutting edges only cut for a comparatively short time and sufficient time is available for cooling. As a consequence sufficient cooling is provided by neat cutting oils. However, the use of neat oils is called for because of the high standards set for a high quality surface finish; the high quality surface finish becomes a prime consideration particularly when the gears are not to be precision shaped afterwards by shaving. Although today water miscible coolants are available and can achieve good results, the majority of machine tools for hobbing and shaping are not designed for water miscible products. The latest trends are towards dry machining and the basis for this is coated tools and chip transportation with compressed air.

Should the quality of surface finish not be adequate when hobbing and shaping, then precision machining may be used to ensure finished gears attain correct load carrying capacity and quiet running. In this context gearwheel shaving and more recently even gearwheel rolling have become established operations. Oils with approximate 40 $mm^2 s^{-1}$ at 40 °C are particularly suitable for gear shaving with a higher module ($m > 2.5$); recommended for smaller modules are less viscous oils with viscosities between 15 and 25 $mm^2 s^{-1}$ at 40 °C.

Gearwheel rolling is not a cutting operation but the area of application is so similar to that of shaving that the requirements for the cutting oil performance in this case have to be considered the same. The surface is smooth and minimum material is displaced by the rolling and sliding motion of the rolling wheel under high surface pressure on the gear flank. This, in turn, can lead to burring. When using oils for this forming operation they should contain polar additives. As a general rule oils for shaping and rolling can be the same at least for gearwheels with a larger module if the additives are balanced.

In the case of very sensitive surfaces, when precision machining of gearwheels sometimes corrosion problems can occur as a result of additives containing chlorine. However, this can be remedied by adding suitable corrosion protection concentrate but in this case it must be ensured that the metal removing capacity of the oils is not reduced.

## 14.5.5
## Deep Hole Drilling

Current deep hole drilling techniques, previously employed for the most part only in the armaments industry (hence the term 'gun drilling' which is often encountered), have been steadily modified and are now concentrated to a greater extent in civil applications.

These techniques are now used in the automotive, shipbuilding, aircraft, machine and tool industries, in industrial plant construction and for hydraulic and pneumatic equipment.

The borderline between conventional drilling using HSS twist-drills and the various special-purpose drills such as deep hole drilling tools or short hole drills equipped with indexable tungsten carbide inserts can best be defined as follows:

- if the holes are shallower than 2 to 3 times the diameter (*d*), and on the assumption that powerful machining centers of great rigidity are available for the purpose, drilling with indexable tungsten carbide inserts is likely to become the most popular method in the future.
- this method gives lower feed and cutting forces than twist drills. On account of the high cutting speeds (5 to 10 times higher than with twist drills), however, correspondingly more powerful drives are needed.
- for holes deeper than 10 × *d*, and in cases where a better quality surface finish, reduced surface irregularity and closer dimensional tolerances are called for, deep hole drilling tools are used.
- in between these two areas, special designs of conventional HSS twist drill are employed.

### 14.5.5.1 Deep Hole Drilling Methods

A distinction is made between three drilling methods, which are known as solid, trepanning and counter-boring methods. Drilling from solid means cutting out the entire volume of material representing the hole to be drilled. Core drilling (trepanning) cuts away only an annular area, leaving the solid core untouched. Counter-boring-diameter a second operation, at the same time improving the surface finish and accuracy of the hole. Counter-boring processes include sciving and finish-rolling or burnishing. The three best-known deep drilling methods, BTA (the initials of the Boring and Trepanning Association), ejector and gun drilling, all have different forms of special cutting fluid supply.

#### Gundrilling System (Fig. 14.22)

The gun drill has internal cutting fluid supply and external chip flow. The cutting fluid is pumped at high pressure through the oil duct to the tip to lubricate the cutting edge and support pads. Because of its high velocity it carries the chips back through the V-shaped groove.

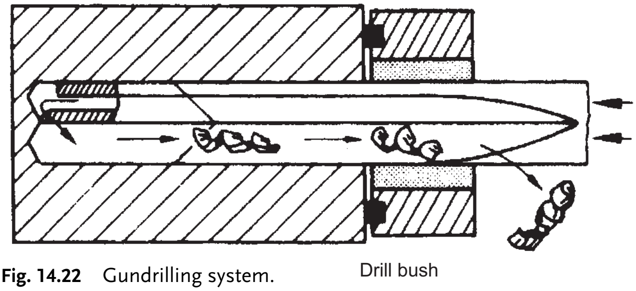


**Fig. 14.22** Gundrilling system.

**Ejector system (Fig. 14.23)**
The Ejector system has an external cutting fluid supply and an internal chip flow. The fluid is pumped between the drill tube and inner tube to the drill head. Most of the fluid is forced through holes in the drill head and cools and lubricates the support pads and cutting tips. The remainder is forced through the nozzle in the inner tube and diverted back to the outlet. This creates a partial vacuum in the inner tube so that the fluid which has performed the lubrication and cooling is sucked into the inner tube together with the chips, and passed to the outlet.

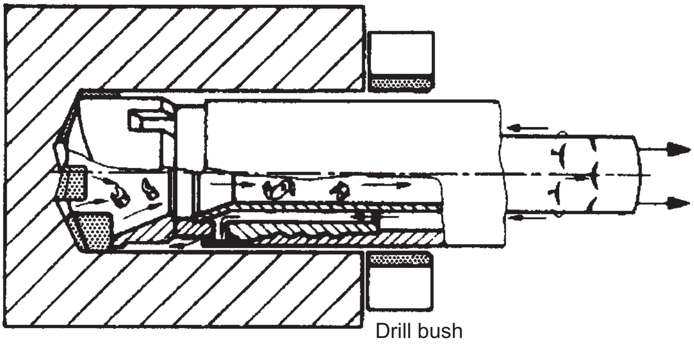


**Fig. 14.23** Ejector system.

**BTA system (Fig. 14.24)**
The BTA (or STS) drill has external cutting fluid supply and internal chip flow. By forcing the cutting oil between the drill tube and the hole being drilled, the velocity of the fluid becomes so high that it is able to provide effective transportation of the chips through the drill tube and the drilling spindle back to the outlet.

### 14.5.5.2 **Tasks to be Fulfilled by the Cutting Fluid**

As one of the process parameters, the cutting fluid can make a considerable contribution to increased economic viability, assuming that all other influencing factors have been optimized. Its principal task is to cool and lubricate the cutting edges. Chemically active extreme-pressure (EP) additives incorporated into the cutting fluid and polar effective substances help to reduce wear and friction.

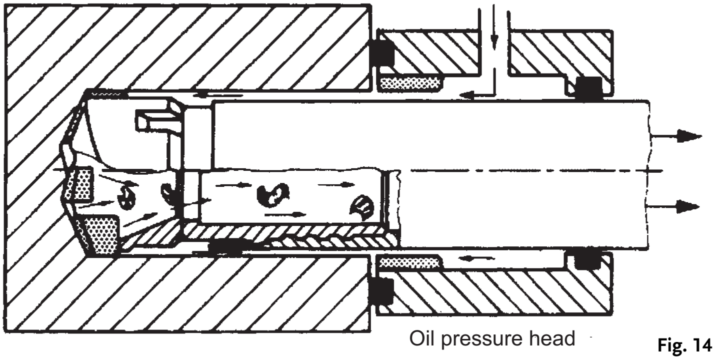


**Fig. 14.24** BTA system.

In addition to the actual cooling and lubricating functions, the cutting fluid, which is fed to the tool at high pressure, must also carry away the swarf from the cutting point. The high-pressure supply system requires not only a carefully designed fluid circuit (with a recirculation rate no higher than 6 $h^{-1}$),but also for products with low foam and in particular low oil-mist characteristics.

Unusually critical conditions occur when deep hole drilling with gun drill tools, which, have an off-center cutter tip. This offset cutting action means that resultant cutting forces must be absorbed at the bore wall, usually by way of support pads. This in turn increases the proportion of friction, as a result of friction between the support pads and the bore wall (Fig. 14.25).

Accordingly, lubrication of these support pads is one of the important tasks which have to be performed by the deep hole drilling oil.

### 14.5.6
### Threading and Tapping

Frequently water miscible products are used in the production of external threads. The focus is clearly on neat cutting oils for tapping where the materials to be cut are difficult or most difficult (material groups 2 and 3, section 1); typical tapping and threading oils of this type are sulfur active oils with viscosities between 15 and 40 $mm^2\ s^{-1}$ at 40 °C; the proportion of polar substances is also important. Tight drill holes and the low pitch of the internal threads call for less viscous oils because of the favorable flushing properties. Here again the risk of cutting edge build-up is great because of the generally low cutting speed, and damage to the cutting edge can result.

Lubrication is also important during reverse movement of the tap. If chips become stuck between the flank and the workpiece in the reverse movement phase this can lead to edge build-up on the flank. Breakaway is more likely when this edge build-up occurs close to the cutting edge. Frequently recommended where tapping is carried out and on difficult and most difficult-to-cut materials are thread cutting pastes (sometimes dilutable with water up to 1:5) or oils containing solvent. In particular, products with chlorinated hydrocarbons, more especially 1,1,1-trichloroethane as solvent, are easy to apply by brush or in drops. Many problems can be solved with such oils when tapping blind

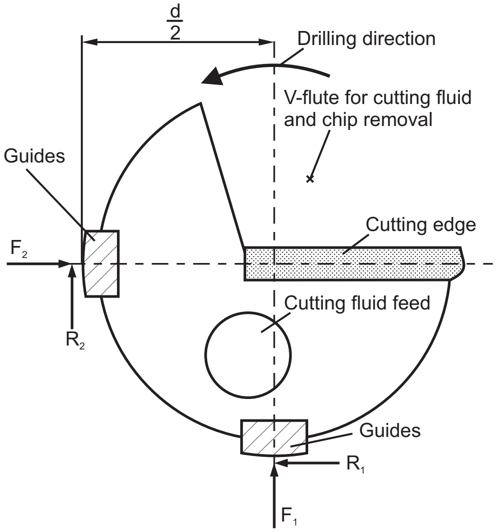


**Fig. 14.25** Sectional view of a gundriller with schematic presentation of friction forces at the guide pads. $F_1$, $F_2$, normal forces transmitted from the guide pads to the bore wall; $R_1$, $R_2$, friction forces at the guide pads; $S$, cutting edge. Drilling force = cutting force + friction force. Friction force = $\mu(F_1 + F_1) \times d/2$

hole threads. Such oils and paste containing solvent are not suitable for coolant circulation systems. Today for environmental reasons such products have been replaced by low viscosity hydrocarbon solvents and/or other synthetic fluids.

### 14.5.7
### Broaching

Broaching is a type of machining operation which is carried out with a clear focus on neat cutting oils. Far more than is the case of many other cutting operations the main consideration is tool wear or tool life. The reason for this is the fact that the broaching tool is a very complicated part and, the tool is manufactured from one single part. The material is predominantly high speed steel; carbide metal as a tool material is only used for gray iron machining. In the case of broaching several teeth engage at the same time and very frequently the chip width is large. Chip removal can be very problematic which then generally also calls for relatively low viscosity oils. In view of the cutting fluid supply internal broaching is more problematic than external broaching and horizontal broaching is more difficult than vertical broaching.

Temperature sensitivity of the broaching tool combined with cutting from the solid workpiece which follows each tooth without any soft initial cut, call for relatively low cutting speeds. As a result this leads us into the build-up cutting edge zone which demands special and even particularly high amounts of additives in broaching oils. A proportion of active sulfur is necessary to reduce the build-up on the cutting edge. Frequently active sulfur additives are used in conjunction with substances containing chlorine [14.74]. Increasing the cutting speed, in so far as machine technology permits, can avoid the critical cutting edge build-up zone; in this case lubrication conditions for broaching oils are generally more difficult. If one is successful in reducing cutting edge build-up, this generally improves the workpiece surface which is an interesting aspect especially in serial production; in mass production, where broaching is very often not a precision machining operation but can be classified as a roughing operation with high stock removal rates, the question of quality of the surface of the workpiece is of secondary importance and under these circumstances protection of the tool by the metalworking fluid has priority. At very high broaching speeds and high stock removal rates good results are achieved on free machining steels by using water miscible coolants , more especially EP emulsions and synthetic products free of mineral oil with relatively high concentrations (10 to 35 %); in this case, compared with neat oils, the improvements of the cooling effect and of chip removal are important factors. Also important when broaching, as far as the actual lubricating operation is concerned, unlike almost all other cutting operations with geometrically defined cutting edge, this operation employs an extremely low clearance angle. As a result of this the front clearance is under considerable stress and flank wear and the wear on the rake can be greater and this should also be noted for the coolant supply.

During linked broaching in simple cutting operations, an attempt should at least be made to use EP emulsions on transfer lines running water miscible coolants in low concentration.

## 14.6
## Machining with Geometric Non-defined Cutting Edges

### 14.6.1
### Grinding

The machining of components by grinding is classified by machining operation, workpiece contour and the workpiece movement as external cylindrical, internal cylindrical, face and lathe grinding according to DIN 8589 or ISO/DP 3002/V 98.

Face or surface grinding is the method used to produce surfaces which are either perfectly flat or run in a straight line in the feed-in direction of the grinding wheel.

The most frequently applied methods in practice are peripheral cross grinding (profile grinding) and peripheral longitudinal grinding (surface grinding). In both methods there is a fundamental difference made between deep and complete traverse grinding. Large feeds are used for deep grinding. The contact length between workpiece and grinding wheel increases with the feed rate, and this makes the supply of cutting fluid in the grinding contact zone more difficult. Consequently, the cutting fluid must be supplied by means of suitable nozzles, high cutting fluid pressures and flow rates [14.75].

External cylindrical grinding is used with symmetrical rotating workpiece contours. We differentiate between centerless grinding and grinding between centers, depending on how the workpiece is mounted. Industrial applications are, for examine [14.74]. Increasing the cutting speed, in so far as machine technology permits, can avoid the critical cutting edge build-up zone; in this case lubrication conditions for broaching oils are generally more difficult. If one is successful in reducing cutting edge build-up, this generally improves the workpiece surface which is an interesting aspect especially in serial production; in mass production, where broaching is very often not a precision machining operation but can be classified as a roughing operation with high stock removal rates, the question of quality of the surface of the workpiece is of secondary importance and under these circumstances protection of the tool by the metalworking fluid has priority. At very high broaching speeds and high stock removal rates good results are achieved on free machining steels by using water miscible coolants , more especially EP emulsions and synthetic products free of mineral oil with relatively high concentrations (10 to 35 %); in this case, compared with neat oils, the improvements of the cooling effect and of chip removal are important factors. Also important when broaching, as far as the actual lubricating operation is concerned, unlike almost all other cutting operations with geometrically defined cutting edge, this operation employs an extremely low clearance angle. As a result of this the front clearance is under considerable stress and flank wear and the wear on the rake can be greater and this should also be noted for the coolant supply.

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ple, shafts and roller bearing elements, cam shaft bearing seats, valve push rods, rotor shafts and injector needles. In the case of external cylindrical grinding, water mixed, generally transparent synthetic cutting fluids, which are used primarily because of the better cooling and flushing properties.

Internal cylindrical grinding is comparable to external cylindrical grinding. However, since the contact zone between workpiece and grinding wheels is considerably larger, the cutting fluid and its supply is of greater importance. As is the case with deep grinding, very coarse porous grinding wheels with lower hardness and open structure are to be used so that the temperature in the contact zone is kept low.

Gear grinding is a special grinding method. In principle, this method can be subdivided into discontinuous and continuous hob and profile grinding. The method selected also depends on both the specific size of the component and the quantity involved, as well as the quality of the toothing required. For example, as far as continuous profile grinding is concerned only very short machining times can be achieved, with average quality machining results, as against partial hob grinding which ensures maximum quality toothing with longer grinding times.

Also important as special grinding methods are thread grinding, form and cut-off grinding and sharp grinding. These grinding methods frequently make special cutting fluids necessary.

### 14.6.1.1 **High-speed Grinding**

High speed grinding at *Vc* > 80 m s$^{-1}$ has been marked by the endeavor to make the grinding process more economical through the higher time-related stock removal rates (Fig. 14.26) [14.76, 14.77]. The principle of high speed grinding is to machine at higher feed control rates and at the same time, at a high cutting speed and at an over proportional rate of feed. As a result, the demands put on grinding

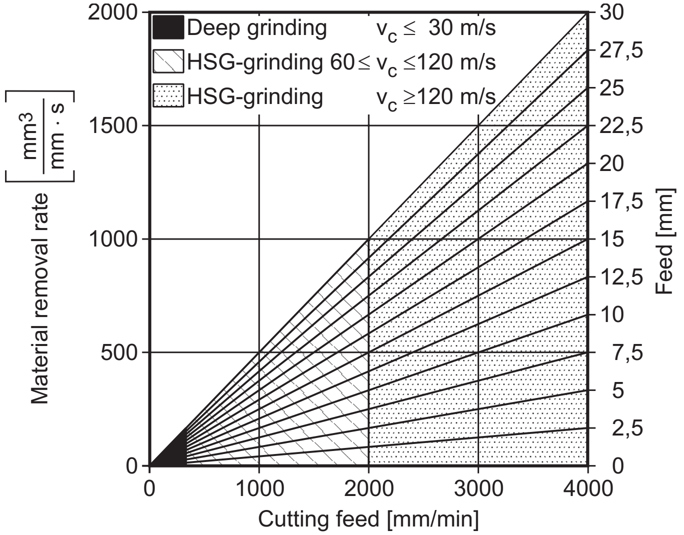


**Fig. 14.26** High-speed grinding technology, capacity ranges.

machine stiffness, grinding wheel driving capacity and workpiece drive are considerably higher than those put on conventional grinding. The use of grinding oils is special to high speed grinding with CBN grinding wheels (cubic boron nitride). The high lubricating effect reduces the friction between workpiece and the grain points and, as a result can prevent the generation of heat. In this way it is possible to prevent thermal problems such as grinding crack, soft skin formation and re-hardening. This is also reflected in the reduced tangential force when suitable oils are used. At the same time better quality surfaces are produced with neat oils.

### 14.6.1.2 **Grinding Wheel Abrasive Materials and Bondings**

Today, abrasive materials are mainly produced synthetically and have replaced natural materials. The most important materials are classified according to increasing hardness: Corundum ($Al_2O_3$), silicon carbide (SiC), cubic boron nitride (CBN) and synthetic diamond. The choice of abrasive materials depends both on the material as well as on the grinding method. Although corundum is used especially for conventional grinding of cast and steel materials, CBN is suitable for high performance grinding of ferrous materials because it is considerably harder and dissipates heat better [14.78] . With regards to their reaction to chemicals CBN and diamond are to be viewed more critically than corundum and silicon carbide. There is a tendency for diamonds to graphitize at 900 °C. CBN is subject to hydrolytic decomposition above 1000 °C. Hydrolysis depends greatly on the machining conditions and the coolant used. This hydrolysis is one of the reasons for using oils.

In all abrasives to DIN 69111 the abrasive grains are bonded with each other to the base material. The purpose of the bond is to secure the abrasive grains until they become dull and then release them. We differentiate between resinoid ceramic and metallic bonds because of the difference in their elasticity, temperature resistance, heat dissipation and dressing capacity. Their resistance to chemicals is particularly important as far as the coolants used are concerned.

Resin bonded grinding wheels are not stable to hydrolysis and can lose their tensile strength when water-mixed coolants are used [14.79].

Ceramic materials are highly resistant to chemicals and temperature but are brittle and glass-like in character. Metallic bonds are becoming more and more important as far as superabrasives are concerned because of their high resistance to wear.

### 14.6.1.3 **Requirements for Grinding Fluids**

The grinding process, as a precision machining process, is used frequently for the final stage machining of workpieces. As a result, high demands are set on component accuracy. Particularly significant in this respect is the coolant.

Its first task is to reduce the friction between the abrasive material and surface of the workpiece in the contact zone and, as a result, lower the temperature in the grinding area. It is necessary to reduce the contact zone temperature to increase the tool life of the grinding wheel and prevent the peripheral zones of the workpiece from being affected in any way.

A further task of the coolant is, of course, to prevent thermal damage to either the workpiece or grinding wheel by dissipating the heat generated. Flushing the grinding wheel clean is an important criterion as far as tool life is concerned. Here the viscosity and quality of the base oil as well as the coolant additives play a decisive role.

We must differentiate between the following criteria to be able to select the optimum lubricant for the respective grinding process.

- workpiece material
- grinding wheel
- grinding method
- cutting parameters (cutting speed, feed, stock removal rate over time, etc.)

Whether a water-miscible coolant or a straight oil is suitable for a process also depends, apart from the above-mentioned criteria, on the peripheral system. Here the machine tool equipment plays a particularly decisive role with regards to extraction, sealing, wiring and type of filter system.

#### 14.6.1.4 **Special Workpiece Material Considerations**

Cemented carbide grinding sets particularly high demands for cooling of the used carbide grinding wheels during use. As a result, synthetic water-miscible coolants are primarily used for this purpose. In this case the coolants should certainly contain inhibitors which prevent cobalt complexes. Water soluble cobalt compounds lead to waste-water disposal problems and are to be considered particularly critical for occupational health reasons. The formation of water soluble cobalt complexes [14.80] can be reduced to a minimum by using suitable inhibitors whereby full stream fine filtration with pre-coated filters and return flushing filters are an advantage. Latest trends in Europe also reflect the use of very low viscosity synthetic hydrocarbon fluids for cemented carbide grinding. Straight cuts with low boiling points and with flash points just above 100 °C are used to ensure adequate heat dissipation.

#### 14.6.1.5 **CBN High-speed Grinding**

In recent years the trend towards high speed grinding has promoted CBN grinding wheels to an ever increasing extent. Cutting speeds from 80 to 250 m $s^{-1}$ are being achieved today in CBN high speed grinding. Galvanic bonded CBN grinding wheels, which can be dressed, have up to 100 times longer tool lives than conventional corundum grinding wheels when a suitable neat grinding oil is used. In the case of CBN grinding wheels, one special feature is that the coolant reduces the friction in the engagement zone of the grinding wheel, which leads to the temperature being reduced considerably. As a result, despite a poorer cooling effect, neat grinding oils ensure considerably lower temperatures in the contact zone (Fig. 14.27).

Consequently, the use of suitable oils for CBN grinding brings the following advantages for the user: longer grinding wheel tool life (factor 10 higher than in the case of corundum wheel grinding with water-miscible coolants) and considerably less thermal damage to the workpiece.

The use of straight oils in CBN grinding wheel also enables the user to filter to a very fine degree using pre-coated filters, which has a favorable effect on both the surface finish of the component and the tool life of the grinding.

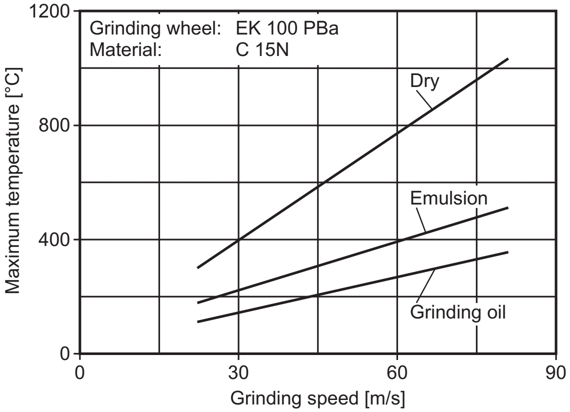


**Fig. 14.27** Workpiece temperature as a function of grinding speed and cutting fluid type.

CBN grinding operations are also carried out to some extent with water mixed coolants for system capacity or process compatibility reasons. Highly additived products are used in this case which, according to their lubricating properties, are almost equivalent to neat cutting oils. Component quality is achieved in this case at a slightly reduced stock removal rate. However, the user has to accept as a disadvantage the clearly reduced grinding wheel tool life because of the higher grain breakaway.

Ester oils have proved particularly favorable for flute and twist drill grinding, as well as for tooth cutting.

Unlike mineral oils, ester oils offer numerous technical cutting advantages as a result of their chemical properties. Their excellent air release behavior proves to be a considerable plus point [14.81], especially when machining with non-defined cutting edges. In the meantime, pressures of over 100 bar are more and more becoming state-of-the art technology for high speed grinding operations. On the other hand, conventional mineral oils take up a great amount of air and tend to foam. The air trapped in the oil while machining leads to considerably poorer heat dissipation. Some ester oils with a specific chemical structure immediately release the air and do not foam. The more favorable heat dissipation frequently permits cutting speeds to be increased [14.145, 14.146]. Apart from their better air release capacity when compared with mineral oils, ester oils also have a more favorable friction coefficient [14.82] . As a result, lower viscosity ester oils can very often be used for the same machining process. Drag-out losses through chippings and machined parts are, as a result, considerably less.

### 14.6.1.6 **Honing**

As with grinding, the honing tool has grit or grain bonded multiple cutting edges. The most important grain materials are corundum, silicon carbide, CBN (cubic boron nitride) and diamonds.

Honing is applied to improve the form and accuracy of a workpiece by continuous surface contact with the tool.

There are four main categories of honing to be considered:

- long-stroke honing
- super finishing
- gear honing (power honing)
- laser honing

In long-stroke honing the complete length is frequently covered by one movement (long strokes with turning honing stone on stationary tool). In the case of super finishing, both the tools and workpiece move; the length of the reversing path is generally < 10 mm, and the honing stone oscillates at a higher frequency. Gear honing is a hard machining process with which a inner tooth honing stone made of high-grade corundum or silicon carbide is used for honing external straight or helical pinion wheels and shafts. Gearwheels with inner toothing are produced in the same way using external toothed tools. Laser honing is a combination of honing and laser processing. Specific amounts of oil are used within a precisely defined area to create precisely defined surfaces [14.147, 14.148].

The advantage of all honing methods is that, with high measurement accuracy, surface roughness values in the region of Rz = 1 μm can be achieved, which lie within the surface class 3 in accordance to DIN 3969, Part 1 [14.83].

Since honing tools have area contact, the hydrodynamic proportion of the stock removal can be controlled by the hold-down pressure, machining speed and honing fluid viscosity. The hydrodynamic effects increase as the honing operation proceeds and this reduces the surface roughness; there is no stock removal where fully hydrodynamic tool floating is concerned. This is made clear in Fig. 14.28 in which the

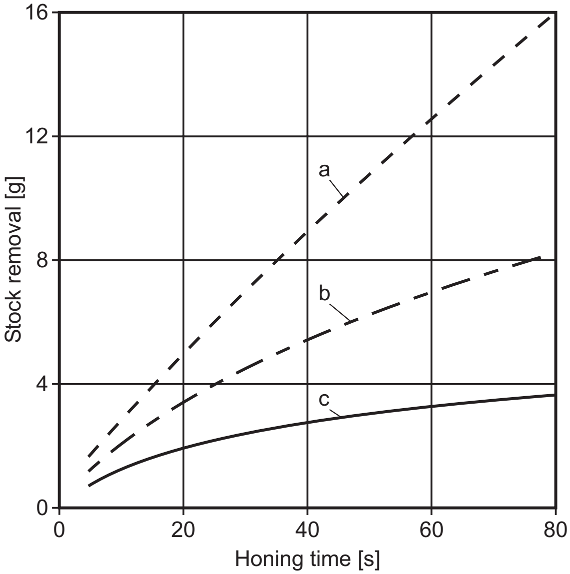


**Fig. 14.28** Stock removal at various contact pressures over honing time. (a) $p = 95\ \mathrm{N\ cm^{-2}}$; (b) $p = 58\ \mathrm{N\ cm^{-2}}$; (c) $p = 34\ \mathrm{N\ cm^{-2}}$.

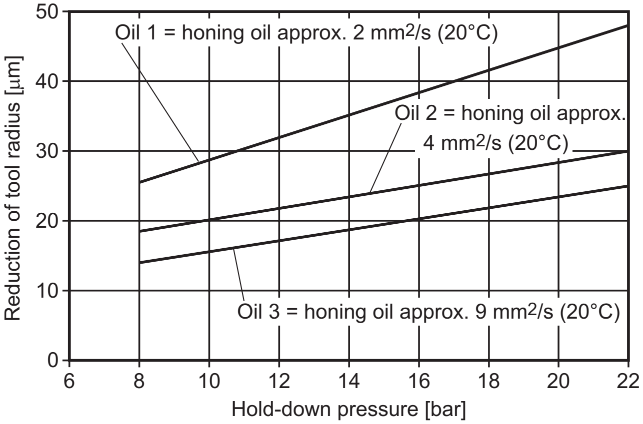


**Fig. 14.29** Effect of honing oil viscosity on the workpiece stock removal of cast iron in relation to hold down pressure (according to Haasis).

stock removal at various hold down pressures is plotted against honing time. In the case of the lowest contact pressure no further stock removal takes place after a honing time of 80 s because the hydrodynamic lubricating film leads to complete separation of the honing stone and workpiece. When the hold-down pressure is not only transmitted via the honing stone but also over the wear guides, this must be maintained as far as possible without wear. This is possible by selecting a honing oil with suitable viscosity and additives, but is frequently contrary to the demands for stock removal, where a fluid with a lower viscosity is required to ensure sufficient stock removal. The same applies for rinsing the honing stones, where low viscosity oils are also an advantage.

Figure 14.29 shows the influence of honing oil viscosity on the workpiece stock removal of cast iron in relation to contact pressure (according to Haasis).

### 14.6.1.7 **Honing Oils**

During honing, due to this type of operation, there is a very large contact zone between tool and workpiece with relatively low pressures. This means that the heat formation is relatively low compared with grinding so that the coolant does not to have to dissipate any great amount of heat.

An important criterion when honing is flushing the honing stone to remove the stock removed material to ensure that the working surface retains its grip and is self sharpening.

As a consequence, very low viscosity oils between 2 and 10 $\text{mm}^2\ \text{s}^{-1}$ at 40 °C are used when honing. These are applied, as a general rule, at low pressure but at a high flow rate through ring nozzles. To achieve the component accuracy required, the honing oil is kept at a constant temperature by chillers. If an attempt is made to associate certain honing oil qualities with the workpiece materials to be machined, short chipping hard materials are associated more with very low viscosity oils, and long-chip tough materials are better machined using higher viscosities [14.84].

There is a special situation, for example, with sulfur bonded honing stones. If the sulfur dissolves in the honing oil this can lead to reduced stock removal. Special inhibitors in the honing oil can prevent the sulfur from dissolving. As is the case when grinding it is essential when selecting a coolant to also consider both the honing-stone bonding and the material to be machined.

Apart from straight oils, water mixed coolants are also used, especially when honing cylinder liners with diamond honing stones in the automobile industry. Reasons for this are to be found frequently in the process line, because previous machining is generally carried out with water-mixed fluids. This leads to reduced process costs when uniform media are used.

#### 14.6.1.8 **Lapping**

Unlike grinding and honing, lapping is a precision finishing process with non-bonded abrasive. In lapping, the workpiece surfaces are machined by the friction between the workpiece surfaces and the appropriate counter surface in the form of a working disc. The stock removal is by means of a lap (grit and liquid) which is applied between the workpiece and lapping wheel surface. The stock removal in this case is effected by the grit movement in the contact zone [14.85]. The grit points press into the material to indent depths of between 5 and 10 % of the grain diameter. In accordance with DIN 8589, lapping is subdivided into surface, round and helical lapping, tooth lapping, form lapping and copy lapping. Further classifications are possible based on the effective surface of the lapping tool. When the tool axis and the workpiece surface are parallel to each other the process is called peripheral lapping; side lapping when the tool axis and the workpiece surface are vertical to each other. A distinction can also be drawn between broad and fine lapping, depending on the fineness of the surface finish.

#### 14.6.1.9 **Lapping Powder and Carrier Media**

Mainly silicon carbide (SiC), corundum ($Al_2O_3$), boron carbide ($B_4C$) and diamond are used as abrasive grain. Preference is given to corundum for soft steels, brass and bronze and castings, while SiC is preferred for machining annealed steels and glass. Boron carbide and diamond are used for very hard materials such as cemented carbides and ceramic materials.

The main characteristics for the quality of the lapping powder are the distribution of the grain by size, its hardness and the type and number of grit cutting edges. The grain size spectrum ranges from 5–40 μm, but the bulk lie between 12 and 18 μm.

The purpose of lapping oil is to induce homogeneous mixing of the grain. However, lapping powder agglomeration must certainly be avoided. The lapping oil has an important task to perform in the operation, in that it lubricates properly to prevent cold welding between workpiece and working disc. The lubricating film must not be applied too thickly as otherwise hydrodynamics will prevent stock removal.

In the case of lapping oils we differentiate between the very low viscous oils, which can only maintain the lapping powder in suspension for a short period of time, and the thixotropic, high viscous products, which suspend the lapping powder for days on end. The advantage of low viscous oils is the very good washing behavior, which is reflected in clean surfaces. Due to the lack of carrier capacity for the lapping powders these products are only used on machines where the lapping medium tank is equipped with an agitator.

## 14.7
## Specific Material Requirements for Machining Operations

### 14.7.1
### Ferrous Metals

Ferrous metals [14.86] include all the alloys whose major alloying element is iron, where cast iron, carbon steel, alloy steel, stainless steel, and tool steel is included.

#### 14.7.1.1 Steel

In general steel is an alloy of iron and carbon. The basic differentiation between cast iron and steel is the carbon content, ranging up to 1.5 % in steel and up to 4 % in cast iron.

The alloying elements used in steel have specific functions and their concentration ranges have been developed for the various grades to produce specific properties.

Carbon determines the ultimate hardness a steel can achieve. Steels with low ranges up to 0.20 % carbon achieve maximum hardness up to about 35 HRC (Rockwell C hardness). When used for applications requiring high hardness, these steels can be carburized, a case-hardening heat treatment. Steels with medium carbon, up to about 0.50 %, may be fully hardened to as high as 60+ HRC.

Manganese, vanadium, chromium, and molybdenum are very effective at increasing hardenability, but unlike manganese, they form also strong carbides. These elements offer increased wear resistance, but this effect leads to increasing process severity when machined.

Nickel generally improves hardenability, promotes toughness and is frequently used for applications requiring high impact resistance.

Sulfur and phosphorus are added to promote machinability while forming nonmetallic inclusions with low melting points. During machining operations these inclusions lubricate the cutting tool and also act as chip breakers. Both sulfur and phosphorus have a negative effect on strength and toughness. Lead is another alloy that falls into the category of a free machining additive but due to its toxicity is being used less and less [14.87]. The alloy steels can contain a maximum of about 5 % alloying elements. Steels for more demanding applications such as the tool steels require much higher alloy additions.

#### 14.7.1.2 Tool Steels

These steels are difficult to categorize. They are used are for molds, bearings, wear parts, and a wide variety of structural components.

#### 14.7.1.3 **High-speed Steels (HSS)**

All metal cutting was once performed with high-carbon steels. This grade is capable of developing high hardness but tends to soften rapidly when it heats up. The addition of tungsten and chromium to cutting steel made it much more resistant to softening when heated and, therefore, made it possible to increase the cutting speed to a remarkable degree. These steels came to be known as high-speed steel.

Compared with this group the other classes of tool steels are less important for the cutting application.

#### 14.7.1.4 **Stainless Steels**

Stainless steels can be categorized by their crystallographic structures: ferritic, martensitic, and austenitic. The most corrosion-resistant grades are to be found amongst the austenitic series followed by the ferritic. The martensitic grades, generally have the poorest corrosion resistance.

The primary mechanism by which stainless steels gain their corrosion resistance is through the development of a stable, protective surface oxide film. It is generally accepted that a minimum chromium content of 12 % is necessary to form the protective oxide film. Stainless steels form this film naturally by reaction with oxygen in the atmosphere.

Stainless steel is subject to a serious reduction in corrosion resistance through a mechanism called sensitization. Chromium has a very strong affinity for carbon and tends to form a very stable carbide. One common fabrication process that can induce sensitization is welding. Sensitization can be reversed by a specific heat-treating process. [14.88, 14.89].

#### 14.7.1.5 **Cast Iron**

Cast iron has good mechanical properties and is easily machinable due to its unique microstructure. In its broadest description, cast irons are alloys of iron, carbon, and silicon. The carbon is usually present in the range of 2.0 to 4.0 % and the silicon in the range of 1.0 to 3.0 %. The form taken by the excess carbon is the basis of the three major subdivisions of cast irons: gray iron, white iron, and malleable iron.

The most common variety of cast iron has the excess carbon present in the form of graphite flakes and is called gray iron.

In irons with the carbon and silicon content minimized and where a very rapid solidification rate was attained, the excess carbon is present as a carbide and there is no free graphite. This type is called white iron, is very hard and frequently used in applications where extreme wear resistance is required.

White iron can be converted to so-called malleable iron by a heat treatment process.

Another method of producing a ductile form of cast iron is by a nodulizing inoculation. If magnesium or rare earth metals are added to the molten iron, the excess carbon forms spheroidal nodules of graphite rather than the flake form found in gray iron. The nodular graphite structure results in a substantial increase in strength and ductility. Ductile iron castings can compete with steel castings or forgings in some near net shape applications.

## 14.7.2
## Aluminum

### 14.7.2.1 Influence of the Type of Aluminum Alloy

In addition to the machining conditions, the type of workpiece material plays an important role.

#### Malleable Aluminum alloys

As these alloys contain few coarse abrasives, the fastest cutting speed possible should be chosen (minimum 100 m $\text{min}^{-1}$) to avoid the formation of cutting edge build-up. The most important machining parameters for malleable Aluminum alloys are chip type and surface finish. The tendency of the material to 'smear' can be reduced by using sharp tools, high cutting speeds, good cooling and large rake angles.

#### Free-cutting Materials

Free-cutting materials are characterized by their heavy metal additives which assist chip breaking. Similar criteria apply to the selection of tool materials and cutting speeds as do for malleable aluminum alloys.

#### Cast Aluminum

As long as the cast Aluminum contains no silicon and is structurally uniform, it can be treated similarly to free-cutting Aluminum and malleable aluminum alloys. Greater difficulties can be expected however when machining cast aluminum–silicon materials.

#### Aluminum–Silicon Alloys

Aluminum–silicon alloys tend to lead to cutting edge build-up. When not forged, cutting edge build-up occurs when the material contains more than 5 % silicon. The cutting edge build-up problem reaches its peak when the silicon content is between 8 % and 12 % (silicon alloys), i.e. when the structure is almost eutectic. Above 13 % cutting edge build-up steadily decreases and at about 22 % disappears altogether. If these figures are compared to the usage of Aluminum alloys in Germany, one can clearly see that 80 % of Aluminum alloys contain a percentage of silicon which makes machining difficult and which causes cutting edge build-up (Table 14.16). It should also be remembered that although a silicon content of over 12.7 % reduces the cutting edge build-up problem and assists chip breaking, it also causes much more wear. This means that the water-miscible cutting fluid must not only provide good cooling but also offer adequate lubrication in order that the demands made on Aluminum machining can be met.

**Tab. 14.16** Aluminum–silicon alloys.

| | | |
|---|---|---|
| 31 % | AlSi 8 Cu 3 | Thermally stable, multipurpose alloy |
| 16 % | AlSi 12 Cu | As AlSi 12 but harder |
| 14 % | AlSi 11 | Automobile wheels |
| 8 % | AlSi 12CuMgNi | Alloys for thin-walled pistons |
| 5 % | AlSi 12 | For thin-walled, cast components |
| 3 % | AlSi 10 mg | As AlSi 12 but hardenable and stronger |
| < 3 % | Other alloys | |

*Hypoeutectic (< 12.7% Silicon)*

Surface finish and chip formation are not problems with hypoeutectic aluminums. The abrasiveness which increases in proportion to the increase in silicon content, requires the use of a cutting fluid.

*Eutectic (approx. 12.7% Silicon)*

The characteristic feature of these aluminum–silicon alloys is their softness compared to other aluminum–silicon alloys. The abrasive structural components are pressed into the material during machining and therefore cannot exercise their abrasive effect. The tool wear they cause is similar to that of cast materials. With regard to surface finish, these alloys tend to 'smear' just like malleable Aluminum alloys.

*Hypereutectic (> 12.7% Silicon)*

The very high tool wear caused by such materials makes the use of a high lubricity cutting fluid a necessity. Although cutting edge build-up is rare, 'false chip' formation is very common.

#### 14.7.2.2 **The Behavior of Aluminum During Machining**

The specific cutting energy required for Aluminum is approximately 25 % less than that of steel. However because of the much higher cutting speeds used, greater machine performance is necessary. The good thermal conductivity of Aluminum serves to transfer heat away from the cutting face as well as reducing heat build-up at the tool's cutting edge. The modulus of elasticity (70 000 N $\text{mm}^{-2}$) of aluminum which is about one third less than that of steel as well as its relatively low tensile strength means that far less energy is needed by the tool to penetrate the material.

#### Cutting Speeds and their Effect on the Surface Finish

The two special problems associated with the machining of Aluminum are cutting edge build-up and the formation of 'false chips' The effect of cutting speed on surface roughness can be seen in Fig. 14.30.

The curve pattern (the extremities and the optimum zone) depends on the material being machined.

### Cutting Edge Build-up

The lower cutting speed limit for Aluminum machining is determined by the point at which cutting edge build-up occurs. This usually means that cutting speeds of less than 90 m $min^{-1}$ should be avoided. If lower cutting speeds are unavoidable, for example when drilling, broaching, thread cutting or boring, countermeasures must be taken. The formation of cutting edge build-up is determined by the following parameters:

- cutting speed
- cutting angle
- lubrication at the cutting zone
- feed
- type of Aluminum.

Measures to eliminate cutting edge build-up can include:

- increasing the cutting speed, insofar as this is technically possible.
- increasing the cutting angle. Naturally this is only possible to a certain extent as thin tools used on brittle materials can break.
- polishing the cutting surfaces.
- the use of suitable lubricants which assist chip transportation and avoid cutting edge build-up by reducing friction.

### False Chip' Formation

An additional form of wear called 'false chip' formation only usually effects cast aluminum–silicon materials which display high wear factors. Higher cutting speeds cause higher temperatures to be generated at the cutting zone. If the temperature rises to such an extent that the workpiece material becomes pasty, the material is squeezed out of the contact zone. This material then solidifies and forms 'false chip'. This usually builds up on the clearance faces of the tool. As the 'false chip' and the workpiece come into contact with each other, the surface of the workpiece is damaged. Apart from high cutting speeds and high fluctuating temperatures, the main cause of false chip formation is tool bluntness. To prevent the formation of false chips, the cutting speed must be reduced. All measures which lead to a reduc-

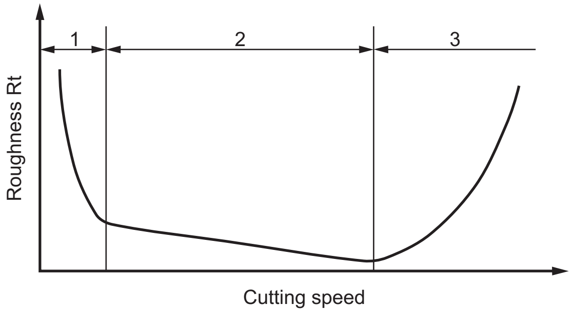


**Fig. 14.30** Effect of cutting speed on surface roughness. 1, Cutting edge build-up; 2, No cutting edge build-up; 3, 'False chip' formation zone.

tion in temperature at the cutting zone either control or completely eliminate the formation of false chips.

The best way of controlling temperature build-up is to use a cutting fluid. Other ways include changing the tool's geometry, changing the cutting parameters, polishing the cutting surfaces and using polycrystalline tools.

#### Wear

Lower cutting forces and temperatures when machining aluminum generally lead to less machine down-time. Important wear factors are for example, imperfections in the workpiece materials such as cavities, metallic and non-metallic impurities and porosity. Wear to the cutting edge is usually accompanied by a rounding of the tool's tip and the permissible wear width is an indicator of tool service life. Erosion which is often encountered when machining steel does not occur. The blunted cutting edge results in higher temperatures at the cutting zone which, if allowed to rise too much, can lead to the formation of 'false chips'. Aluminum is mostly machined with carbide tools. When selecting the most suitable type of carbide it should be remembered that the P-type containing titanium carbide has a greater affinity to Aluminum than the tungsten carbide K-types and therefore more sticking problems will occur. K-types should therefore be chosen to avoid this diffusion wear.

### 14.7.2.3 Tool Materials

#### HSS

This relatively cheap and tough but not very thermally stable material is suitable for slightly abrasive, temperable and non-temperable, soft malleable alloys and pure Aluminum. Satisfactory results can also be obtained on free-cutting and cold, work-hardened malleable materials.

#### Carbides

Carbides are almost exclusively (over 90 %) used for the machining of Aluminum, whereby of the K-types, K10 is the most popular. The harder type K01, which displays greater resistance to wear but which is less tough is preferred for machining processes which do not produce shock loads such as drilling and the turning of high silicon cast alloys. The tougher K20 type is ideal for shock-loaded operations such as milling. Coated carbides (titanium carbide, titanium nitride) are unsuitable because of their chemical similarity to Aluminum.

#### Diamond

In recent years diamond has gained popularity over carbides for the machining of hyper-eutectic aluminum–silicon alloys and to some extent for almost eutectic alloys, because of its greater wear resistance. Polycrystalline diamonds are better than monocrystalline and compact diamonds because of their superior characteristics.

**CBN**

CBN has been unable to displace diamond for the machining of aluminum–silicon alloys because of its lower resistance to wear.

### 14.7.3
### Magnesium and its Alloys

The use of magnesium alloys in the automotive and engineering industries has been on the increase since the mid-nineties. Compared with die-cast aluminum alloys, cast magnesium alloys have the advantage of lower weight. As far as machinability of the materials is concerned, factors such as corrosion and flammability have to be considered in the case of magnesium and these ultimately decide the type of cutting fluid used.

When freshly machined, magnesium oxidizes rapidly. If water is present, the oxidation process generates hydrogen gas which can explode. And even at low temperatures, there is a danger that chips will self-ignite. In Germany, machining magnesium with water-based cutting fluids was prohibited by the TRGA 509 [14.90] until 1995. The reasons for this were not just the formation of hydrogen gas and the obvious danger of explosions but also the hazards connected with the storage and transportation of chips. These days, machining magnesium with water-based fluids is permitted throughout the world if the necessary precautions are taken: These include the avoidance of chip build-up in the machine by the use of high fluid feed pressures and corresponding chip chutes and vapor extraction to remove any hydrogen from inside the machine. If water-miscible cutting fluids are used, their stability is of great importance because large amounts of magnesium are dissolved in the fluid during machining and this causes the hardness of the water to increase sharply. Normal aluminum and steel cutting fluids are unsuitable for machining magnesium because they split in very short periods of time as a result of the salt increase. Advanced magnesium cutting fluids remain stable even when the water's hardness exceeds 8500 ppm $CaCO_3$. Furthermore, special formulations can also help reduce the amount of hydrogen formed.

Even though magnesium can be machined without a cutting fluid, the danger of magnesium dust explosions is such that magnesium is almost never machined dry [14.91].

In recent years, neat cutting oils have been used increasingly because the risk of oil fires or detonations is significantly lower than that of hydrogen explosions. Fundamental research has shown that the use of ester oils and low-evaporation, hydrocracked oils can greatly reduce the risk of explosions. Tests on explosive behavior at the Institute of Combustion Technology at the University of Karlsruhe, Germany [14.92] have shown that the risks of magnesium dust explosions are eliminated if cutting oils are used.

**Project SAMMI (Period: from 1st September 1998 to 31st May 2001) [14.149]**

The objective of the project was to introduce safe and highly efficient processing of magnesium castings, using an adapted machining process with adapted tools, cooling lubricants, and a high-speed cutting (HSC) machining center with integrated safety devices. A wide range of uncoated and diamond-coated inserts for milling tools, drills, and reamers have therefore been developed. Coating conditions, especially, and also pretreatment of the substrates and geometry have been varied. The tools have been evaluated in laboratory tests in which mainly machining forces, workpiece quality, and tool wear have been analyzed. The tests have been performed both dry and with cooling lubricants (pure oil and emulsion). Optimized tools were used for final machining tests under industrial conditions with a high-speed machine center as conventional milling centre. HSC machining of magnesium, using uncoated or coated tools, has been established.

The best formulation for the water-miscible lubricant had been successfully tested by the end-users in this project and the product works very well in terms of cutting conditions; there are, however, still problems with formation of salt, which obstructs filters, affects accuracy when present on tool holders, and makes ecological recycling of the coolant and chips difficult. As the main result of this project we can conclude that magnesium machining using neat oils is easier to perform than when using water-miscible coolants.

### 14.7.4
### Cobalt

When alloys containing cobalt, such as carbides, are machined, the metalworking fluid used plays a critical role both in terms of lubrication and toxicity. Carbides contain the highest concentrations of cobalt and these are manufactured by sintering or pressing. However, depending on the ultimate application, combinations of tungsten carbide, titanium carbide but also tantalum, molybdenum, niobium, zirconium, and vanadium carbides can be present in a cobalt, iron or nickel matrix. These alloys offer outstanding resistance to wear along with excellent thermal and corrosion resistance [14.150].

#### 14.7.4.1 The Health and Safety Aspects of Carbides

A MAK (Maximum Workplace Concentration) value of 0.5 mg $m^{-3}$ applied in Germany until 1979 when it was replaced by a TRK (Technical Guideline Concentration) value of 0.1 mg $m^{-3}$ total dust-borne cobalt.

The background to this threshold value are medical observations on the effect of cobalt on health which started in the 1940s. Experiments on rats exposed to cobalt dust showed that lung scarring takes place, a condition called bronchial fibrosis. In humans, the symptoms are coughing and exertion-related breathlessness. Apart from bronchial fibrosis, influences have also been recorded on the heart, skin, thyroid glands and blood corpuscles. Bronchial fibrosis was recognized as an occupational disease in the field of carbide manufacturing in 1961 and in the field of carbide machining since 1980. Health and safety studies show that the highest cobalt concentrations are found in ultra-fine dusts (1.4–3.8 μm) [14.93].

This is of particular significance to humans because particles smaller than 5 μm can enter the lungs. Cobalt measurements on people have shown that 92 % of all measurements are below the TRK figure. Cobalt concentration values of 0.005 and

0.77 mg Co $m^{-3}$ were recorded. No significant differences were recorded between dry and wet machining operations. Another study [14.93] however showed that the cobalt level in the urine of wet machining operators was twice that of those engaged in dry machining operations. This approximately correlates with the cobalt contents in cutting fluids [14.94, 14.95].

It is therefore absolutely necessary to use special water-miscible cutting fluids which prevents cobalt leaching when grinding carbides. The visual signal for dissolved cobalt is when the cutting fluid turns reddish-pink. Carbide-compatible cutting fluids have a pH between 8 and 9.0 and contain inhibitors which limit the formation of cobalt complex compounds. Apart from using special cutting fluids, it is recommended that the fluid is finely filtered to reduce the time in which the grinding sludge with its large surface area is in contact with the cutting fluid.

#### 14.7.4.2 Use of Cutting Oils in Carbide Machining Processes

In recent years, more and more neat oils have been used for the grinding of carbides [14.96]. In machining terms, oils offer poorer heat dissipation than water-miscible, synthetic fluids. As a result, low-viscosity product have to be used. To satisfy safety at work considerations (flashpoint and oil mist and oil evaporation), high-quality base oils such as PAO, white oils and HC oils with esters are used. Apart from the advantages of long life and good corrosion protection, pure oils do not dissolve cobalt and thus have a worthwhile advantage over water-miscible products in terms of toxicity.

### 14.7.5
### Titanium

Titanium and titanium alloys are among the most difficult materials to machine. For example, when machining the titanium alloy $TiAl_6V_4$, the cutting temperature of 550 °C is already reached at a cutting speed of 35 m $min^{-1}$, while with normally machinable steels this temperature is reached only at 150 m $min^{-1}$, or 1700 m $min^{-1}$ when machining aluminum. The use of cutting oils with a high additive content has therefore proven successful in numerous machining methods with regard to the tool wear. Here, the use of cutting oils with a high chlorine content will lead to a particularly good cutting performance.

The widespread use of titanium alloys with a high rupture stress in the aerospace industry has strongly limited the range of suitable cutting oils due to the possible stress corrosion cracking caused by halogens, especially chlorine and fluorine. This limitation is also accepted by several users who machine titanium alloys which, however, are not subject to these extremely high safety requirements. In this context, it is also worth mentioning that producers of titanium alloys point to the favorable properties of chlorinated cutting oils [14.97]. The excellent suitability of cutting oils and water-miscible products containing chlorinated paraffin as an EP additive for use in the machining of titanium alloys is also described [14.98].

Iodine-containing cutting oils have also shown favorable properties in the machining of titanium alloys [14.99]. However, due to formulation problems the use of iodine is not generally accepted.

The above-mentioned corrosion sensitivity of titanium is limited to intergranular stress corrosion cracking. With regard to other types of corrosion, titanium shows favorable behavior. It could, for example, also be proven that pit corrosion will only start with much higher chloride content levels than is known from CrNi steels and aluminum alloys [14.100].

With regard to chlorinated products, the above-mentioned limitations in cutting oil selection include a degree of chlorine-freedom in the ppm-range. Normally, these requirements are only met by specific products developed at great expense. When working with water-miscible cutting fluids, this requirement demands the use of completely desalinated water. However, the user should always carefully consider if the requirements on the parts machined by him justify this expense in cutting oils.

Further work has led to development of special chlorine-free cutting oils with phosphorous-containing EP additives, which give satisfactory results in the machining of titanium. For the most difficult machining operations, however, they are clearly inferior to chlorinated products.

Most recently, special chlorine and hydrocarbon-free ester oils have been developed which are used successfully.

### 14.7.6
### Nickel and nickel alloys

Besides pure nickel, Ni–Cu, Ni–Cr, and Ni–Cr–Co/W alloys are used for special applications in the medical, chemical, marine, and aeronautical industries because of their outstanding corrosion-resistance and high-temperature creep-resistance. Common alloy names are based on proprietary names usually from Inco, Haynes, and Krupp, e.g. the Inconel types.

Copper–nickel alloys, e.g. Monels, have a long history of use to combat corrosion in marine environments. Typical applications include large desalination plants and steam-turbine blades.

The Ni–Cr alloys Nimonics and Inconel are used to manufacture gas-turbine blades and disks, because of their high-temperature creep and oxidation resistance.

Nickel super alloys contain Ni, Cr, Fe, Mo, and W. They are known under the name Hastalloy. These alloys are resistant to reducing HCl and oxidizing acids at very high temperatures and are used in chemical plants and as jet engine components. Because of their very high tensile strength at temperatures <800 °C cutting is very difficult.

The machinability of nickel alloys mostly depends on correct selection of tool material – fine corned carbide tools – and coolant – one with a very high lubricity effect. Coolants containing large amounts of friction modifiers and lubricity additives therefore give the best results in machining operations [14.151, 14.162].

## 14.8
## Metalworking Fluid Circulation System

The technical results as well as the metalworking fluid costs depend to a great extent on the type of coolant and the design of the fluid circulation system. The metalworking fluid must be developed to meet the individual requirements of each circulation system. The right coolant can only be selected with full knowledge of all the essential details. Foaming properties, microbial stability, electrolyte stability, tramp oil compatibility, filterability, grinding swarf drag-out characteristics and further properties are determined largely by the circulation system.

### 14.8.1
### Metalworking Fluid Supply

The metalworking fluid can only cool and lubricate when it is delivered to the correct locations in the cutting process. Consequently, the metalworking fluid supply system has considerable significance on the process.

Even the complete removal of chips depends decisively on the correct metalworking fluid supply.

The dissipation of heat, which takes place in the primary sheering zone when cutting, has no importance for tool life. The heat which develops in the contact surface area between tool, workpiece and chip is of prime importance. Nevertheless, the metalworking fluid cannot provide direct cooling because this area is only accessible to a very limited extent. As a consequence the metalworking fluid supply for heat dissipation is particularly effective in close proximity to this heat source.

Flooding the tool while giving special consideration to the cutting edge is possible on the majority of machine tools without any particular technical problems. Frequently, the purpose of flooding the tool, chip and workpiece is not only to extend the life of the tool by cooling and lubricating but the cooling also has to reduce thermal expansion where workpiece tolerances are tight as well as to ensure accuracy. It may be necessary, depending upon the type of cutting process and amount of chips, to cool the chips which are produced. This prevents troublesome evaporation and decomposition of the residual oil and remains of the coolant itself. Figure 14.31 shows how the dissipated cutting heat is distributed and the considerable amount of heat developing in the chip.

Apart from the wide range of flood-applied processes, a few special methods have been developed for drilling and in this respect delivery of the metalworking fluid through an internal system has gained particular significance. In the case of deep hole drilling with internal metalworking fluid supply the coolant is delivered through the hollow drill under high pressure via the support to the cutting edge. In the case of deep hole drilling by the ejector method, the coolant flows in the annular area between the drilling tube and inner tube to the drilling crown to then get to the cutting edge from the outside; as a result of the ejector effect a proportion of the delivered coolant used for cooling and lubrication is returned by the drill to the inside. Where the BTA method is concerned a special coolant infeed unit transports

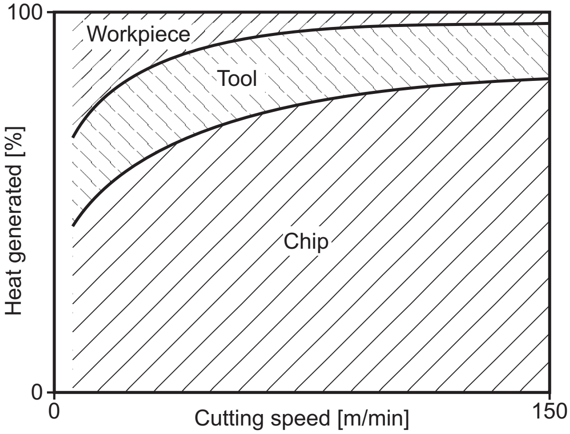


**Fig. 14.31** Example of the distribution of cutting heat on the workpiece, tool, and chip.

the coolant between the drill tube and bore wall to the cutting edge; return is through the inside to the drill tube. In this case improvements have been achieved in the resistance to wear compared with the usual external cooling even when drilling normally with twist drills in difficult-to-cut austenitic chromium nickel steels (Fig. 14.32 [14.101]).

In the case of the Hi-Jet method, very narrow jets of coolant are directed in the cutting tool zone at high velocity (> 70 m $s^{-1}$) against the cutting edge. It has not been possible to employ this process widely due to technical problems and the con-

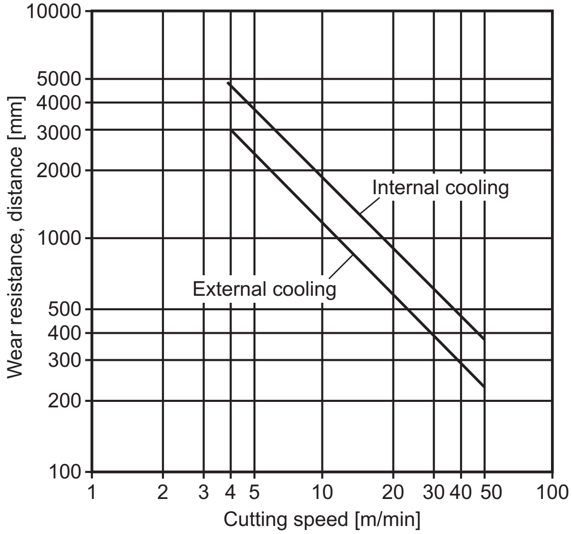


**Fig. 14.32** The influence of internal and external metalworking fluid supply on tool wear when drilling an austenitic chromium nickel steel with twist drills [14.101].

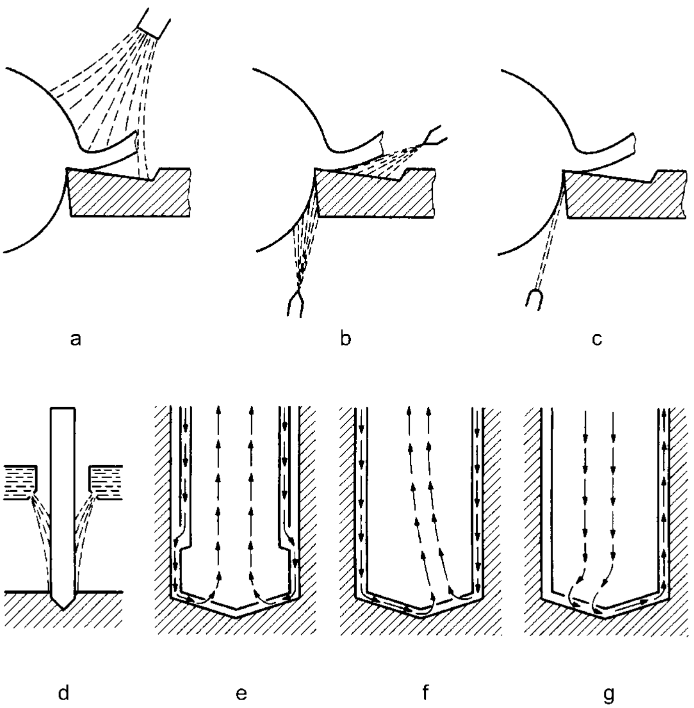


**Fig. 14.33** Schematic representation of some examples of metalworking fluid supply for cutting with a geometrically defined cutting edge. (a) frequently used flood method; (b) spray mist dosing; (c) microdosing, 'Hi-Jet'; (d) coolant supply using slug flow; (e) external coolant supply in gundrilling operations using ejector system; (f) external coolant supply in gundrilling operations, using BTA system; (g) internal coolant supply in gundrilling.

siderable nuisance caused by oil mist created by the coolant. Likewise, because of the considerable workplace nuisance through oil mist, spray lubrication cutting can only be applied in some special cases (for example, thread cutting). Hi-Jet and spray lubrication of the nineteen seventies and eighties can be considered to be the forerunner of minimum quantity lubrication.

Figure 14.33 shows some possible metalworking fluid supply systems for cutting with geometrically defined cutting edge.

### 14.8.1.1 **Grinding**

Where cylindrical grinding, centerless grinding and peripheral grinding are concerned the coolant is supplied in the turning direction of the grinding wheel. In this way the coolant is delivered on the one hand by the grinding wheel but on the other hand by the workpiece being wetted with coolant in the tool work interface.

Through the high periphery speed of the grinding wheel a rotating air belt is built up which repeatedly attempts to spin off the coolant before it gets to the grinding wheel. As a result the coolant has to be delivered under pressure; by this means it has also been possible to improve the free flushing of the grinding wheel. Coolant nozzles are also mounted at some distance away from the grinding point merely to deflect this rotating air belt. Delivery under pressure has a considerable influence on the development of foam which must be taken into consideration when selecting the metalworking fluid.

Many special variations have been developed especially in nozzle design; special nozzles to deflect the rotating air ring are significant in this respect. Central supply of coolant through the spindle has proved successful when grinding smaller holes and blind holes, and is worthy of mention here.

Compulsory pressure chamber cooling has proved successful in the case of flute grinding of twist drills. The workpiece and grinding wheel are sealed to such an extent from the pressure chamber, which is filled with coolant , that the main flow of fluid has to leave the chamber via the grinding gap. This ensures particularly effective wetting of the grinding wheel.

The installation of coolant nozzle rings around the grinding wheel to improve the cleanliness of the grinding wheel has proved successful to ensure good workpiece surface finishes.

In deep grinding, a high performance grinding method, it is not the breaking of the rotating air ring but the matching of the coolant exit speed to the peripheral speed of the grinding wheel which leads to improved wetting [14.102].

Figure 14.34 shows some examples of coolant delivery for grinding. IWT Bremen recently developed a test procedure for evaluation of the optimum volume of grinding fluids and the pressure distribution and maximum coolant pressure in the grinding arch [14.152].

### 14.8.2
### Individually-filled Machines and Central Systems

Cutting fluids can be supplied to a machine tool from a central circuit or from a tank which is an integral part of the machine. If a number of machines are set-up to machine the same material and the operations can be performed with one cutting fluid (or emulsion in the case of water-miscible cutting fluids), most end-users choose a central system.

In his very thorough cost analysis at DaimlerChrysler, A. Kiechle compared a 5 cubic meter individually-filled machine with a 150 $m^3$ central system. [14.103]

The costs analyzed in this study included the mixing and maintenance of cutting fluids, their disposal, the investment costs of a central system and energy consumption. The costs relate to 1 cubic meter of fluid. A. Kiechle came to the conclusion: “In principal, it should be noted that cutting fluid costs, calculated in DM $m^{-3}$ $year^{-1}$ are lower in large central systems than in individually-filled machines. In the two previously-mentioned examples, costs after all possible measures have been taken, amount to DM 1108 $m^{-3}$ for the central system and DM 9835 $m^{-3}$ for the single machine. This means that cubic meter cutting fluid costs in a central system are about one tenth that of individually-filled machines. Put another way: 150 $m^3$ of cutting fluid which generate annual costs of DM 166 000 in central systems would cost DM 1 474 000 if split between 30 single machines”.

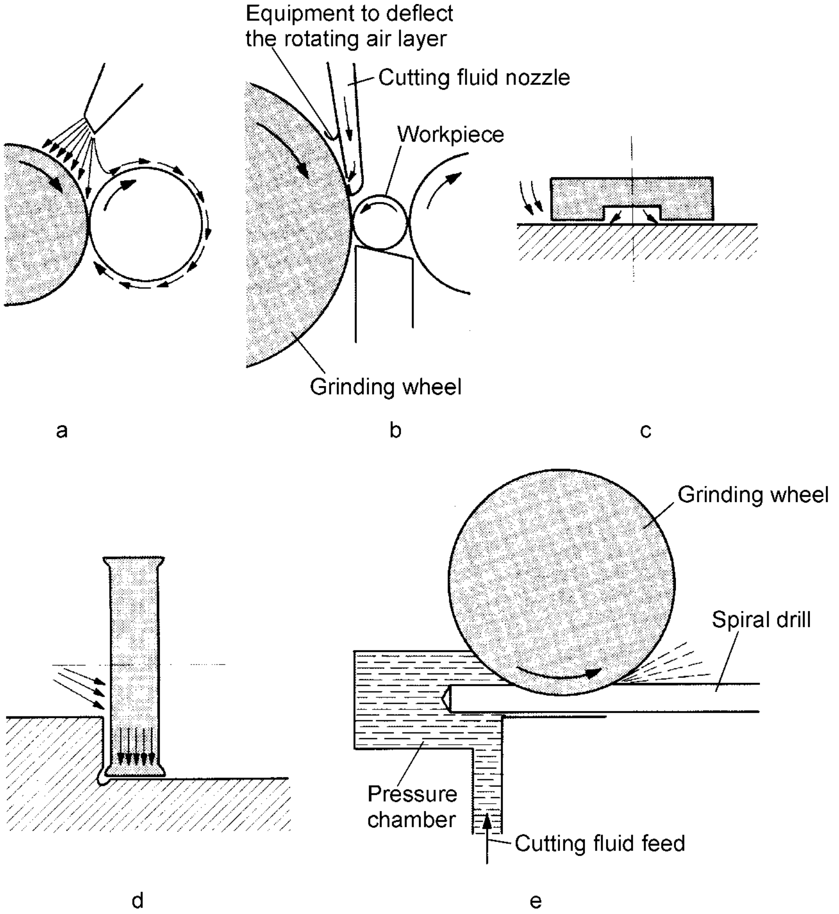


**Fig. 14.34** Schematic presentations of metalworking fluid supply when grinding. (a) Normal supply for cylindrical grinding (b) Supply for centerless grinding (c) Face grinding; external and internal coolant supply (d) Additional coolant supply for surface grinding of vertical surfaces (e) Coolant supply through pressure chamber for flute grinding of spiral drills (according to Gühring).

### 14.8.3
### Tramp Oil in Coolants

Tramp oils (hydraulic oil, gear oil, way oil, corrosion protection oil), which get into the water-miscible coolants should be removed as quickly as possible. It is seldom desirable to emulsify these with oil where the occurrence of tramp oil is low; the emulsions used for this purpose must be basically suitable. The coolant properties are altered when emulsifying and frequently result in the stability and corrosion protection being detrimentally influenced. Floating tramp oil prevents the supply of oxygen and promotes anaerobic degradation of the coolant which is often associated with considerable Monday morning odour.

Centrifuges are suitable for separating tramp oil in central systems; unlike other separators they do not depend on a floating tramp oil layer. However, this type of separation calls for careful coolant control. Where separator operation is continuous even some of the emulsion can also be separated which, in turn, leads to the emulsion being weakened.

Floating skimmers are very suitable and call for relatively little maintenance. The top layer of fluid in a coolant tank which is the tramp oil to be separated or which is at least enriched with tramp oil, is delivered by this facility to a tank with static oil separation or a centrifugal separator.

Belt skimming plants also have some application. In this case a circulating belt is immersed in the floating oil phase and the oil remains adhered to the hydrophobic belt surface and is removed. Examples of band material are steel, coated steel, plastics and textile materials. A high skimming rate can also be achieved even with very thin layers of oil by using band material made of highly oil absorbent felt with high oil storage capacity, and this has good regeneration by squeezing.

### 14.8.4
### Separation of Solid Particles

The object of the cleaning stage in a coolant circulation system is to separate metal chips, tool abrasion, solid contamination and graphite from cast parts and tramp oil; in some cases it also takes over the task of suppressing and dispersing the fluid foam. The function of the machine tool and the coolant circulation system itself is ensured by coolant solids separation; it also makes an essential contribution to achieving good surface finishes on the workpiece; over and above this, good coolant cleaning has a favorable influence on the sump life of the coolant. Solids separation of coolants in central circulation systems is a particularly economic process, with optimal systems of low intrinsic costs. Coolant solids separation in individually filled machines are frequently only a compromise for cost reasons and call for intensive maintenance (VDI directive 3397 [14.104, 14.105]).

#### 14.8.4.1 Swarf Concentration and Filter Fineness

Dirt concentration and filter fineness have particular significance in finishing operations. Although coarse chips can be easy separated from the fluid in the case of rough machining operations very often abrasion occurs which no longer sediments because the particles are so small in size and, in the case of grinding wheel abrasion and graphite, because of low specific gravity. Where grinding is concerned the total concentration of solid particles before solids separation is generally in excess of 1000 mg $l^{-1}$ but should be between 10 to 20 mg $l^{-1}$ after solids separation. The maximum particle size (filter fineness) should be between 5 to 10 μm. Solid particle concentration between 50 and 100 mg $l^{-1}$ in the cleaned fluid with, for example, a maximum particle size between 20 and 50 μm is usually expected in the case of rough machining on transfer lines.

Filter fineness below 5 m is not possible with conventional emulsions since the filter fineness in this case would be within the particle size of the oil droplets.

Where deep filtration is used for non-water-miscible oils (for example, honing oils) or synthetic water-miscible products free of mineral oil (solutions) even filter fineness up to some 0.5 μm can be achieved.

#### 14.8.4.2 Full, Partial or Main Flow Solids Separation

As shown in Fig. 14.35 the solids separation stage can be incorporated in a coolant circulation system in various ways [14.106].

**Main Flow Solids Separation**

In this case the solids separation plant is located either downstream or upstream directly in the main flow of the circulation system. It must be capable of accommodating the total volume in circulation. The advantage of such an arrangement is an almost constant degree of solids separation of the coolant but the disadvantage is the susceptibility of the system to irregularities; in the case of cleaning system failure, delivery of the coolant will be interrupted and the cleaning plant will no longer operate when the machine tools supplied with the coolant come to a standstill (Fig. 14.35a).

**Part Flow Solids Separation**

By comparison the cleaning plant can be smaller in design and size for part flow cleaning because only 10 to 20 % of the volume of the circulation system is cleaned in this case. However, coolant contamination will be at a relatively high level although the investment costs are low (Fig. 14.35b).

**Full Flow Cleaning**

Even in the case of full flow cleaning the cleaning circulation system works independently of the actual circulation system. In this case unlike the part flow cleaning some 100 to 110 % of the circulation system volume is cleaned in the cleaning stage so that the contamination level is much lower (Fig. 14.35c).

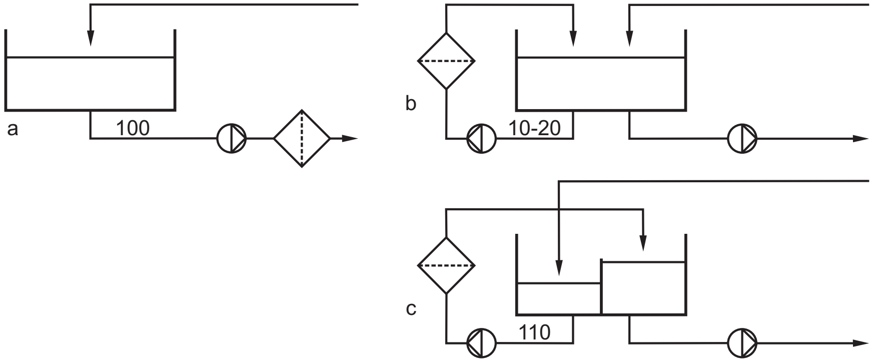


**Fig. 14.35** The various arrangements of cleaning stages in coolant circulation systems; the numbers are the percentages of the volume related to the circulation system. (a) main flow cleaning; (b) part flow cleaning; (c) full flow cleaning.

### 14.8.4.3 Filtration Processes

Different processes can be applied to clean the coolant, depending upon the type of machining, particle size, swarf concentration, specific gravity of solid particles, necessary filter fineness and type of coolant.

#### Sedimentation

The sedimentation of coarser chips by gravity in a sedimentation tank in the coolant circulation system provided for this purpose, while maintaining a steady flow of coolant is an essential part of pre-clarification. This can largely relieve the main cleaning operation. The sinking speed of the particles through the fluid is defined by Stoke's equation.

#### Flotation

Specifically lighter chips float on the surface of the coolant and, where necessary these can be removed by a skimmer. The actual flotation is achieved by attaching the solid particles to lighter droplets of fluid (thicker drops of oil in the emulsions) or to air bubbles. In this way the smaller solid particles are carried rapidly to the surface together with those which are heavier than the coolant. Use of air flow into the coolant for flotation is not usual for coolants used for cutting but has proved successful in some applications, for example, for cleaning emulsions used for copper wire drawing. Likewise, flotation is exploited by specifically adding oil to water mixed coolants, not where cutting is concerned but occasionally with rolling emulsions. By removal of tramp oil (for example hydraulic oils), the solid particles are also removed by flotation.

#### Screen Filtration

In this case particles, which are larger than the mesh or filter gaps and screen, are separated from the fluid by screens or filter belts. The retained solid particles must be quickly removed so that no flow-retarding filter cakes can form. The filter fineness corresponds with the mesh and gap width of the screen and filter.

#### Cake Filtration

In this case a filter cake is produced intentionally on the filter media. The filter cake comprising retained solid particles then takes over the actual filtering operation. In this case the resulting filter fineness is obviously less than the gaps and mesh of the filter media. As far as cake filtration is concerned there are considerable interactions between coolant and the solid particles which are important for the filtering operation. The degree of dispersion of the emulsions, the surface tension and the specific interface phenomena between coolant content and solid particle surface are the most important aspects. With cake filtration the aim is to build up a filter cake which is as thick as possible but still allows good flow properties. An adequate capillary area or 'peek' must be formed for this purpose in the filter cake and the water must be drained rapidly from filter cake. There is a rule of thumb which states that the remaining humidity of the filter cake and the filtering speed are indirectly proportional. Very fine grinding abrasion frequently forms dense filter cakes which result in filter medium consumption being high. In this case it is expedient to loosen the filter cake as far as possible by means of coarser grinding chips from rougher machining operations which are then mixed with the coolant.

Too coarsely dispersed emulsions and high amounts of tramp oil cause filter belts to stick and oily, difficult to handle, grinding sludge. Considerable improvements can be achieved by topping-up with specially formulated concentrates containing a high percentage of surface-active materials.

In the early stages of the cake filtration process the chemical nature of the filter medium, fiber tissue or cloth can have a great influence (ester groups in the case of polyester, alcohol hydroxyl groups in the case of cellulose). Consequently change of filter medium is one of the measures to be taken where filter problems occur. There can be considerable differences in filter media even with the same basic substance as a result of type of production, fiber direction and hydrophobic character.

### Deep Bed Filtration

In the case of deep bed filtration, filtering is mainly by means of a filter layer of fine grain material flushed onto a fabric (precoat filter). The layer made of diatomaceous earth or other filter media can react mechanically on the one hand and is absorptive in the filter operation on the other. In the case of mechanical separation, coarse particles remain occluded in the filter medium pores although where absorptive separation is concerned the particles are considerably smaller than the pores and absorption occurs on the pore walls through the interaction of the surfaces.

The mesh width of the fabric is about 100 to 160 μm, the particle size of the filtering agent is 1 to 50 μm. The filter medium form factor is the ratio of the average pore diameter and the average part diameter (for example 0.7 for diatomaceous earth).

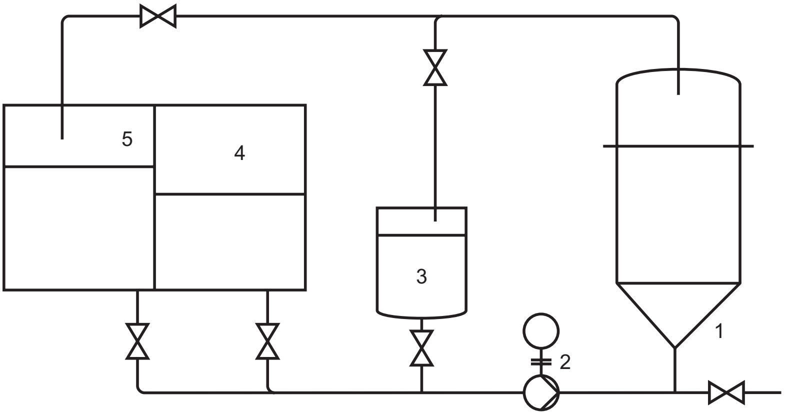
As far as the actual filtering operation is concerned there are transitions between deep filtration as precoat filtration and cake filtration since the filter cake made of solid impurities shows partial absorptive and mechanical deep filtration properties in the same way as a precoat filter.

The filter medium is frequently floated in filter cartridges. If the flow through such a filter achieves a minimum level because of the dirt in the filter medium, this can be discharged in the opposite direction by a rotation liquid. The flotation liquid (also a coolant) is cleaned by a belt filter. The filtering operation must be interrupted during the flotation operation period. With a special type of filter the filter cake can be dried in a filter unit and spun off.

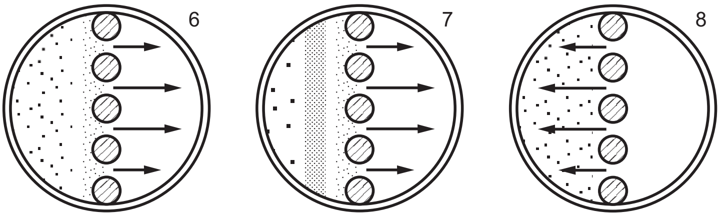
Precoat filtration is successfully used for grinding and honing oils. In the case of water-miscible coolant, application is limited to real solutions and to very finely dispersed emulsions; however this method has not become widespread in use to any great extent.

Figure 14.36 shows the principle of a precoat filter.

Figure 14.37 shows the filter process of cake filtration, deep-bed filtration and screen filtration [14.107].



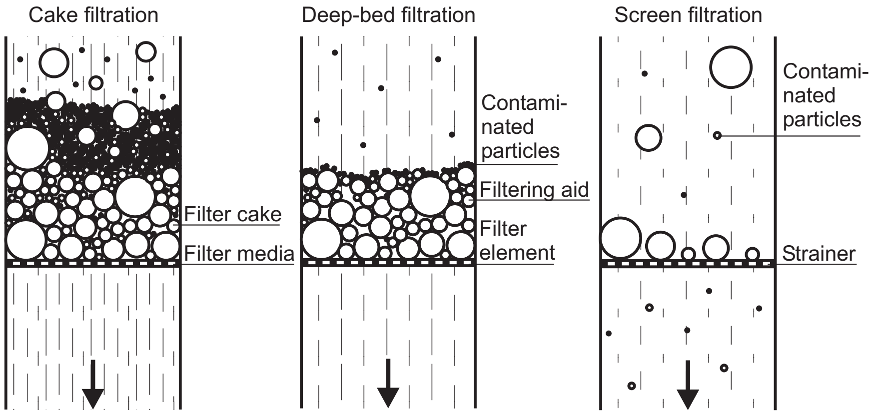
a) Basic arrangement of a precoat filter system



b) Description of filter process

| | | | |
|---|---|---|---|
| 1 | Precoat filter | 5 | Clean liquid tank |
| 2 | Filter pump | 6 | Precoating |
| 3 | Precoating tank | 7 | Filtration |
| 4 | Dirty liquid tank | 8 | Regeneration |

**Fig. 14.36** Principle of a precoat filter.



e.g. Pressure belt type filter, Vacuum type filter

e.g. Precoat filter, Cartridge filter

e.g. Membrane filter, Reversed osmosis

**Fig. 14.37** Filter process for cake filtration, deep bed filtration, and screen filtration.

### Centrifuging

The solid particles in coolants can also be separated by centrifuging. The centrifugal separation, according to the Stokes Law, is considerably higher than simple gravitational acceleration $g$ ($g$ acts in the case of normal sedimentation). For coolant cleaning by use of centrifugal separators $r\omega^2 \leq 8000g$, where $r$ is the radius, $\omega$ the angular velocity, and $g$ the acceleration due to gravity.

### Magnetic Separation

Ferromagnetic particles can be separated by magnets. With a particularly favorable agglomeration of non-ferromagnetic solid particles such as graphite or the bonding substance from grinding wheels on the ferromagnetic metal particles, separation is, to some extent, also possible by means of magnet separators. One significant aspect to be considered in the design of magnet separators is the severe reduction of the magnetic forces as the abrasion particles collect on the surface of the magnet.

### 14.8.4.4 Solids Separation Equipment

Numerous types of equipment are in use for cleaning coolants, using the methods described above. Certain types have gained particular importance and the most significant of these are described briefly below.

### Belt-type of 'Flat Bed' Filters

This type of filter has gained the greatest importance, especially in central coolant circulation systems because of their automatic operation. Either endless fabric belts are used as filter medium for continuous cleaning (generally only as screen filtration), or paper, tissue and fabric as filtering media are laid on the circulating underlay; such supported underlays are generally made of coarser fabric.

As can be seen in Fig. 14.38 belt-type filters [14.107] can be designed and built as gravity, vacuum and pressure belt filters.

In the case of gravity belt filters (Fig. 14.38a) it is only the pressure from the fluid columns above the filter which activates the mechanism. Relatively quickly, low flow rate activates feed of fresh filter medium by a float control system. As a result, relatively high filter medium costs are incurred using the cake filtration method. The equipment also needs a great deal of space.

As far as the so-called vacuum belt filters are concerned (Fig. 14.38b) a vacuum of up to max. 0.5 bar is achieved in the vacuum chamber. About four times the flow capacity is achieved per filter surface unit compared with gravity belt filters which, in turn, means that these are particularly suitable for large central systems.

The sealing between the filter media and the vacuum chamber and the enormous pressure on the container makes construction more expensive. The amount of space required and the costs for filter media are lower than for gravity band filters.

Filtering under pressure (Fig. 14.38c) in pressure belt filters takes place chargewise under a pressure of up to one bar on the feed side since filter changing makes it necessary to open the pressure tank. The specific throughput rate is about twice that of the vacuum belt filter; here again this makes this system particularly suitable for very large central systems. The pressure load calls for ruggedly built plants.

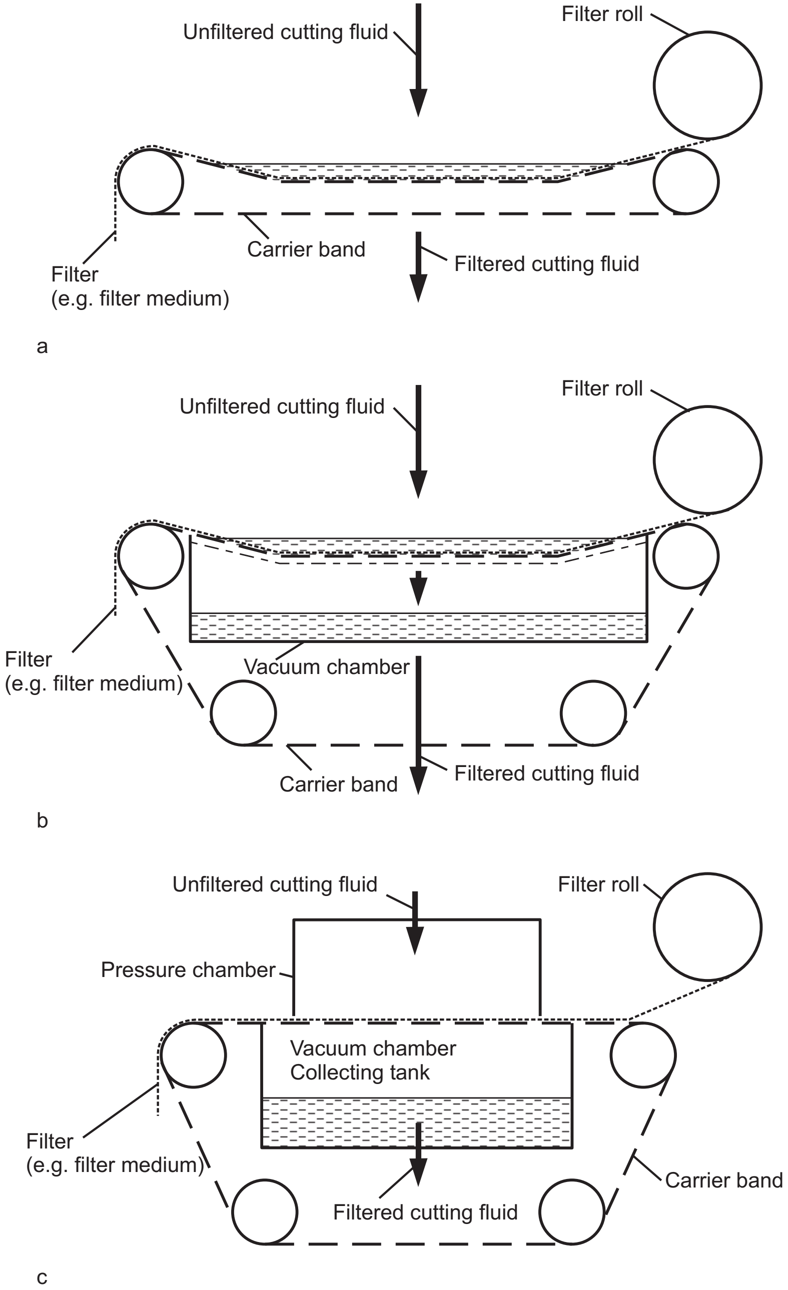


**Fig. 14.38** Belt-type filters.

The types of belt filter can be used for a wide range of metal machining processes both with water miscible fluids as well as neat cutting oils. Special pre-tests are necessary for the fine machining of gray cast iron. An upper viscosity limit of 30 $\text{mm}^2\ \text{s}^{-1}$ at 40 °C must be considered as a general rule for gravity and pressure belt filters when working with neat oils. Pre-separation is necessary when using pressure belt filters for rough machining operations.

**Hydrocyclones**

Hydrocyclones (Fig. 14.39) are only suitable primarily for cleaning water-miscible coolants, the fluid being delivered at high velocity by the pressure at approx. 2.5 bar and into the upper cyclone chamber. The particles which are heavier than the fluid are removed by centrifugal separation in a primary fluid cyclone; this means that where emulsions are concerned no oil is separated because of the specifically lighter oil phase. Although the separated solid particles on the cyclone wall are discharged downwards (run down the wall) the cleaned liquid is brought to the overrun by the secondary fluid cyclone; as a result of this even the secondary fluid cyclone has essential separation function. If the flow volume of the overrun is defined as 100 % then the underflow of the concentrated solid particles is about 2–6 %, the complete flow being 102 to 106 %.

The particle size which could be separated in practice for steel machining is between 15 and 25 μm, depending on the particle geometry. The lower density graphite will not be separated when machining cast iron. Since the cyclone discharge opening is but a few millimeters in size the machine is sensitive to arrested coarse particles or unfavorable agglomerations through clogging. As a result, cyclones are only suitable for grinding operations where specifically heavy and geometrically favorable particles > 20 μm in size have to be separated (for example, cylindrical grinding of steel). Normally the various hydrocyclones have a low capacity; this is why batteries of cyclones comprising several cyclones are installed for central systems.

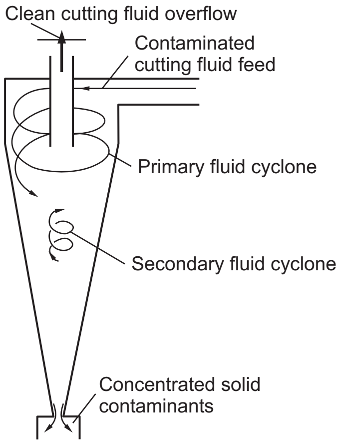


**Fig. 14.39** Hydrocyclones.

### Cartridge Filters

These are used frequently in the case of stand-alone machines with low volume flows and with a lower contamination load than pressurized filters. Filter inserts of very different types can be fitted in the standard filter houses. Paper and cloth filter are particularly popular as filters. The degree of contamination of the filter can be indicated by the differential pressure measurement. A very high degree of purity can be achieved, when the filter media is adapted to the cleaning problem. Unfortunately this method requires high filter media costs and high maintenance expenditures

### Edge Filters

The edge filter is a filter for micro-filtering of grinding oils, particularly for tool grinding, that does not require the use of consumables. Because grinding dust from carbide metals is very fine-grained, however, filtration using conventional filter facilities, for example as belt filters and centrifuges, does not meet the requirements imposed for these applications [14.153].

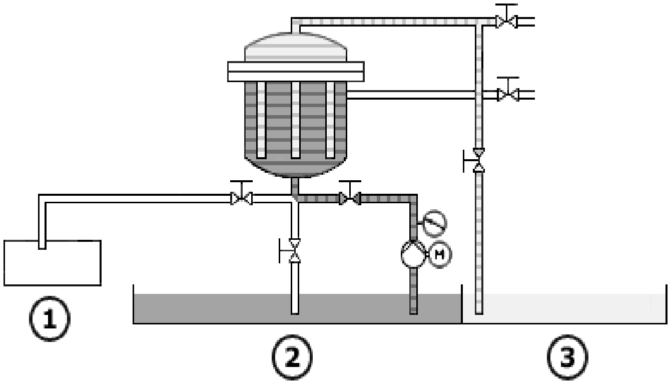


**Fig. 14.39a** Edge Filter
1. Sludge box 2. Dirty tank section 3. Clean tank section

In normal operation, dirty oil from the machines is taken from the dirty oil storage area and introduced into the filter vessel. Oil is forced to the center of each element. All particles larger than 1 µm are trapped on the edge of the filter elements. Clean oil is then deposited in the clean oil storage area and is delivered to the machines on demand [14.154].

The differential pressure in the filter vessel increases as the amount of very fine-grained carbide metals on the filter cartridges increases, resulting in a drop in the rate of throughput. Cleaning can be performed quickly by blowing out the dirt and rinsing the filter cartridges. Continuous supply of oil to the grinding machine is ensured at all times by the rapid, automatic cleaning process.

Although edge filtration enables 1 μm (absolute) filtration for EDM and industrial grinding applications that use oil, for reasons of economy only products with a viscosity much lower than 10 $mm^2\ s^{-1}$ at 40 °C should be used.

#### Automatic Backwash Filters

A few types of automatic backwash filter are extremely versatile and offer very wide scope in application in coolant circulation systems. This is why built-in low maintenance suction filters with no filter consumption have proved successful in the tank of central systems. The filter cake is removed by compressed air in the contamination basin during a break in filtering and transported away with the sedimented coarse impurities by scraping conveyors. As a rule, an average degree of cleanliness is achieved in such systems.

#### Precoat Filters

Precoat filter systems cause relatively high plant expenditure and take up a great deal of space with comparably high filter media costs. Since the majority of systems are fully automatic in operation monitoring and maintenance costs are low. Their deep filter action makes these a favorite for the finest cleaning of neat cutting oils such as honing and grinding oils when a very high degree of purity is demanded. The alternating action of the contents of the coolant on the active surface of the filter media, which can cause selective discharge of these contents, is very often over-estimated in practice. Through the increasing use of low viscous oils, this type of filtration will gain in importance.

#### Centrifuges

These are also centrifugal separators. They have gained certain significance both with water miscible as well as neat cutting oils. However, the area of application is restricted by the high costs of the equipment and the considerable wear. Even in the case of self-discharging machines some of the coolant is discharged together with the separated solid particles, the recovery of which causes additional expense. Centrifugal separators can also separate fluid phases with different specific gravities, which is why they play a more important role for separating tramp oil from water-miscible coolants.

#### Magnetic Separators

These are used both for grinding and rough machining operations as well as for processing both water miscible as well as neat cutting fluids. However these are clearly more suitable for water-miscible coolants than for neat cutting oils. In the case of manually cleaned magnetic cartridges which can be immersed in the fluid, it is best to consider a low flow rate. Moreover, it is often expedient to set up several magnetic cartridges in a row. The most telling disadvantage of the discontinuous working magnetic separator is the considerable costs involved for monitoring and maintenance.

The continuously working magnetic separators are available as drum and belt versions; Fig. 14.40 shows a magnetic drum-type separator. Their advantages lie in the fact that no filter medium is required and the maintenance and monitoring costs are low. Generally an average degree of purity is achieved with magnetic filters.

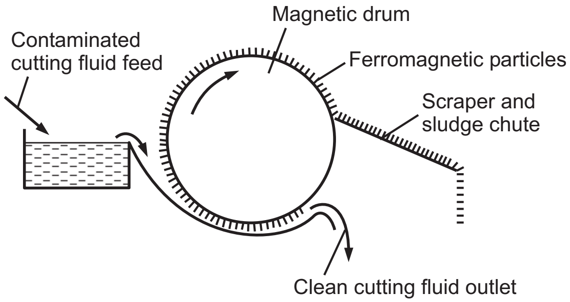


**Fig. 14.40** Magnetic drum-type separator.

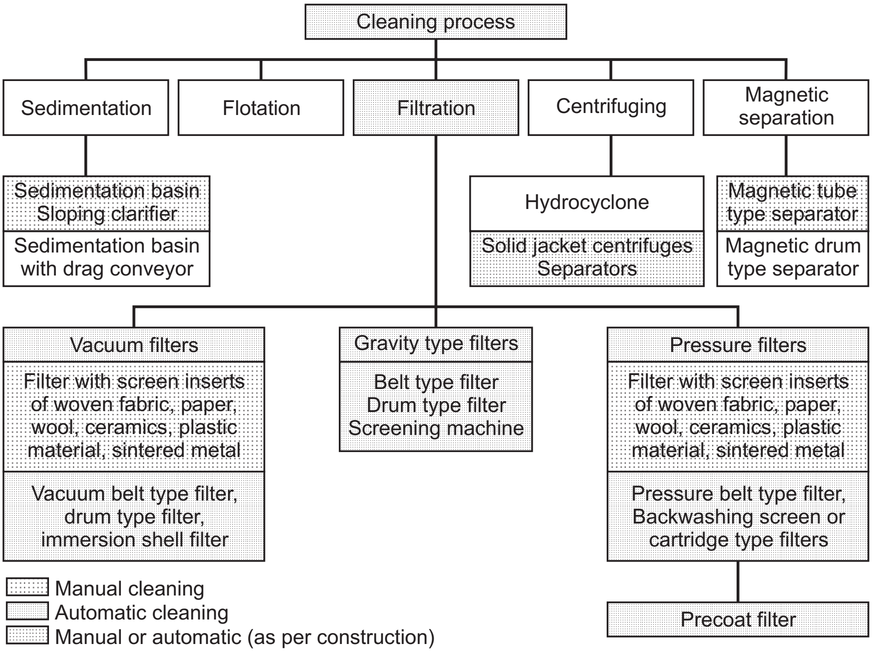


**Fig. 14.41** Cleaning processes and devices according to the VDI guidelines 3397, sheet 1, P. 4.

Figure 14.41 shows the cleaning processes and devices according to the VDI guidelines 3397, sheet 1, P. 4 [14.107].

### 14.8.5
### Plastics and Sealing Materials in Machine Tools – Compatibility with Cutting Fluids

Machine tools contain a variety of plastics which come into contact with the cutting fluid and all the other lubricants used. Figure 14.42 shows the possibilities of using plastics in machine tools.

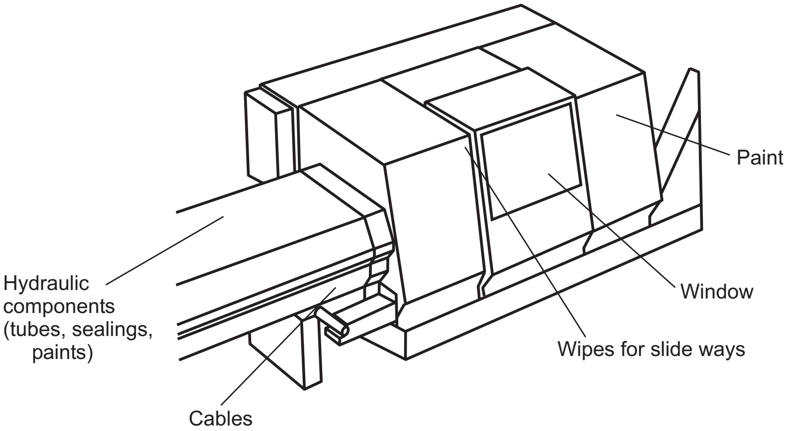


**Fig. 14.42** Possibilities of using plastic materials in machine tools.

On average, a machine tool has seven different lubrication areas: the cutting fluid circuit, the hydraulic circuit, slideway lubrication, spindle lubrication, bearing lubrication, gearbox lubrication and in some cases, clutch lubrication. In spite of attempts to reduce the variety of lubricants used in machine tools, each of the seven areas above are still often lubricated with a different product. This makes life difficult for the design engineer who has to select plastics which are compatible with the respective lubricants.

Naturally, the greatest significance is attached to the cutting fluid because this, in general, has contact with the machine's finish, cables, hoses, slideway bellows, screens and bearing seals. The materials used must therefore be matched to the cutting fluid type to avoid breakdowns. Hydraulic seals, pistons and tank finishes also have to be compatible with the hydraulic oil. A method of testing hydraulic oils was published in the form of the VDMA Guideline 24563. According to this, compatible plastics have to withstand 1000 h at 80 °C in the medium and still remain below the thresholds:

| | |
|---|---|
| Volume change: | 10 % |
| Change in Shore hardness: | 10 % |
| Elongation: | ± 30 % |
| Tensile strength: | ± 30 % |

Machine tool operators and design engineers should obtain corresponding information about compatible plastics from the plastic and lubricant manufacturers.

## 14.8.6
## Monitoring and Maintenance of Neat and Water-miscible Cutting Fluids

### 14.8.6.1 Storage of Cutting Fluids

Whereas neat cutting oils can be stored for a number of years, water-miscible fluids can be stored for between 6 months and maximum 1 year. Water-miscible products should be stored between +5 °C and + 40 °C. If the ambient storage temperature falls below 0 °C, the concentrate should be stirred (rolling the drum suffices) and warmed before use.

Containers should, preferably, be stored indoors. If drums are stored outdoors, they should be placed horizontally to avoid water and/or moisture seeping past the bung when the drum 'breathes'.

Containers used for the storage of cutting fluids should be clean, sealable and only used for one grade of cutting fluid. Do not use internally-galvanized drums because zinc soaps may be formed, thus negatively influencing the quality of the product. Storage tanks should be routinely inspected for contamination and cleaned if necessary.

If cutting fluids are stored in tanks, we advise performing bacteriological examinations once a year. If the result is positive, i.e. the contents are contaminated, the tank and any connected circuitry should be disinfected with the correct microbiocide.

#### 14.8.6.2 Mixing Water-miscible Cutting Fluids

If the mixing procedure described below is not observed, serious emulsion errors can occur. Emulsifiable concentrates which form oil-in-water emulsions must be mixed into water. Never reverse this procedure because lumps and gel can form. In the worst case, the mixture will have to be discarded and a fresh emulsion mixed. No special mixing procedures need be observed for genuine water-based solutions which contain neither mineral oil nor emulsifiers.

A major factor in an optimum emulsion is the quality of water used. The mixing water (preferably drinking water) should have a hardness of 175–350 ppm $CaCO_3$. The quality of the water should be known in order to avoid problems such as bacteria or extreme pH value. Local water companies can supply further details. If necessary, treat the mixing water (softening, demineralizing, filtering, hardening, bacteriological purification etc.) prior to mixing. The water used for the preparation of cutting fluids should be clean and neutral (with a pH of approx. 7). River or well water should only be used if it has been tested and, if necessary, treated because such water can be contaminated with microorganisms. These in turn can detrimentally affect the quality of cutting emulsions or solutions.

The water should not be too hard (precipitation of soaps) or too soft (foaming), but soft water can be hardened easily with calcium acetate.

Emulsions should be prepared with the assistance of mechanical stirrers, injectors or metering pumps. It is necessary to pay attention to pressure, temperature and viscosity parameters when using injector mixing equipment. These factors do not influence metering pumps. Pressure-reducing valves can stabilize variations in tap water pressure. It is important to follow mixing ratio recommendations and after mixing, the concentration has to be checked with a refractometer.

#### 14.8.6.3 Monitoring Cutting Fluids

Ideally, cutting fluids should be constantly monitored. Most importantly, solid and liquid impurities such as chips, abrasion residue and tramp oils should be removed by various centrifuges and/or filters because only clean cutting fluids increase efficiency and reduce scrap. The condition of the emulsion in the tanks should be checked at least weekly, better daily. Such measures can significantly increase the life of the cutting fluid.

### Neat Cutting Fluids

In general, neat cutting fluids are simple to monitor because they are less prone to microbiological attack and have a longer service life (longer oil change intervals) than water-miscible cutting fluids. The main parameters which need to be checked are physical contamination, thermal exposure, viscosity and additive level. These fluids are found in central systems as well as individually-filled machines.

### Water-Miscible Cutting Fluids

Water-miscible cutting fluids require much more monitoring than neat oils. Compared to neat cutting fluids, their change intervals are sometimes very short. The maintenance measures required for small tank capacities are often more complex than for larger central systems.

For both neat and water-miscible fluids, monitoring should not just be restricted to appearance and smell. Complex fluid tests should only be performed by highly-trained personnel. If concentration adjustments which can only be determined by sample analyzes become necessary, only about 48 h should elapse between the taking the sample and determining the result.

Tables 14.17 and 14.18 show the principal methods of testing the condition of neat and water-miscible cutting fluids.

**Tab. 14.17** The principal methods of testing the condition of neat cutting fluids.

| ***Properties*** | Norm test |
|---|---|
| Odour, appearance | Optical, sensory testing |
| Density | ASTM-D 1217, IP 249, DIN 51 757 |
| Liquid impurities | Refractive Index ASTM-D 1218, DIN 974, DIN 51 558 |
| Solid impurities | ASTM-D 893 |
| ***Corrosion protection:*** | |
| Nonferrous metals | ASTM-D 130, DIN 51 759-1 |
| Steel | IP 19, DIN 51 589 |
| Viscosity | ASTM-D 445, DIN 51 562 |
| Water content | Karl Fischer method, ASTM-D 808, DIN 51 777 |
| EP properties | Four ball app.; ASTM-D 2783, DIN 51 350-2/3 |

Tab. 14.18 The principal methods of testing the condition of water-miscible cutting fluids.

| ***Individually-filled machines:*** | | ***Central systems: (additionally)*** | |
|---|---|---|---|
| ***Properties*** | ***Norm Test*** | ***Properties*** | ***Norm Test*** |
| Odour, appearance | Optical, sensory testing | Tramp oil, non-emulsified Stand test | DIN 51 367 |
| pH value | Electrometrically;<br>DIN 51 369 | Solid impurities | ASTM-D 893 |
| Concentration | Hand refractometer | Electrolyte content | Conductivity measurements |
| Nitrite content,<br>max. 20 ppm<br>max. 50 ppm | Testing kit, photometric<br>Testing kit, photometric | Chloride content | Titration methods |
| Bacteriological contamination, (fungi and yeasts) | Dip-Slide method | Corrosion | Chip-filter test,<br>ASTM D-4627<br>IP 287<br>DIN 51 360-2 |

**Central systems**

The same tests and frequency as applicable for individually-filled should also be used for central systems. The test listed in Table 14.18 should only be used if required.

The amount of nitrite and nitrate in used emulsions and solutions should not exceed 20 mg $l^{-1}$ or 50 mg $l^{-1}$ respectively. If these values are exceeded, a full or partial exchange of the water-miscible cutting fluid may be necessary according to regulations valid in Germany, because these nitrosamine-forming substances (nitrite and nitrate) together with diethanolamines (secondary amines) which may be present, can form carcinogenic *N*-nitrosodiethanolamines.

### 14.8.6.4 Cutting Fluid Maintenance

Maintaining cutting fluids means taking measures to considerably extend the life of neat and water-miscible cutting fluids while the fluids are in use. Good maintenance will increase cutting performance and lower scrap. In addition, well maintained cutting fluids are less of a burden to machine shop personnel.

**Neat Cutting Fluids**

In general, neat cutting fluids are easier to maintain than water-miscible products. Important points are the removal of solid impurities and the maintenance of the original viscosity.

Dragged-in tramp oils such as hydraulic fluids can influence the viscosity and additive level to such a degree that reasonable cutting performance almost disappears.

If this condition occurs, suitable oil concentrates can return the additive level and viscosity of the used oil to that of the fresh oil and thus restore positive cutting performance.

#### Water-miscible Cutting Fluids

Water-miscible cutting fluids are a natural culture for microorganisms such as bacteria, yeasts and fungi. This means that these emulsions and solutions must be checked more often than neat cutting oils.

Of particular importance is the efficient removal of solids and tramp oils via filters or centrifuges. Maintaining the prescribed concentration is also very important because insufficient concentration promotes microbiological growth, lowers emulsion stability, lowers machining performance and accelerates corrosion problems on machines and workpieces. On the other hand, excess concentration can cause foaming and thus poor component quality as well as less efficient cooling and therefore less efficient machining. High concentration might affect machine paints, and could lead to dermatitis.

A crucial factor for these fluids is pH value. At low concentrations (5 %), the pH value should not drop below 8.8 and at higher concentrations (10 %), the pH value should not exceed 9.3 if the emulsion is fresh. In this pH range, emulsions are considered ‘non-irritating’. Depending on their age, the pH value of used emulsions can rise or fall. pH value increases are normally due to the presence of alkaline machine or system cleaners. A fall in pH value is normally caused by bacteriological attack.

This in turn can cause corrosion on machines and workpieces. Unpleasant odors can also be created and the emulsion may also become unstable.

Use of Dip Slides is recommended to determine the level of microbiological (bacteria, yeasts, fungi) contamination. If a microorganism content of more than one million bacteria, yeasts or fungi $ml^{-1}$ fluid is established, users should urgently contact their cutting fluid supplier to discuss biocide treatment.

Another worthwhile maintenance measure is to remove all floating contaminants (tramp oils and abrasive residues). Apart from potential blockage problems, these contaminants can weaken the stability of the emulsion as well as making it more prone to bacteriological attack.

Emulsion instability can also be caused by salts such as chlorides or sulfates dissolved in the water. This is why excessively hard water (> 440 ppm $CaCO_3$) should be avoided. Such water can also cause the formation of soaps. As too soft water (< 175 ppm $CaCO_3$) can result in heavy foaming, such water may have to be artificially hardened.

Another contributory factor to emulsion instability is high temperature. Large amounts of water can evaporate. This can result in higher salt concentrations (electrolyte) and ultimately lead to the emulsion splitting. Remedial measures include a partial exchange of the emulsion or topping-up with fully demineralized water.

A further maintenance measure is aeration of the emulsion. Bacteria have been found to multiply rapidly in emulsions which are cut-off from air supplies or if the emulsion remains stationary for too long. Sulfonates may also be reduced to $H_2S$.

This leads to highly unpleasant odors. If machining operations are shut-down, it is worthwhile simply turning on the circulation pumps to allow a degree of gas-exchange to take place.

Maintaining a constant (cool) temperature also increases the stability and performance of a water-miscible cutting fluid. Only in very rare cases is it necessary to heat an emulsion.

Cooling a cutting fluid usually improves tool life apart from reducing vapors. Another bonus of cooling is that microorganisms grow less quickly.

#### 14.8.6.5 **Corrective Maintenance for Neat and Water-miscible Cutting Fluids**

Both types of cutting fluids are required for metalworking operations and both occasionally generate problems which need to be solved (Tables 14.19 and 14.20).

**Tab. 14.19** Corrective maintenance for neat cutting fluids.

| *Problem* | *Corrective measure* |
|---|---|
| Fluid level in tank too low | Top-up; check for leaks |
| Excess solid impurities | Fit suitable filtering systems, centrifuges or exchange the fluid centrifuges or exchange the fluid |
| Liquid impurities (water or solvents) | Heat fluid, fit separators |
| Viscosity too high or too low | Top-up with an oil of different viscosity containing the same additives, determine causes, check for leaks, replacement |
| Corrosion (steel, metals) | Remove water, add corrosion inhibitors, replacement |
| Tramp oils | Reduce leaks of slideway, hydraulic and lubricating oils, replacement |
| Foaming | Check pumps for air leaks, check fluid level, add de-foamers, replacement |
| Oil mist, oil vapors | Use low-misting oils, add anti-mist , fit extraction to machines, improve cutting fluid feed to the cutting zone |

**Tab. 14.20** Corrective maintenance for water-miscible cutting fluids.

| *Problem* | *Corrective measure* |
|---|---|
| Fluid level too low | Add pre-mixed cutting fluid, check evaporation potential, check for leaks |
| Microbiological level 1 million (unpleasant odour) | Add pre-mixed cutting fluid including biocides |
| Fungal or yeast attack | Add biocides, test bacteriological contamination with Dip Slides |

### Changing Cutting Fluids

Even if a cutting fluid is optimally maintained, there is a point at which it may nevertheless need changing. The life of a new fluid batch largely depends on the cleanliness and disinfection of the system, particularly in the case of water-miscible cutting fluids.

A cutting fluid should be changed if specific values cannot be reached in spite of corrective maintenance. It is always worthwhile to perform a fluid change if system or machine cleaning operations are being undertaken.

#### *Neat Cutting Fluids*

In general, neat cutting fluids are very long-lasting. However, sometimes small-capacity circuits have to be changed more often than large central systems.

A neat fluid should be changed if solid and fluid impurities cannot be removed, tool life drops, filtering becomes difficult, the fluid ages excessively, machining finishes deteriorate, odour problems arise, or skin compatibility deteriorates.

Old fluids should be re-conditioned or correctly disposed-of. All pipework, tanks valves, nozzles etc. should be thoroughly cleaned.

#### *Water-Miscible Cutting Fluids*

As a rule, water-miscible cutting fluids are changed more frequently than neat oils.

A water-miscible cutting fluid should be changed if the fluid has an unpleasant odour, it causes gumming, there are observed heavy deposits on the machine, or thick foam floats to the top of tanks, pH value drops or rises, electrolyte levels increase, water is too hard, soaps are formed, the fluid itself thickens or becomes unstable, cutting performance declines, excess solid or liquid impurities are found, tool life falls, microorganisms flourish, fluid circuitry clogs, filterability deteriorates, or surface finish deteriorates.

A water-miscible fluid is changed in a similar way to neat oils, however, a system cleaner must be added before the system is drained.

This ensures that bacteria, yeasts and fungi in pipework are eliminated. Depending on the degree of contamination, the system cleaner should be added to the system 8–24 h before draining. After the old fluid and cleaner are drained, the whole system, should be disinfected again. Such thoroughness is necessary to ensure that the fresh fluid is not contaminated with residual microorganisms.

Used water-miscible cutting fluids can be reconditioned or disposed-of.

### Handling Cutting Fluids

The function of cutting fluids in machining operations is to minimize tool wear, maximize machine performance and to increase the quality of the components manufactured.

In many cases, machine personnel make unavoidable contact with cutting fluids. Without doubt, even dermatologically-tested cutting fluids can cause skin irritation. In addition, bronchial passages and mucus membranes can be affected.

A major problem is allergies. It is therefore absolutely necessary to take protective measures to minimize the hazards to people and the environment. Table 14.21 shows the precautions to be taken.

**Tab. 14.21** Precautions to be taken when handling cutting fluids.

| | |
|---|---|
| Avoid contact with cutting fluids | Safety instructions should always be readily available |
| Wear oil-resistant protective clothing | Observe safety-at-work regulations |
| Wear eye protection | Only use cutting fluids which are free of nitrite, chlorine and diethanolamines |
| Offer skin protection advice | Encapsulate machines |
| Apply a barrier cream on the hands and arms repeatedly throughout the day<br>If working with oils, use a water-soluble cream<br>If working with emulsions, use an oil-soluble cream | Ensure adequate ventilation in machine shops |
| Do not use abrasive hand cleaners | Fit oil mist and oil vapor extraction to machines |
| Only use pH-neutral soaps | Be aware of fire hazards when machining magnesium or magnesium alloys |
| Do not clean oily hands in cutting fluids<br>Do not wipe hands with dirty rags<br>Dry hands with clean, soft paper towels<br>Change soiled clothing | Use suitable binders (granulate) on soiled or oily floors |

### Disposal of Cutting Fluids

Observe local waste regulations when disposing of used cutting fluids. Every operator is naturally interested in reducing the disposal costs for used cutting fluids. This highlights the absolute necessity of selecting the best cutting fluid for the job in hand and of maintaining it properly in order to minimize the amount of cutting fluid which has to be disposed-of.

## 14.8.7
## Splitting and Disposal

### 14.8.7.1 Disposal of Cutting Fluids

Maintenance procedures allow cutting fluids to remain in use for long periods of time. Nevertheless, a point can come at which the level of solid and fluid contaminants has reached such a stage that the fluid has to be disposed-of and the system refilled with fresh fluid.

This poses no particular problem in the case of neat cutting and grinding oils which may either be regenerated on-site and used for less demanding applications, used as heating oil or commercially disposed-of.

The situation is totally different for water-miscible products which, containing an average of 3 % organic compounds, are water-polluting substances and cannot thus be discharged into rivers or water purification plants. In most cases, expensive treatment is thus necessary. The various separation processes available are not always ideal for the different types of cutting fluid in use. Moreover, the evaluation of the water phase remaining after separation depends on not just national legislation but also local waste water regulations.

### 14.8.7.2 Evaluation Criteria for Cutting Fluid Water Phases

#### Microorganism Toxicity

To feed cutting fluid water phases into waste water treatment plants which operate on biological principles, it is important to know to what extent the substances added retard the plant's sludge flora and to what extent they are decomposed by the existing flora in the plants sludge. Some substances are toxic to the plant's biological system and the use of biocides in cutting fluids is often discussed.

#### Biochemical Oxygen Demand (BOD)

The first important stage of microbial breakdown of organic substances is the oxidation of carbon and hydrogen to $CO_2$ and $H_2O$. The oxygen necessary for this process is taken from the water in which about 5 to 10 mg $l^{-1}$ is dissolved. The oxygen required by the microorganisms to perform this oxidation is called the BOD. For practical and analytic reasons, the value often used is the BOD after 5 days ($BOD_5$) determined in exactly defined laboratory conditions.

#### Chemical Oxygen Demand (COD)

Chemical oxidation (dichromate, iodine and permanganate processes) can determine the oxygen required to fully incinerate the product. The different processes produce different results. German waste water law foresees the use of the dichromate process. The difference between the $BOD_5$ and the COD is known as the non-biodegradable part.

#### Mineral Oil Content

The petroleum hydrocarbon content is an important value for waste water treatment plants as well as for determining the degree of water pollution. Mineral oil in the water phase of an emulsion indicates incomplete separation. But even mineral oil-free cutting fluids can generate water-phases which contain mineral oils resulting from dragged-in tramp oils. Values (normally in mg $l^{-1}$) must be accompanied by the name of the process used.

### Petroleum Ether-extractable Components

The presence of petroleum hydrocarbons are most often determined by extraction with petroleum ether. However, it should be remembered that other products may also be extracted. Exact determination of hydrocarbon content requires another analytical step (e.g. chromatography). Petroleum ether extractables have been divided up into saponifiable and non-saponifiable components.

### Thresholds and Legislation

There are no national guidelines on the quality of separated-water in Germany and in Britain which can assist the operator of cutting fluid separation plants. Thresholds are set by local water authorities which can also reflect the local situation regarding waste water treatment plants and the condition of rivers and streams. This leads to greatly differing evaluations between areas. Apart from $BOD_5$ and COD and petroleum ether extractables, pH value, insoluble solids, a series of metals and ions such as sulfate, cyanide and nitrite have thresholds. As regards the latter, the importance is either their toxicity or, as in the case of sulfate, the damage it causes to concrete pipes and drains.

In Germany, national legislation only applies indirectly to water separated-out of emulsions. Among other things, the German Law on Water regulates the disposal of wastes into public sewers and rivers, the notification obligation if petroleum products are stored or the location of protected water zones.

German Waste Disposal Law deals with the treatment of sludges which remain after emulsion separation processes. This includes incineration, land-fill and special land-fill sites for hazardous wastes.

The German Law on Emissions is focussed on keeping the air clean. As regards disposal of cutting fluids, this is limited to incineration of separated oils and used emulsions. Extreme Pressure (EP) agents with high levels of sulfur and chlorine can be problematic.

Waste Oil Provisions, whose focal point up to now was protecting the ground, water and air, have gained economic significance with the increase in recycling.

The German Waste Water Levy affected all who disposed of waste water into rivers and streams. The level of the payments depend on the contaminants in the water. Such contaminants are determined by the amount of insoluble solids, Chemical Oxygen Demand and fish toxicity. Even though this levy does not directly affect the operator of emulsion separation facilities, but mainly the operator of waste water treatment plants, it does influence emulsion separation technologies and the possible initial formulation of emulsion concentrates. Those disposing of waste water into rivers etc. try to use the ‘causer pays’ principle to transfer the costs to the user of the cutting fluids [14.108–14.110, 14.155].

## 14.8.7.3 **Electrolyte Separation**

### Salt Splitting

Splitting water-miscible cutting fluids with salts is still widely practiced for conventional emulsions. The addition of salts such as sodium chloride, magnesium chlo-

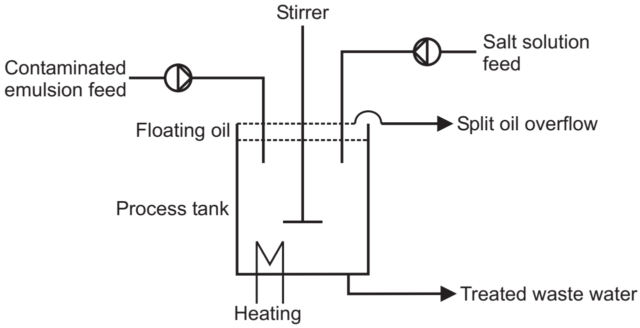


**Fig. 14.43** Emulsions undergoing salt splitting.

ride or calcium chloride affects the efficiency of the emulsifiers. This breaks down the emulsion and the lighter oil phase floats to the top. This floating oil can be collected by an overflow pipe or by pumping the water phase out from below. Large separation plants use centrifuges to accelerate the process. Overall, the capital investment costs of salt separation are comparatively low. Figure 14.43 illustrates the simple principle of salt splitting.

The splitting process can also be accelerated by heating the emulsion up to 90 °C. The emulsion and the splitting salts should be stirred vigorously to assist the process.

Price is a major factor in the selection of the salt. Sodium chloride is the cheapest but also the slowest. Ferrous and aluminum salts have good separation properties but require subsequent hydroxide precipitation with adsorption.

Separated water normally contains more than 150 mg $l^{-1}$ petroleum ether extractable components. As most thresholds range from 10 to 20 mg $l^{-1}$, this means that subsequent treatment is necessary. The considerable salt contamination in separated water which can be over 1500 mg $l^{-1}$, is a further limitation to the use of salt splitting because such salt concentrations are unacceptable for waster water treatment plants and, of course, rivers. The best salt splitting results are obtained with conventional emulsions using high levels of anionic emulsifiers. As the general trend is towards greater electrolytic stability of emulsions, electrolytic splitting processes are gradually disappearing due to their less satisfactory results with more modern coolants.

#### Acid Splitting

Similarly to salt splitting, emulsions can be split with the acids which many plants have used for etching processes (sulfuric and hydrochloric acid). Separating emulsions with acids is faster than with salts and, in the case of stable emulsions, also more effective.

Particularly effective acid separation processes are often combined with physical or physico-chemical splitting methods. Successful processes are those which employ splitting temperatures of over 90 °C and a downstream separation column filled with inorganic solids to accelerate the splitting process. Such acid splitting combinations can generate petroleum ether extractable values of under 20 mg $l^{-1}$. Neutralization of the separated water is necessary. The electrolyte content in the separated water, as in salt splitting, is also a problem.

### 14.8.7.4 Emulsion Separation by Flotation

In this procedure, fluid droplets are dispersed by air bubbles. Emulsion oil droplets and solid impurities are carried to the surface by the air. However, as only hydrophobic oil droplets float, hydrophilic oil droplets must be treated by breaking-down the emulsion. The air bubbles necessary for flotation can be created in a number of ways [14.111]:

- in release flotation, the air bubbles are formed as water which is saturated with air is de-pressurized.
- in stirring flotation, air is distributed throughout the fluid by rapid agitation.

Electro-flotation has become an accepted form of emulsion separation. The electrolytic breakdown of water releases hydrogen and oxygen. If total-loss electrodes are used, the metal ions (iron, magnesium and aluminum) which end-up in solution split the emulsion. The precipitating hydroxide floc absorb the oil and then float back to the surface through a layer of oily sludge. This method is a combination of salt splitting, adsorption splitting and flotation. A major hazard of electro-flotation is the formation of mixed oxygen and hydrogen gases.

### 14.8.7.5 Splitting of Emulsions with Adsorbents

#### Adsorption with Amorphous Silica

Adsorption with hygroscopic, fine-grain (ca 10 μm) amorphous silica has been gaining popularity. The process involves the powdery adsorption medium being placed in the emulsion. The oily and watery sludges formed can be de-watered with belt-type filters or filter presses.

To keep the cost of the expensive adsorption medium under control, adsorption separation is often preceded by salt splitting because the adsorbent consumption depends on the amount of oil in the emulsion. A rough guide to the consumption of adsorbent is 30 % weight of the oil in the emulsion.

It must be remembered that non-ionic emulsifiers which are often unaffected by electrolyte splitting are easily adsorbed by amorphous silica. On the other hand, anionic emulsifiers are poorly adsorbed [14.112]. Hydrophilic amorphous silica, which is of major importance to adsorptive emulsion separation, is most effective on anionic-active emulsifiers.

#### Adsorption with Metal Hydroxides

In the case of salt splitting with ferrous or aluminum salts, subsequent alkalization can precipitate the metals as hydroxides. The hydroxide flakes thus formed readily adsorb oil droplets and emulsifiers. Anionic emulsifiers are much easier to process than non-ionic products.

In general, such combinations of salt and adsorption separation processes using aluminum and ferrous salts can provide good splitting results with low oil and salt levels in the separated water.

However, the disposal of oily hydroxide sludges is a big problem. The problem is somewhat eased if filter presses are used to de-water the sludge.

Figure 14.44 shows a schematic diagram of the emulsion splitting process with the two stages: salt splitting and adsorptive hydroxide precipitation.

### 14.8.7.6 **Separating Water-miscible Cutting Fluids by Thermal Methods**

Thermal separation processes use heat to evaporate the water in cutting fluid emulsions which is then re-condensed. Such processes require the implementation of some sort of energy recovery. Although disadvantages include expensive technical equipment and high energy costs, the most important advantage is its suitability for almost all types of water-miscible cutting fluids. Contrary to the previously-mentioned methods, thermal processes do not require a dispersed organic phase but can also separate real organic solutions, popularly known as fully synthetic cutting fluids.

#### Immersion Heaters

Immersion heater separation involves a gas- or oil-fired immersion heater being lowered into the emulsion. The water which evaporates and then re-condenses must normally be treated because organic compounds can be released during the evaporation process. This method has never found great acceptance because the exhaust gas problems can only be eliminated at great expense.

#### Thin-film Evaporator

This method has a promising future. The method involves the water in the emulsion being evaporated on indirectly-heated evaporation plates. The quality of the evaporated water normally requires some subsequent treatment but generally less than in the immersion heater method. Active charcoal filters have proved successful for treating the water.

#### Incineration

The incineration or thermal treatment of water-miscible cutting fluids works with some highly-concentrated cutting fluids. Burning cutting fluids in heating systems requires the cutting fluid to be clean and also some modification to the nozzles. In the case of conventional emulsions, heat output is reduced if the emulsion concentration is less than 6 %. The incineration of cutting fluids containing chlorine compounds can cause damage to the furnaces. And finally, cutting fluids with large proportions of EP additives can cause exhaust gas emission problems.

### 14.8.7.7 **Ultrafiltration**

This method of separating water-miscible cutting fluids has gained the most acceptance over the past few years. Ultrafiltration involves the cutting fluid being passed through a semi-permeable membrane under pressure. Water and low-molecular-

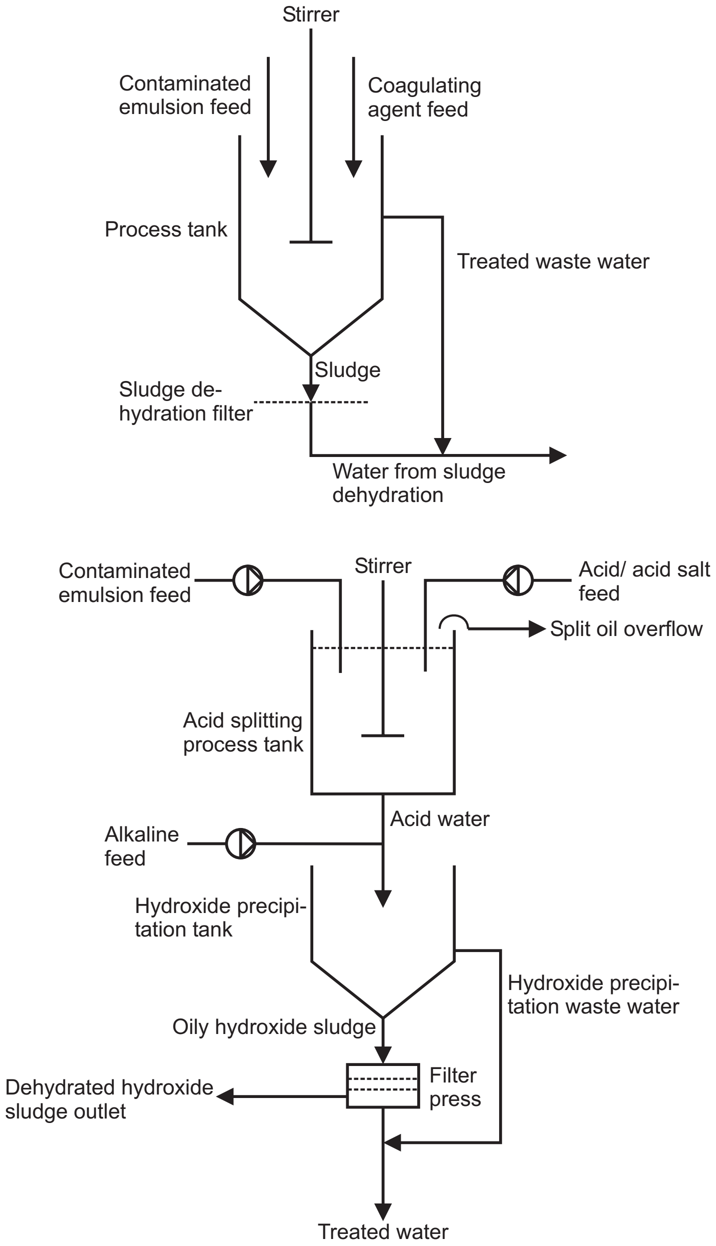


**Fig. 14.44** Splitting of emulsions with adsorbents and schematic diagram of the emulsion splitting process with the two stages; salt splitting and adsorptive hydroxide precipitation.

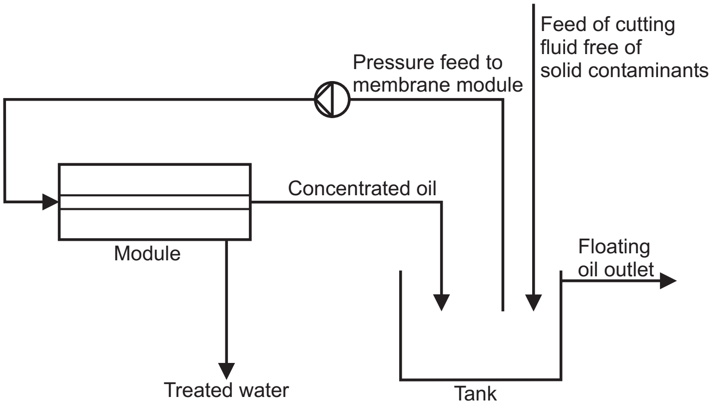


**Fig. 14.45** The continuous separation of water-miscible cutting fluids by ultrafiltration.

weight substances can pass through the membrane and form the permeate while oil droplets and large-molecular-weight substances are retained. The actual filter, which is the heart of ultrafiltration equipment, is often a stacked module in which organic materials such as polyamides, cellulose acetates or inorganic materials are used. If modules of organic materials are used, make sure that the fluid to be separated is free of solvents which can destroy the membranes. Figure 14.45 illustrates the ultrafiltration principle.

As separation performance falls with increasing concentration in the retained material, 30–50 % has proved to be an acceptable figure. The enriched, retained material could be treated by evaporators to produce material with less than 10 % water content.

Ultrafiltration plants consume little energy and they can mostly be run continuously and automatically. The capacity can be varied to meet needs by selecting the number of modules in the circuit. There are ultrafiltration plants with capacities ranging from a few hundred liters per day through to 1000 $l\ h^{-1}$.

The retained filtrate has a low hydrocarbon content but often a relatively high COD. This could become an important factor if ultrafiltration is evaluated according to the German waste water levy system. In some circumstances, ultrafiltered fluids can be reused similarly to the recycling of degreasing baths. With the necessary technical complexity through to reverse osmosis which can generate drinking water quality, this type of cutting fluid separation offers a wealth of further development opportunities.

#### 14.8.7.8 Evaluation of Disposal Methods

The selection of the disposal method must take into consideration the applicable thresholds, the cutting fluid volumes, the disposal of filter residues, investment costs, operating costs and personnel costs. If a metalworking factory only generates small quantities of used cutting fluid emulsions (e.g. less than 3 $m^3$ per week), in-house splitting is not recommended and it is cheaper to hire a disposal company. The most reliable method of meeting all waste water thresholds for all types of cutting fluids including fully synthetic solutions are the thermal processes. However, these normally require high capital expenditures. For lower investment costs, good waste water values, low personnel requirements and very low operating costs, ultrafiltration is a good alternative. Salt and acid splitting methods require the least investment but high operating costs, high personnel requirements and above all, residue disposal problems limit their practicability. For special applications, electroflotation and thermal acid splitting in columns are possible solutions.

Optimized combination of evaporation with ultrafiltration as methods of treatment could eliminate production of waste water in workshops. This complete water-recycling concept has been realized in some European manufacturing plants.

## 14.9
## Coolant Costs

This issue has had a considerable influence on the development of coolants in the nineties. This was not so much a matter of the costs of the volume of coolants bought by the consumer but rather the costs for the use of the coolants which are considerable, at approx. 12 % of production costs (serial production, central circulation systems). In this case the actual coolant costs themselves are only about 1 %. Pioneer work in system cost analysis has been done especially by Fuchs Petrolub AG [14.113], Daimler Chrysler [14.114], K. Weinert [14.115].

### 14.9.1
### Coolant Application Costs

Coolant application costs include the costs for the operation of all equipment, the investment costs as well as the coolant costs, costs for their preparation and disposal, energy costs, costs for coolant care as well as the costs for all auxiliaries.

However, to assess coolant systems the influence on the entire production sequence also has to be considered, for example, on tool costs, setting up times for tool changing, degree of machine utilization, workplace safety, workpiece quality.

#### 14.9.1.1 Investment Costs (Depreciation, Financing Costs, Maintenance Costs)

This is the most significant proportion of the costs and is some 60 % of the coolant application costs in serial production with central systems. This includes the costs of the plant tanks, pumps, pipelines, filter equipment, delivery of the coolant to the machine tool, de-oiling the chips, tramp oil separators and extraction systems. It is also possible to consider depreciation and maintenance for coolant splitting and disposal systems. Since preparation and disposal are frequently carried out by third parties it is more expedient to total these costs in a cost analysis (for example, as costs $m^{-3}$).

#### 14.9.1.2 Energy Costs

These include all the energy costs to operate the above-mentioned equipment including lighting.

#### 14.9.1.3 Coolant and Coolant Additives

Summarized under this are the costs for buying the coolant, chemical additives as well as the mix water.

#### 14.9.1.4 Coolant Monitoring

These are the costs for the analysis and labor costs for coolant care and monitoring.

#### 14.9.1.5 Other Auxiliaries

These are filter materials for coolant filtration and extraction.

#### 14.9.1.6 Coolant Separation and Disposal

Summarized here are the costs for the splitting of water-miscible products and the disposal of the oil and water phase.

### 14.9.2
### Coolant Application Costs with Constant System

Shown in the following is an analysis for the use of water-miscible coolants with a standard continuous system. It is assumed that the coolant circulation system is predetermined and cost minimization can only be achieved by selecting the right water-miscible coolants [14.113].

#### 14.9.2.1 Specific Coolant Costs

To make the following optimization considerations more understandable it is first necessary to define some terms.

##### Circulation Factor, *f*

This shows how many times the total volume of a coolant system – whether it is an individually filled machine or a central system – is circulated by pumping in 1 h. For example, the same number of machines can be supplied with a small volume in a central system and higher circulation number as with a high volume and a small circulation number. Here it is also obvious that the coolant in the system with a high circulation number is more stressed and the specific drag out losses – related to the volume – are greater.

The coolant requirement for machine tools is normally given in $l\ min^{-1}$. If the circulation factor, *f*, is only defined for the volume flow which runs through the machine tools and possible hydraulic chip transport by special coolant nozzles is ignored, then the following is valid:

$$f = 0.06B/V\ (\mathrm{h}^{-1})$$

where $V$ is the coolant system volume and $B$ coolant machine tool requirement in $\mathrm{l\ min}^{-1}$.

**Drag-out Coefficient,** $\alpha$

This is understood to be the number of times the volume of a plant is changed per month. A drag-out coefficient $\alpha = 1$ means that the total volume is taken out of the system and has to be replaced every month; a drag-out coefficient $\alpha = 4$ means change every week and $\alpha = 0.25$ change of the volume after 4 months.

Figure 14.46 shows the most important working area of coolant circulation systems concerning the drag-out coefficient and circulation factor.

The coolant losses take place on the one hand by chips, especially in the serial production sector with close interlinking of different working processes and through drag-out losses through geometrically complex parts such as, for example, engine crank or transmission housings.

High costs are incurred for the coolant itself and for the disposal of the coolant from systems with high drag-out coefficient $\alpha$.

In the case of a lower drag-out coefficient, these costs are low by comparison; however, the maintenance costs are higher through the rapid increase in the concen-

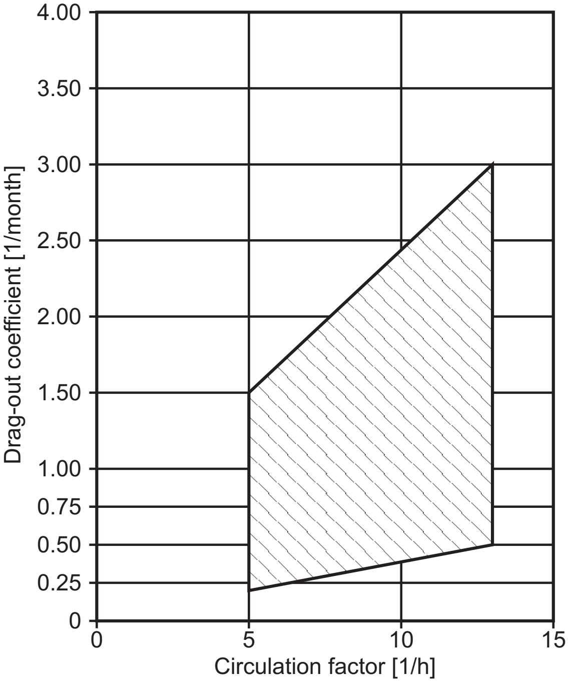


**Fig. 14.46** Most important working area for central circulation systems in respect of drag-out coefficient and circulation factor.

tration of harmful substances. Since, in systems with high drag-out, fresh coolant has to be filled over and over again, the concentration of harmful substances remains at a lower level. In this case the most significant harmful substances are tramp oils from leakage, other dragged-in coolants, corrosion protection agents from machined parts, dragged-in cleaners, heat treatment salts, non-filterable contamination from outside, water salt enrichment, decomposition products of coolant and microorganisms.

The following consequences arise due to over-concentration of harmful substances due to drag-out: either high care costs are obtained when using coolants with low drag-out coefficients, or more stable and thus also more expensive products must be used.

Figure 14.47 shows how the maintenance costs per kilo of water-miscible coolant used can depend on the drag-out coefficient; the curve shows the average values given in serial production over many years in studies.

The easiest way is to consider costs for coolant systems when both the different types of costs and the full costs are related to 1 m$^3$ volume of the system; 1 year is selected as the calculation period. The various costs can then be expressed as costs units (*KE*) per m$^3$ and year. The following breakdown has been split into three cost groups for the specific total costs *K*:

$k_1$ = Costs for coolant change
$k_2$ = Costs for drag-out losses
$k_3$ = Costs for coolant maintenance

This is then applicable:

$$K = k_1 + k_2 + k_3$$

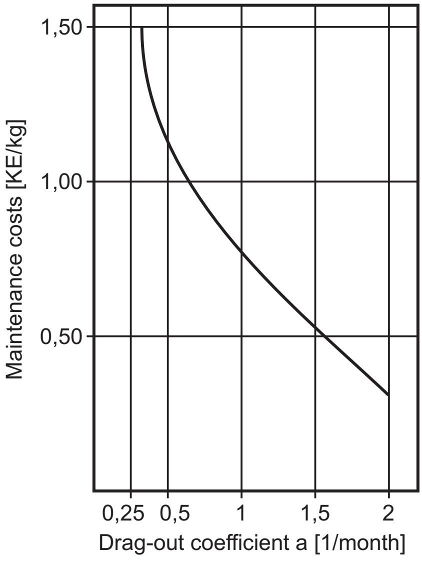


**Fig. 14.47** Maintenance costs.

$k_1$ are the costs of changing the coolant. Included here are firstly the costs for the new mix, that is to say, coolant concentrate + water, and secondly the disposal costs for the used coolant. Also considered is the work necessary to empty the coolant system and refill it; in the main these are labor costs, possibly machine and energy costs. If cleaning work has to be carried out which is only necessary in the case of coolant related changing, these costs are likewise to be included under k1; coolant changing is often carried out in the usual factory standstill periods to avoid interruption to the working process. However, if the coolant change has to be carried out while the factory is in operation and machine downtime costs are incurred, these have to be also considered under the costs for changing the coolant.

$k_2$ are the losses for coolant drag-out. In this case it can be assumed that the total volume of coolant carried off has to be disposed off through a separate drain, from the chip centrifuges or even through the washing fluid. In this case the disposal costs have to be considered.

The $k_3$ cost is the expenditure for coolant maintenance and monitoring. The costs for microbiocide and alkaline tankside additions must be considered here. However, also the laboratory monitoring costs to determine fluid condition, and also the microbiological tests are significant.

These costs can be calculated easily by determining the following variables.

Specific costs for the coolant change $k_1$ in KE $m^{-3}$ $year^{-1}$:

$$k_1 = w(10cP + E + N + WA)$$

Costs for the coolant discharge $k_2$ in KE $m^{-3}$ $year^{-1}$:

$$k_2 = 12a(10cP + E + WA)$$

Maintenance and monitoring costs $k_3$ in KE $m^{-3}$ $year^{-1}$:

The maintenance and monitoring expenditure costs must be determined individually. The total costs for one circulation system over one year must be determined and the result must be divided by the circulating volume of coolant.

The following equation is obtained for the specific total costs:

$$\mathrm{K} = (12a + w) \times (10cP + E + WA) + wN + k_3$$

In these equations $KE$ is the cost unit, $P$ is the Coolant price in KE $m^{-3}$, $WA$ is the price of water in KE $m^{-3}$, $E$ are the disposal costs in KE $m^{-3}$, $N$ is the working hours per change, i.e. possible machine standstill costs in KE $m^{-3}$, $a$ is the drag-out coefficient in 1 month, $c$ is the coolant concentration in %, and $w$ is the number of changes per year.

By means of these equations the specific coolant costs can be determined relatively easily for the different system situations.

The costs for changing the coolant and drag-out are easy to determine from factory records. At least the labor costs can be estimated to a large extent. Specific costs $K$, $k_1$, $k_2$ and $k_3$ can be presented by graphics, with their dependency on the frequency of the coolant change w according to the example shown in Fig. 14.48. There is no mathematical dependency for the care costs $k_3$; normally the maintenance costs increase with the extension of the coolant service life (reduction of the

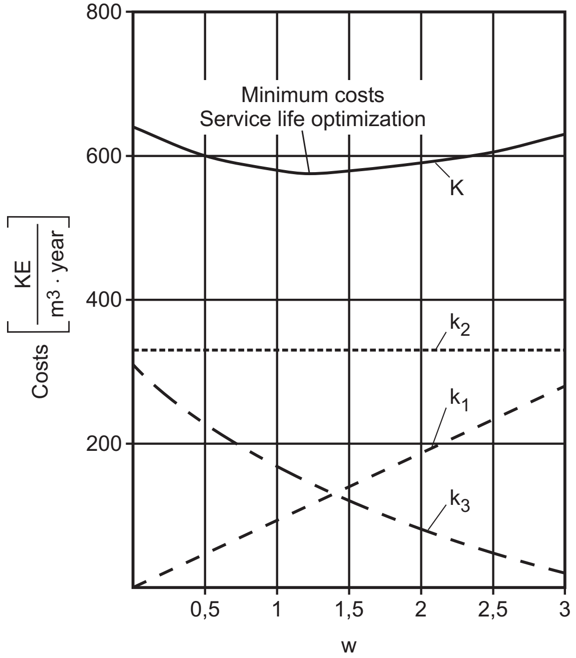


**Fig. 14.48** Dependency of the specific coolant costs on the number of coolant changes every year (from observations of a large central system).

change number *w*). The example in Fig. 14.48 shows observations for a large central system and also reflects many years of experience gained in calculating maintenance costs with different service lives. The addition of specific partial costs shows minimum costs (service life optimization) in the course of specific total costs, *K*.

### 14.9.2.2 **Optimization of Coolant use by Computer**

The cost equation developed for specific total costs *K* offers a favorable computerized route to optimize costs for coolant systems. If a number of different working conditions are programmed by drawing up a cost matrix, a document can be produced which simplifies both selection of coolant as well as measures for the coolant circulation system. Integration of care costs, coolant price, coolant concentration and coolant service life as variables is recommended; these are included in the cost equation by use of the terms *U*, *V*, *X* and *Z*:

$$K = (48C/B + 48/XD) + (10VFZS + Q + R) +$$
$$48(L + M + O)/XD\,B + 12U\,(G + H + J)/\,B$$

where $B$ = coolant circulation volume, $m^3$; $C$ = coolant drag-out, $m^3$ $week^{-1}$; $D$ = coolant service life, weeks; $F$ = concentration, %; $G$ = maintenance and monitoring: biocides, Euro $month^{-1}$; $H$ = maintenance and monitoring: tankside handling, Euro $month^{-1}$; $J$ = maintenance and monitoring: labor cost, Euro $month^{-1}$;

$L$ = coolant changing: labor cost, Euro change$^{-1}$; $M$ = coolant changing: chemicals, Euro change$^{-1}$; $O$ = coolant changing: machine downtime costs, Euro change$^{-1}$; $Q$ = disposal costs: Euro m$^{-3}$; $R$ = water costs: Euro m$^{-3}$; $S$ = coolant price: Euro kg$^{-1}$ (these definitions were used to adapt the usual records made in practice for the specific total costs in the cost equation [14.116]).

The overall cost equation is highly suitable for computer evaluation. The number of different variations of $U$, $V$, $X$, and $Z$ can be selected as required from the values 10, 20 or 30. Calculating about 20 different values has proved adequate in practice.

Because, in the first instance, the actual status of a coolant system is determined with a view to cost, the following questions can be answered:

- How are the specific total costs changed by the price of the water-miscible coolant (for example, by being 30 % higher, 30 % lower or half the price; in this case 1.3, 0.7 and 0.5 are programmed for Z in the cost matrix.
- How are the specific total costs changed when the coolant concentration is altered, the service life is changed or the coolant care costs are amended?

Results can also be obtained with the following problem when selecting coolant: By changing over to a new coolant which is 50 % higher in price ($Z$ = 1.5), only half the maintenance expenditure ($U$ = 0.5) should be necessary in the opinion of the coolant manufacturer or an own estimate; at the same time coolant service life is doubled ($X$ = 2.0) and a 30 % lower concentration ($V$ = 0.7) should be possible without negative effects to corrosion protection and cutting performance. The computer calculation of these variants in the cost matrix now shows whether the specific total costs for the studied system are lower or higher than the actual costs achieved.

When studying these costs it is assumed that the total coolant volume dragged out must be disposed of at full costs. If this is not the case this can also be considered in the cost formula [14.116].

Cost analysis for a large number of coolant systems show that the total specific coolant costs for individually filled machines and small central systems (up to approx. 3 m$^3$) are 5 to 20 times higher than those for large central systems (> 50 m$^3$). It can also be shown that coolants in the upper price bracket (three times the price of the bottom price bracket) with high stability and low requirements on maintenance in small systems, especially individually filled machines and even systems with particularly low drag-out, can result in lower overall costs than the low priced, less stable products. On the other hand it is also revealed that the lowest total costs are given in medium size and large central systems with particularly high carry-off of low priced products.

## 14.10
## New Trends in Coolant Technology

### 14.10.1
### Oil Instead of Emulsion

Back in the early nineties, the discussion about replacing conventional emulsions with neat oils was based on the concept of a total process cost analysis [14.117]. The background was the high cost of the machining fluids application (5–17 %) compared to overall process costs caused by the expense of maintaining and disposing of water-miscible cutting fluids.

These days, the 'oil instead of emulsion' trend is seen as an answer to a number of problems. Not only are cost benefits realizable, but environmental, safety-at-work and technical performance are superior. From a safety-at-work point of view, neat oils are more skin compatible than emulsions. They do not contain biocides and fungicides. Another major aspect is the almost unlimited life of oils compared to the fluid change cycles of water-miscible fluids (6 weeks for individually-filled machines and 2–3 years max. for central systems). Oils are also better in terms of environmental protection and protecting resources. As regards pure machining performance, oils can satisfy more than 90 % of all machining operations.

Replacing emulsions with oils offers better lubricity, improved surface finishes and significantly longer tool life. A cost analysis performed in a gearbox plant generated the factor 2 as an average of all machining operations [14.118].

Ten to twenty times longer tool life was recorded in CBN grinding and deep hole drilling trials. In addition, in-house corrosion protection for corrosion-prone materials such as cast iron and mild steel is normally not required. The same applies to machine tools which are protected against corrosion even if the paint finish is damaged.

The only disadvantages of machining with oils are in processes which generate a lot of heat. With a four-fold reduction in cooling, tool and machining problems can occur. Critical operations include the manufacture of carbide drills from solid stock.

To perform these processes with oil, the viscosity has to be reduced to a minimum. This generates the first conflict between technical performance and safety-at-work. The evaporation of conventional oils based on paraffinic solvent raffinates rises almost exponentially to falling viscosity. At the same time, the flashpoint falls. This problem can only be solved by the use of unconventional base oils which combine the advantages of a high flashpoint with low evaporation at very low viscosities.

In response to these requirements, the first cutting oils based on hydrocracked oils with esters appeared at the end of the eighties with pure ester oils established on the market in the early nineties.

The evaporation characteristics of different organic base oils are shown in Chapter 4. The very low values for ester oils are particularly interesting. Ester oils include products with different chemical structures, originating, above all, from oleochemical products, i.e. from animal and vegetable raw materials.

Apart from low evaporation, ester oils also display tribological advantages due to their significantly better friction behavior [14.119].

Even without any additives, they also offer improved friction and wear characteristics due to their polarity. This advantage as well as a high viscosity index, low water pollution hazards and good biodegradability predestines this group of base oils for use in machine tools as cutting and machine lubricants.

There are some special oils in use which, as alternatives to pure ester oils, are mixtures of hydrocracked oils and ester oils and thus combine excellent tribological properties with the somewhat lower price of hydrocracked products.

#### 14.10.1.1 **Fluid Families and Multifunctional Fluids for Machine Tools**

The decisive step towards cost optimization by harmonizing machine lubricants was first made possible by the trend towards metalworking with oils.

A much underestimated aspect of total cost analyzes is the influence of tramp oils on cutting fluids. Investigations in Europe and the USA have shown that 3 to 10 times the entire hydraulic fluid volume gets mixed with total cutting fluid volume every year. [14.120].

Figure 14.49 illustrates this effect over a period of 10 years at a European automobile manufacturer. In the case of water-miscible cutting fluids, the significant quantities of dragged-in oils caused some pronounced changes to the emulsion which led to machining problems, corrective action and considerable costs. In the case of neat oils, contamination with tramp oils cannot be seen and can only be assumed when problems such as poor dimensional accuracy and increased tool wear are noticed.

The trend towards machining with neat oils presented a range of cost-reduction possibilities. One analysis by a leading German machine tool manufacturer showed that an average of seven different lubricants are used in every machine tool. Apart form the leakage problem and the incompatibility of some lubricants, costs are also generated by the stocking of all necessary lubricants. Incorrect application of lubricants or grade confusion can cause considerable damage to machines or even production stoppages.

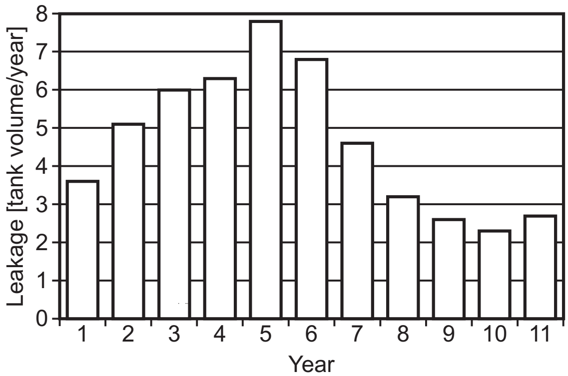


**Fig. 14.49** Hydraulic oil losses in machine tools (example taken from mass production in the European automotive industry).

One possible solution is the introduction of ‘multifunctional’ products which satisfy a wide range of machining operations at the same time as fulfilling the demands of advanced machine tool lubrication. A solution to the problems arising from the various lubricants necessary could take the form of a universal fluid for all machine lubrication points.

A factor acting against the introduction of such a universal fluid solution is the fact that conventional machine tools require ISO VG 32 or 46 hydraulic fluids because controls, valves and pumps are all designed around these viscosity grades.

On the other hand, the trend in metalworking fluids is towards lower viscosities which offer lower drag-out losses on chips and grinding sludge. Another reason for low viscosity oils is the trend towards high speed cutting which requires good heat dissipation for good results. This divergence between the viscosity of metalworking oils and machine tool lubricants still allows process cost optimization by harmonizing the additives in all machine tool lubricants. A machine tool fluid family contains the same or compatible additives in lubricants of different viscosities.

The advantages are:

- the unavoidable leaks of hydraulic fluids and slideway oils have no negative effect on the cutting fluid.
- manufacturing quality remains constant without complex analytical measures.
- tramp oils function as cutting fluids and thus do not create additional costs.
- higher process reliability, good machining results and reduced tool wear all serve to lower manufacturing costs.
- universal application.

Users can benefit from enormous rationalization potential regarding lubricant grades because a fluid family not only satisfies all lubrication requirements of machine tools but also those of various machining processes and materials. A typical example is engine manufacturing. One oil can be used for the rough machining of the block through to the honing of the cylinders. This technology offers sensational savings potential.

#### 14.10.1.2 **Washing Lines**

In process lines, water-based cleaning operations can be eliminated because undesirable mixtures are avoided as a result of compatible lubricants. Chips and solid contaminants are removed from the cutting fluid by ultra-fine filtration. Apart from the high investment costs of washing lines, detergent, energy, water and monitoring costs are also eliminated.

#### 14.10.1.3 **De-oiling of Chips and Machined Components**

The harmonized additives in all process oils allows the economical recycling of residual oil from chips, grinding sludge and components. Up to 50 % of the oil can be returned to the cutting fluid circuit.

#### 14.10.1.4 **Future Perspectives – Unifluid**

In the near future there will be one low viscosity oil which can be used as hydraulic oil and for different cutting and grinding operations. This 'universal fluid' (Unifluid) has been developed and tested in a German research project sponsored by the Ministry for Agriculture. [14.121]. The "Unifluid" with 10 $mm^2s^{-1}$ at 40 °C shows excellent results in a german automotive engine plant for machining and lubricating a complete transfer line (including the hydraulic system).

### 14.10.2
### Minimum Quantity Lubrication

The ever-changing legislation and an ever increasing awareness of the environment have led to a change in the production processes used up to now and especially in the production auxiliaries such as coolants. The increasing pressure on costs in consideration of international competitiveness for the German industry likewise calls for reduced prices within the given possibilities.

The analysis of an automobile manufacturer [14.122] published early in the nineties revealed that a considerable proportion of the process costs are caused by the application of metal working fluids. In this respect the costs for coolant concentrates themselves played an insignificant role. The actual costs are caused by system costs, personnel costs for the care and monitoring of materials, the high personnel and investment costs associated with splitting plants associated with water purification systems and last but not least, the costs for disposal (Fig. 14.50).

This has led to much more attention being paid to the difference between dry machining and minimum quantity lubrication [14.123]. The drastic reduction in the amount of coolants used as a result of these new technologies only offers a considerable potential for savings in process costs. However, dispensing with coolants also means that the primary functions of the coolants such as cooling, lubrication and chip transportation have to be replaced by other suitable technical solutions.

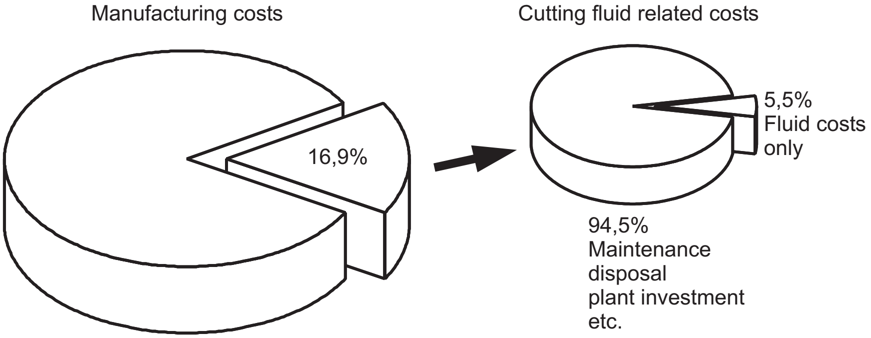


**Fig. 14.50** Coolant process costs. [14.22]

#### 14.10.2.1 Considerations When Dispensing with Coolants

If there is no coolant available in the process then the heat generated when machining by the workpiece, tool and chips cannot be dissipated. Inadequate heat dissipation can have considerable effects on the peripheral zones such as structural transformation, re-hardening and cracking and from deviations in measurement, which can make the component unusable.

Apart from the component properties, the influence on the tools should be especially noted [14.124]. A decisive role in this is firstly the heat dissipation and secondly the lubrication of the chips when the lubricant flows over the cutting edge. In the case of carbide metals the cooling effect has a negative influence on the service life because cooling can lead to cracks in the material. As a general rule, however, cooling the tool and especially the favorable friction conditions when the chips are carried off lead to the service life of the tool being increased. However, it has been shown that, particularly in the case of processes with a geometrically non-defined cutting edge, both cooling as well as lubrication are necessary to increase the service life of a tool. In the case of grinding and honing the cooling and rinsing effect of the lubricant is particularly important to the process. The coolant system plays an important part in the functionality of a machine tool. The chip transport plays a important role apart from controlling the temperature of the machine. Since the chip take up 80 % of the cutting heat the coolant has a double function in that it first dissipates this heat and secondly, the process heat and the chips can be removed from the machine tool to prevent the system becoming overheated, which can cause considerable dimensional fluctuations.

Figure 14.51 shows the coolant requirement dependent on the machining operation. Although dry machining is already possible today with interrupted cutting

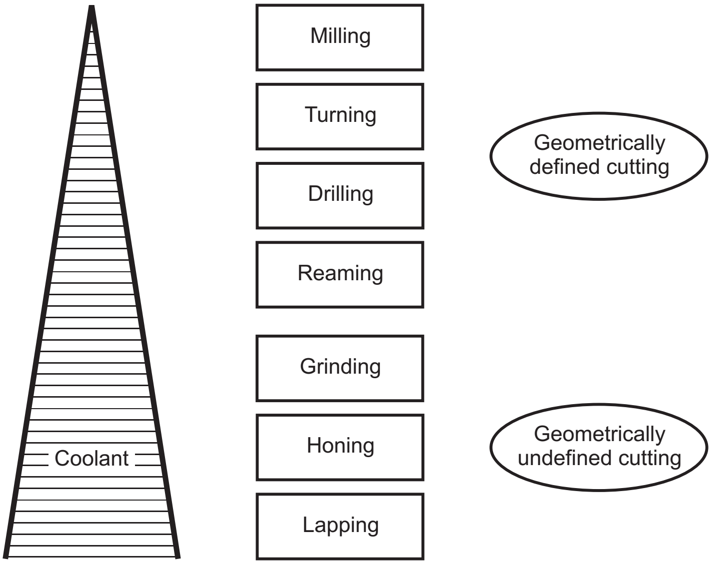


**Fig. 14.51** Dependence of coolant requirement on the machining operation.

such as milling, operations such as turning or drilling are only possible to a limited extent without coolant. Dry machining with geometrically non-defined cutting edges cannot be carried out because the cooling and rinsing effect of the coolant has a decisive influence on the quality of the workpiece and the tool service life.

The dry milling of cast iron and steel is quite achievable today with the help of tool geometries. However, the chips have to be cleared from the machine tool by other methods such as extraction or compressed air. As a result of this a number of new problems have arisen such as, for example, the noise nuisance, additional costs for compressed air as well as finest filtration of dust. Cr–Ni and Co dusts need to be assessed critically from the toxicological point of view, for which there are already threshold values on the workplace. Not to be ignored is the risk of dust explosion particularly when machining aluminum and magnesium.

#### 14.10.2.2 **Minimum Quantity Lubrication Systems**

By definition one talks of minimum quantity lubrication when the coolant volume flow does not exceed 50 ml $h^{-1}$. A minimum quantity lubrication system design is shown in the following Fig. 14.52 which reflects the general principle.

By means of a proportioning device a small amount of coolant spray (max. 50 ml $h^{-1}$ is applied to the machining point. Of all the dosing equipment on the market only two systems have proved successful [14.125]. The pressure systems have found widespread use, whereby differences can be made in this case between two strategies for preparing the mix. Firstly, there are the spraying systems where the medium (oil) and compressed air are mixed in the tank and the aerosol/air mix is transported by means of a supply hose directly to the machining point. Secondly, there are units with separate medium and compressed air supplies, where the medium and compressed air are delivered separately to the nozzle by the positive pressure in the storage tank. The volume per piston stroke and the piston frequency can be freely adjusted over a wide range. The amount of compressed air is dosed separately from this. Of advantage in the case of dosing pump systems is the possibility for computer control during a complex working process, apart from the many possi-

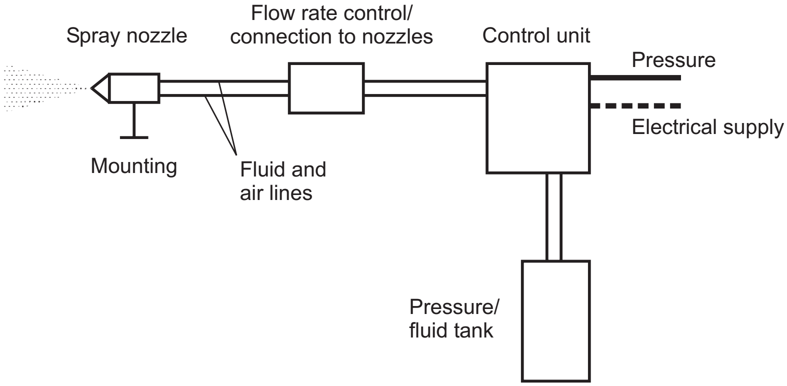


**Fig. 14.52** General principle of minimum quantity spraying system.

bilities for adjustment. Several setting possibilities can be adjusted to the process and be called up one after the other.

Because of the very low medium volume it is decisively important in the case of minimum quantity lubrication that the medium is applied very accurately on the machining point. There are distinct differences between internal and external fluid supply. In the case of external supply the mix is sprayed by one or several nozzles from the outside onto the tool. Conversion of a machining center is not a very costly business, installation is easy and the work involved is not extensive. However, in the case of external supply of the mix the length/diameter ratio is restricted to $l/d < 3$. Moreover, positioning errors can be made by the operator when changing the tool. The internal supply system allows the aerosol to be directly placed precisely in front of the machining point through the tool. In this way it is possible to achieve very deep bore holes ($l/d > 3$) and positioning errors by the operator are avoided. Apart from this chip removal from blind drill holes is greatly assisted.

The minimum tool diameter is limited to approx. 4 mm because of the coolant channels. Since the medium has to be supplied through the machine spindle system conversion is generally associated with high costs.

All minimum quantity coolant systems have one thing in common. The coolant is dispersed into more or less fine drops which reach the machining point as an aerosol. However this causes the first problems with regards to toxicological and workplace hygiene. Although for some years now, in the case of conventional coolant flooding at the workplace, efforts have been made to minimize spray development by new technologies and low emission coolants, sprays are now being intentionally developed for minimum quantity lubrication which are released into the workshop atmosphere.

The large number of minimum quantity coolant systems on the market [14.126] leads to wide choice of droplet size combination with greatly varying coolants. which still have not been studied sufficiently concerning concentration and particle size.

#### 14.10.2.3 **Coolants for Minimum Quantity Lubrication**

Today greases and oils as well as esters and fatty alcohols are in use, apart from conventional mineral oils and water-miscible coolants. Since minimum quantity lubrication is a matter of pure total loss lubrication and the coolant is frequently completely dispersed in the working area in the form of vapors and mist, particular attention should be paid to safety aspects. Ester oils and fatty alcohols with additives with low toxicological values have proved particularly successful in this respect.

Natural oils and fats have the disadvantage of poor oxidative stability. Ester oils and fatty alcohols should be given preference in the case of processes where high heat is developed to avoid residues on workpieces and machines. Table 14.22 shows the differences between esters and fatty alcohols [14.127].

For low-emission metalworking with minimum quantity lubrication, correct selection of the lubricant is of decisive importance. To minimize emissions, the lubricants used should be toxicologically and dermatologically safe, with favorable lubrication properties and high thermal stress capacity.

Synthetic ester oils and fatty alcohols, in particular, which have low evaporation characteristics, are toxicologically harmless, and have a high flashpoint, have proven themselves in practice [14.157].

Flashpoint (DIN EN ISO 2592) and Noack evaporation loss (DIN 51581 T 01), in particular, have been shown to be suitable reference values for selection of a low-emitting lubricant. The lubricant should have a flashpoint of at least 150 °C, an evaporation loss at 250 °C of 65 % maximum, and a viscosity at 40 °C of > 10 $mm^2\ s^{-1}$ [14.156, 14.158].

| | | | | |
|---|---|---|---|---|
| • | Viscosity at 40 °C | ($mm^2\ s^{-1}$) | > 10 | DIN 51562 |
| • | Flashpoint open cup | °C | > 150 | DIN EN ISO 2592 |
| • | Noack evaporation loss | % | < 65 | DIN 51581 T 01 |

**Tab. 14.22** Differences between ester/fatty alcohols [14.123].

| | *Ester* | *Fatty alcohol* |
|---|---|---|
| Evaporation | Very low | High |
| Lubrication | Very good | Low |
| Flashpoint | High | Low |
| Water pollution category | – / 1 | – / 1 |

Equiviscous fatty alcohols have a lower flash point than ester oils. They evaporate relatively easily and as a result have a low cooling effect. Compared with ester oils their lubricating effect is relatively low.

This is why fatty alcohols are used with preference for cutting operations where the lubricating effect takes second place to the cooling effect. Examples of this are the machining of gray cast iron where the graphite in the material provides the lubricating effect, or also when sawing cast iron, steel and aluminum. Fatty alcohols offer the advantage that the machined parts are dry as a result of the rapid evaporation. However this evaporation must be considered critically because of the threshold values for oil mist and oil vapor in the workplace (10 $mg\ m^{-3}$).

Ester oils are used with preference for all machining operations in which the lubrication effect between tool and workpiece and the passing chips is paramount. Examples of this are thread cutting, drilling and turning.

Ester oils have the advantage that they have a high boiling range and flash point despite lower viscosity. As a result of this considerably less vapor will be emitted into the workroom. At the same time the thin film remaining on the workpiece has a corrosion protecting effect. Beside these properties ester oils are rapidly biodegradable and, due to their biodegradable properties, are classified as non-water hazardous or as water hazard class 1 [14.128]. Table 14.23 shows a few examples of applications where ester and fatty alcohols are specifically used.

**Tab. 14.23** Examples of applications for the minimum quantity lubrication technique.

| *MQL-Lubricants (base oil)* | *Material* | *Process* | *Component* |
|---|---|---|---|
| Ester | Die cast Al | Sawing | Profiles (sections)<br>No residues up to 210 °C |
| Fatty alcohol | CK 45 | Drilling, reaming, milling | Housing components |
| Ester | 42 CrMo4 | Thread rolling | High surface quality |
| Fatty alcohol | St37 | Pipe bending | Exhaust systems |
| Ester | 17MnCr5 | Drilling, rolling, shaping | Splining of drive shafts |
| Ester | CK 45 | Thread rolling | Gear parts |
| Fatty alcohol | AlSi9Cu3 | Sawing | Gear box |

The main points to be considered when developing a coolant for minimum quantity processing are shown below. Apart from the performance aspect it is particularly important to make available low emission, dermatologically and toxicologically acceptable media with a high flash point. New research results are being presented which have been achieved in the fields of emission and of optimization of a coolant medium for minimum quantity lubrication areas.

### 14.10.2.4 **Oil Mist Tests with Minimum Quantity Lubrication**

When metalworking with minimum quantity lubrication systems, aerosols are generated which need to be delivered to the machining point, and high concentrations of the aerosol gets into the working atmosphere, particularly when using external spraying systems. Under less favorable conditions a major proportion of the sprayed aerosols are given off as an oil mist which, with a particle size between 1 μm and 5 μm, are defined as in a range accessible to the lung and should be viewed very critically.

The factors which influence the development of oil mist have been studied [14.129] (Fig. 14.53). It was of particular interest to assess the influence of viscosity of the fluid on development of oil mist. The aim was to discover whether and how greatly the oil mist (measured variable: oil mist index) can be reduced by increasing the viscosity. Also determined was whether the development of oil mist in the lung accessible range could be reduced by adding anti-mist additives. In a further series of test the delivery pressure of the spraying equipment used was varied to see whether significant differences were apparent in respect of the tendency to emit. A

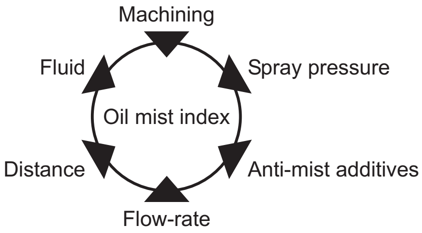


**Fig. 14.53** The factors affecting the development of an oil mist.

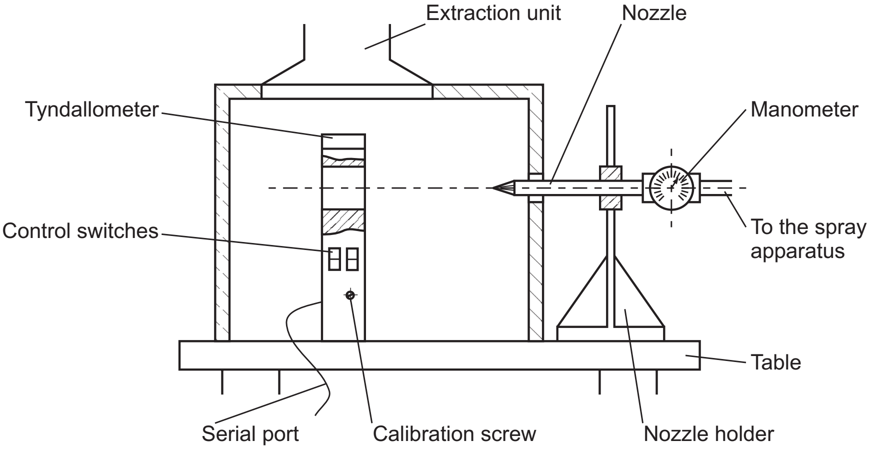


**Fig. 14.54** Structure of oil-mist measuring facility.

testing facility was built for this purpose with which it was possible to measure the given oil mist when spraying, by means of a Tyndallometer (particle size up to 5 μm) (Fig. 14.54).

The Tyndallometer used to measure the oil mist was positioned at a certain distance from the spray nozzle. The data determined by the measuring instrument was down-loaded to a computer which enabled the oil mist content and the characteristic curve of the spraying unit to be shown.

From the results it can be seen that the oil mist increases enormously as the spraying pressure increases. Especially in the case of low viscous esters, increasing the pressure has a very strong influence on the development of oil mist. Doubling the spraying pressure also led to the aerosol volume being doubled. However, where the spraying pressure was very low the starting behavior of the equipment was poor, i.e. the period before which the equipment is able to provide a constant volume of lubricant is extended. At the same time the oil mist index strongly increased with the reduction of viscosity of the medium. On the other hand, the starting behavior of the spraying equipment where low viscous media was concerned was considerably better than that with high viscosity media.

As a general rule it is possible, by adding special anti mist additives, to reduce the oil mist behavior in all the media of varying viscosity used (Fig. 14.55). Primarily with low viscosity media use of anti-mist additive enables the development of oil mist to be reduced by more than 80%. The measuring methods presented also make detailed statements possible on the spraying properties of a system in respect of start-up , spraying consistency and oil mist behavior.

It can be seen from the findings presented here that the emission can be considerably reduced by selecting the right spraying pressure and media viscosity. Adding special anti-mist additives also reduced the emission in all cases.

#### 14.10.2.5 **Product Optimization of a Minimum Quantity Coolant Medium for Drilling**

The machining of materials with minimum quantity lubrication (deep hole drilling ($l/d > 3$) with external spraying) was examined in a further study [14.130]. The drilling tests were carried out on a DMG milling machine (Table 14.24). The workpiece was a high alloyed steel (X90MoCrV18); this had a tensile strength of 1000 N mm$^{-2}$ for drilling blind holes. A full carbide metal drill with SE shaft with high edge and bending fracture resistance with a PVD-TIN coating. By selecting a high alloyed steel (X90MoCrV18) for machining as well as external spraying the lubricant used was tested and optimized under very demanding conditions. The aim of the test was to determine on tone hand, how the viscosity of the used ester and, on the other hand, the addition of specific combinations of additive, effected service life when drilling.

**Tab. 14.24** Drilling of steel with minimum quantity lubrication.

| | |
|---|---|
| Tool | Tungsten carbide with a TIN-layer<br>Type: SE Drill<br>D = 8.5 mm<br>L > 26 mm |
| Material | X90MoCrV18, high alloyed steel |
| Chuck | Hydraulic-expansion chuck |

The test bed allows the cutting forces to be measured in z direction with a Kistler measuring platform. The performance of the working spindle of the machine during the entire drilling operation was measured continuously at the same time. The two measuring methods allowed a statement to be made on both the forces used in a single drilling, as well as the continuous forces over the entire drilling test. Occurring faults such as, for example, chips sticking as well as the tilting of drills could be

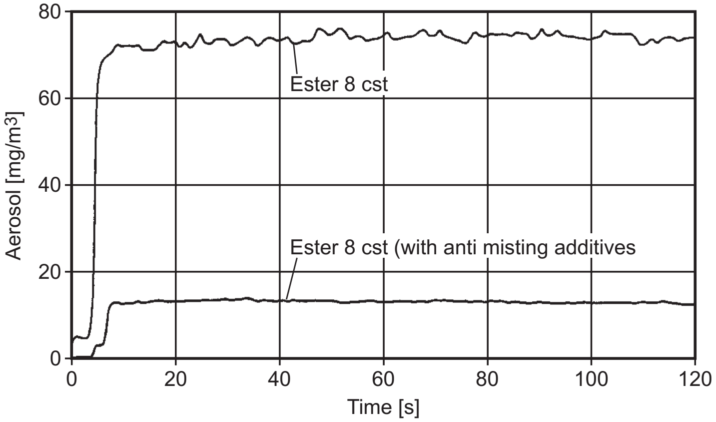


**Fig. 14.55** Aerosol behavior: influence of anti misting additives.
Ester (viscosity 40 °C = 8 mm$^2$ s$^{-1}$, pressure: 2.5 bar).

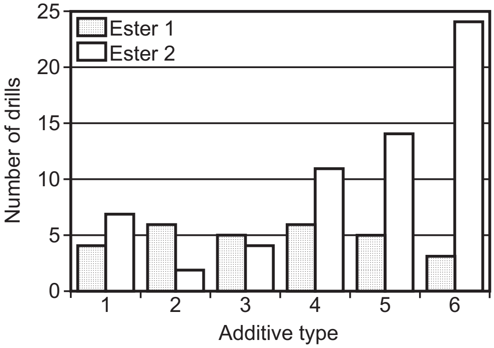


**Fig. 14.56** Influence of additives on two different esters with EP-additive; volume 10 g $h^{-1}$.

easily registered and as a result the series of measurements could be assessed better with regards to quality.

Figure 14.56 shows the performance of two esters, each with the same additives. As a general rule the drilling capacity of both media can be increased by selecting suitable additives. However, it could be clearly seen that special combinations of additive to both base oils in precisely the same dose had extremely different effects.

Finally it only remains to establish that the occurring oil mist emission can be decisively reduced by selecting suitable base oils, correct equipment setting and by incorporating anti mist additives. This leads to improvement in working safety and the improvement of toxicological, dermatological and safety implications in the workplace. Through the correct combination of additives the efficiency of a coolant for minimum quantity lubrication can also be considerably improved which, last but not least, leads to longer service lives and better workpiece finish.

# 15
# Forming Lubricants

According to DIN 8582 one differentiates between six main groups of manufacturing processes: primary shaping, forming, separating, assembling; changing properties of materials and coating. The processes listed under forming (DIN 8583 forming under compressive conditions, DIN 8584 forming under combination of tensile and compressive conditions, DIN 8585 forming under tensile conditions. DIN 8586 forming under bending conditions, DIN 8587 forming under shearing conditions), all refer to non-cutting production. Over and above this, forming technology also includes some sub-divisions under severing operations (DIN 8588 severing operations, shear cutting) and jointing (DIN 8589 jointing by forming).

Apart from the differentiation according to DIN 8582, in practical applications one normally divides the area of forming technology into sheet-metal forming and extrusion forming. This conventional distinction is based on the fact that unlike extrusion, sheet metal forming workpieces are produced with nearly uniform metal thickness over the complete product. Additionally, extrusion forming uses much higher forces than are required when forming sheet metal.

## 15.1
## Sheet Metal Working Lubricants

*Theo Mang, Franz Kubicki, Achim Losch and Wolfgang Buss*

The tribological system consists of three components: The workpiece, the tool and the lubricant. The workpiece and the tool are characterized by their metallurgical features. Their lattice structure is determined by their composition, heat treatment as well as by subsequent cold processes such as temper rolling or deep drawing. These determine the physical stability, the flow characteristics, the anisotropy and finally, the formability of the material. As friction and wear take place at the surface, the material's micro structure near the surface and any coatings have a fundamental effect on any operations on the material. Finally, the lubricant is described by its chemical additives and physical properties such as viscosity.

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### 15.1.1
### Processes

Sheet metal forming processes have not only gained considerable importance in the thin sheet metal processing sector but significant progress has also been made in materials which in earlier times could not be processed by forming. Especially noteworthy in this respect is the forming of stainless, austenitic and ferritic, sheet metal which initiated great activity in both the development of lubricants and materials for the tools. While the main objective in sheet metalworking is to produce the maximum possible forming in as few working steps as possible without the production of scrap parts, whether as a result of cracks or surface imperfections, the main focus of tribological development in high tensile steels, titanium and stainless materials lies in reducing tool wear.

During large scale production, for example in the production of parts for vehicle bodies, the greatest expense in development over recent years as far as lubricants are concerned was not for the actual lubrication but for fulfilling new secondary requirements of the lubricant. Advancements in production technologies made it necessary for lubricants to fulfill specific requirements for removability, new application methods, corrosion protection, behavior during sheet metal adhesive bonding, welding, temperature resistance, compatibility with special coatings along with evolving workplace health and safety concerns.

There are so many processes in sheet metal working that these cannot all be covered here. Besides deep- and stress-drawing stamping and fineblanking will be covered in this chapter. Process combinations are frequently used and secondary demands placed on lubricants in forming operations including stamping and fineblanking are often similar.

### 15.1.2
### Basic Terms in Forming Processes

To properly understand the friction and wear in forming, it is essential to become familiar with some of the basic terms used. It is especially necessary to accurately estimate the degree of difficulty of the forming process in order to be thus able to select the correct lubricant.

#### 15.1.2.1 Lattice Structure of Metals

If the shear stress applied to a metallic body achieves a specific level then the molecule layers are displaced in the glide planes determined by the lattice structure of the material. Forming of metals is based on being possible to make this displacement without losing material cohesion, i.e., without the occurrence of material fracture. The higher the pressure normal to the lattice plane the more the material is able to displace without fracture in the glide plane.

The shear stress which causes this displacement with a permanent change in form is called yield point.

### 15.1.2.2 Yield Strength

If plasticity conditions are achieved in a material after applying yield shear stress then the now present material strength is designated yield strength $k_1$ (flow stress). The yield strength can be determined in the laboratory, through compression and tension tests (cone compression test) as well as, with certain restrictions, tensile tests. Knowledge of the yield strength, and particularly its dependence on the level of the forming, temperature and the forming speed are of great importance for being able to estimate the degree of difficulty of a forming operation. As a result of high yield strength values, high forming forces and high surface pressures with difficult friction conditions occur during forming. These same conditions may also favor the prevention of cracks in the workpiece.

The yield strength increases in carbon steels in that the carbon content increases and in steels in general as much as the alloy concentration increase. The latter is also true for copper and aluminum alloys.

### 15.1.2.3 Strain

To define strain, reference is made to the law of constant volume. Here the interrelationships can be illustrated by using the exemple of a compressed rectangular body with initial dimensions $h_0$, $b_0$, and $l_0$. Through compression the height of the body $h_0$ changes to $h_1$. The following is valid:

$$h_0 \times b_0 \times l_0 = h_1 \times b_1 \times l_1$$

$$h_1/\mathrm{h}_0 \times b_1/b_0 \times l_1/l_0 = 1$$

or

$$\ln h_1/\mathrm{h}_0 + \ln b_1/b_0 + \ln l_1/l_0 = 0$$

The logarithmic addends in this last equation are termed logarithmic strain or $\phi_1$, $\phi_2$, and $\phi_3$. Therefore the following is applicable:

$$\phi_1 + \phi_2 + \phi_3 = 0$$

or

$$\phi_1 = -(\phi_2 + \phi_3)$$

### 15.1.2.4 Flow Curve

In a forming operation, the material hardness increases in that the strain increases. If a material is formed so that different degrees of strain are achieved and then the values for the flow stress, which are to be determined for every degree of strain, are spread over the whole strain $\phi$, then one gets the flow curve of a material (flow stress versus strain). In tensile testing, the flow stress is similar to the strain strength values determined in other tests. Figure 15.1 shows the flow curves of some materials up to strain $\phi = 1.0$. This also shows that less than half of the forming work has to be performed to form the copper material listed here than is the case with steel C35.

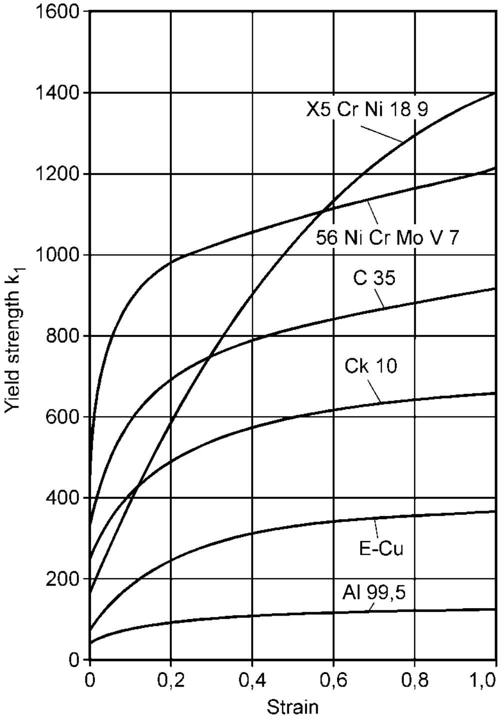

**Fig. 15.1** Flow curves for some metals up to strain $\phi = 1.0$.

For exact understanding and evaluation of flow curves for forming it is essential to know the annealing conditions, grain structure, strength properties and the test conditions.

The most important mathematical interpretation of the flow curve is the exponential equation, which, in its simplest form, is approximately correct for a variety of applications:

$$k_1 = a \times \phi^n$$

where $a$ is a material-specific factor and $n$ a hardening exponent. Using logarithms the equation changes to:

$$\log k_1 = \log a + n \log \phi$$

If one assumes that the forming area material falls in line with this equation then, using a double logarithmic calculation, $a$ is the ordinate value when $\phi = 1$ and $n$ the increase in the straight line.

The hardness exponent n has considerable significance in the description of forming operation.

High $n$ values lead to high surface pressures during the forming operation. The achievable degree of forming normally declines with the n value during deep drawing ($\beta_{0\text{max}}$, Section 15.1.3) and increases during stretch forming (Erichsen test).

#### 15.1.2.5 **Efficiency of Deformation, Resistance to Forming, Surface Pressure**

The forming force to be applied for a forming operation is given from the product of stressed surface $A$ and the yield strength $k_1$; in this case one also talks about the ideal forming force:

$$F_{\text{id}} = A \times k_1$$

However, if one also considers the additional frictional force and other losses, where applicable, the following applies:

$$F = A \times k_{\text{w}}$$

where $F$ is the total forming force and $k_{\text{w}}$ the resistance to forming. $k_{\text{w}}$ is larger than $k_1$ whereby their relationship is found in the formula

$$\eta_{\text{F}} = k_1 / k_{\text{w}}$$

where $\eta_{\text{F}}$ is the efficiency of deformation.

This relationship is particularly important for friction and lubrication because in many forming processes the plastic deformation surface pressure between the material and the tool roughly equals the resistance to forming and can therefore be much larger than the yield strength. This means, for example, that for thinner sheets and a corresponding reduction of the forming efficiency during a drawing operation, difficult friction conditions can arise (see Fig. 15.6).

#### 15.1.2.6 **Strain Rate**

Strain rate is defined as $\mathrm{d}\phi/\mathrm{d}t$ or $\phi$, is measured in $s^{-1}$, and can be calculated from the forming velocity. Its influence on the yield strength in sheet metal working and many other processes is relatively low. In fast turning roller frames and during wet wire drawing, with yield speeds of $10^3$ to $10^4$ $\mathrm{s}^{-1}$, the yield strength can be 50 % higher than the value calculated from the flow curve. In warm forming processes the influence of the strain rate on the yield strength can be much larger than in cold forming processes.

More important than the influence on the yield strength, though, is the material heating which takes place because of the forming and heat from friction generated at high forming speeds. This can have a considerable influence on the reaction of polar lubricant additives and EP additives under boundary lubrication conditions. On top of this, even small temperature changes at low temperatures, e.g. well below 100 ° C, bringing about the melting of lubricant compounds (e.g. waxes) can have a considerable effect on the lubrication process.

#### 15.1.2.7 **Anisotropy, Texture, *R* value**

Above all in the area of sheet metal forming, especially in deep drawing, different material properties working in different directions (anisotropy) can have a significant effect on the degree of difficulty of the forming operation. Textures and orientation of the crystal structure, e.g. to the rolling direction of the sheet metal, lead to this anisotropic directional behavior. In deep drawing the result can be wrinkle formation and earing. The directional variations in thickness are of particular importance for the surface pressures and the lubrication.

The $R$ value has been created to denote this deviation in thickness. This is determined in a tensile test on a sheet metal specimen and states whether an abnormal reduction in thickness occurs during stressing with an approx. 20 % uniform elongation.

The following applies:

$$R = (\ln b_0/b)/(\ln d_0/d) = \phi_b/\phi_d$$

where $b_0$ is the initial width and $d_0$ the initial thickness of the sheet metal. $d$ and $b$ are the end values after tensile stress. Depending on the rolling direction the $R$ value, for example for St-14 sheet metal, can fluctuate between 1.8 and 1.3. Essential for the forming operation is not only the absolute size of the $R$ value in one direction (for example, in the longitudinal direction) but the differences $\Delta R$ in the sheet plane at various angles to the rolling direction (planar anisotropy). Although high $\Delta R$ values are unfavorable for material flow, contributing to earing and friction in the production of cylindrical hollow bodies, they can have a favorable effect on forming square hollow bodies. This effect is used specifically for the production of square hollow bodies from stainless sheet metals.

## 15.1.3
## Deep drawing

During deep drawing, hollow bodies are produced from metal blanks using punches, dies and blankholders. In no other forming operation are the friction and, as a result, the lubricating conditions so complex. In one drawing operation example described in detail here, a particularly low coefficient of friction is required in one area and a particularly high coefficient of friction in another.

The friction conditions when drawing can be best explained by means of the example of drawing a cylindrical cup. Figure 15.2 shows the different friction areas and generally illustrates the deep drawing operation. Here the sheet metal blank, in this case a shaped blank (circular shaped sheet metal cut-out) is pressed on the die by the blankholder with a blank holder force $F_N$. The punch starts the forming operation with the total drawing force $F_{\text{ges}}$. Friction occurs between the sheet metal and the blankholder and the sheet metal and the die (area A), between the sheet metal and the die radius (area B) and between the sheet metal and punch edges (area C).

### 15.1.3.1 Friction and Lubrication in the Different Areas of a Deep Drawing Operation

**Punch Edge (Friction Area C in Fig. 15.2)**

The drawing force in the flange necessary to form the sheet metal is applied by the punch in the base of the cup and transferred from here through the wall into the flange. This transmission of force calls for the highest possible coefficient of friction on the punch edge. This demonstrates the first significant rule for lubrication in this case: neither the punch nor the sheet metal blank should be lubricated in this area. Even if this consideration was optimal for an isolated forming operation and

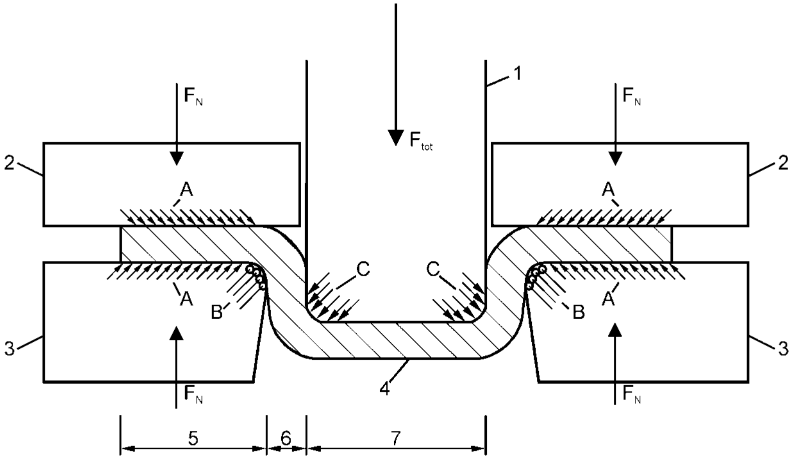

**Fig. 15.2** Friction areas when deep drawing a cup. A, friction area between sheet metal blank and holder and sheet metal blank die; B, friction area between sheet metal blank and the die radius; C, friction area between sheet metal blank and the punch edges; $F_{\text{ges},}$ total drawing force; $F_{N,}$ blank holder force; 1, punch; 2, blank holder; 3, die; 4, cup; 5, flange area; 6, cup wall; 7, base of the cup.

force transfer one still must consider punch wear. This friction area requires lubrication in the boundary mode with a high coefficient of friction and anti-wear behavior. This is possible with low viscosity drawing oils, polar substances or EP additives. Moreover, in this case the thickness of the lubricant film must not be excessive. It should be somewhere in the range of the sheet metal roughness $R_a$. For forming sheet metal for car bodies using prelubes applied in the rolling mill, an oil layer thickness of 1 to 2.5 μm is generally adequate for lubricating the sheet metal on the punch side. However, when forming high tensile materials and stainless austenitic and ferritic materials, considerably greater attention must be paid to lubrication. High activity EP oils are often used in this area in order to reduce wear. A high coefficient of friction along with anti-wear performance can also be achieved in some cases by lubricants with a high filler content. These filling materials or solid lubricants should be blunt in structure but not molybdenum disulfide or graphite.

Since the best possible lubrication and lowest coefficient of friction possible are required on the punch in the blankholder area of the sheet metal flange, a compromise frequently has to be made concerning the lubrication. It is often not practicable to lubricate one side of the sheet differently from the other, especially with small drawn-parts. The roughening of the punch may be done to selectively increase the coefficient of friction for better force transfer while uniformly oiling the sheet metal blank. This may also be done by using sheet metals which have been dressed with a higher roughness on one side in the rolling mill to achieve a higher coefficient of fiction at the punch.

The cup bottom and the connection to the wall have the least yield during deep drawing. The yield increases with frame height and, as a consequence, also the yield strength. If the drawing force transferred from the base of the cup is too high the sheet metal will tear at the point with the lowest strength resulting in bottom tearing, the most significant and most difficult deep drawing fault to be analyzed [15.1]. Here, the lubricant has the important task of reducing the considerable frictional component of the total drawing force in the region of the blankholder and the die radius.

The influence of punch edge friction on the transfer of force is also clearly dependent on the punch edge radius $r_s$ or the ratio $r_s/d_0$ whereby $d_0$ is the diameter of the punch. Particularly, with a high $r_s/d_0$ ratio, a reduction of the coefficient of fiction results in a reduction of the so-called bottom tearing force [15.2].

### Holder and Die Radius (Friction Areas A and B in Fig. 15.2)

While the above dealt with the friction conditions in the force transfer area, the friction situation in the deformation area is explained by studying the friction in the region of the blankholder and the die radius.

As a result of the tangential pressure stresses and radial tensile stresses in the flange, there is a tendency towards material compression on the outer rim. Because of this thickening of the sheet metal the blankholder can only transfer the blankholder force to a limited section of the sheet surface on the blankholder side, which leads to high surface pressures and difficult friction conditions. As a result of the lack of contact by the blankholder in the other areas, there is also the risk of fold or wrinkle formation. This wrinkling also creates special friction conditions since the backs of the wrinkles cause more or less line contact with the blankholder, producing local areas of high surface pressure.

Figure 15.3 shows an example of such thickening of the sheet on the rim of the cup flange. Two areas, area 1 (with holder contact) and area 2 (without blankholder contact) develop in the region of the flange. These situations arise when deep drawing is carried out without lubricants or with solid lubricants. Increasing the blankholder force reduces contact free area 2 and as a result the development of wrinkles is also reduced. However, the blankholder force can only be altered over a relatively narrow range.

Figure 15.4 shows the lubricated cup drawing when a drawing oil or drawing compound is used. Here a hydrostatic supporting lubricant bed develops on the die

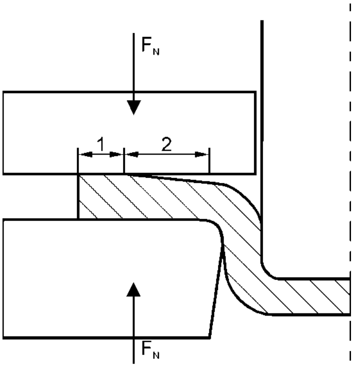

**Fig. 15.3** Tribological conditions in the flange area when deep drawing without lubricant or using solid lubricant. 1, flange area with holder contact; 2, flange area without holder contact.

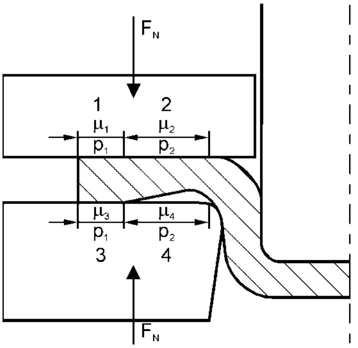

**Fig. 15.4** Tribological conditions in the flange area with lubricated deep drawing and the build up of a hydrostatic supporting lubricant bed [15.3] (subdividing the friction area into areas 1, 2, 3 and 4).

which presses the sheet metal against the blankholder and as a result counteracts wrinkling. Figure 15.4 also shows four friction areas with four different coefficients of friction and 2 different pressure values in the flange area. The coefficient of friction $\mu_4$ (contact between lubricant bed and die) is 0. By experimentally measuring of the surface areas in which $p_1$ and $p_2$ act, determination of the surface pressures $p_1$ and $p_2$ and the coefficients of friction $\mu_1$, $\mu_2$, and $\mu_3$ (for example, in a strip drawing test) it is possible to determine the total friction in the region of the flange.

The main aim of deep drawing lubrication is to achieve minimum friction in the blankholder area $F_{RN}$. This includes the lubrication on the blankholder side, which, as much as possible, should be a lubricant ring in order to reduce the friction in the region of the punch edge as little as possible. However, in the case of high viscosity oils and drawing pastes, if the lubricant film applied is excessive, there is the risk that hydrostatic effects on the blankholder side of the flange can cause reduction of sheet metal contact with the blankholder resulting in wrinkling.

The friction conditions on the die are less complex than in the blankholder area. Here the surface pressure can be relatively accurately estimated and the coefficient of friction determined in deflection strip drawing tests. In this case the aim of lubrication is to achieve a minimum coefficient of friction with minimum wear. Even excessive lubrication of the sheet metal surface on the die side normally does not create a problem. Only on parts with large surface areas and a high percentage of stretch drawing, such as car body parts (Section 5.3) can excessive lubricant quantities lead to unacceptable deviations in form.

The geometrical design of the die has a significant influence on friction. Whereas a quarter circle rounding (dependent on $D_0 - d_0$) is generally used in the initial drawing operation, more complicated conical geometries are also applied in subsequent drawing operations. Higher surface pressures are necessary when smaller radii are employed which leads to lower coefficients of friction in the strip drawing test with deflection. Here it is obvious that there are limits to minimizing the coefficient of friction by using very small die radii. It has been demonstrated that under identical lubricating conditions the total drawing force was higher when using small die radii [15.4]. If very small die radii are necessary for technical reasons, EP lubricants have proven to have a positive influence on the workpiece surface quality as well as die wear in spite of the high surface pressures occuring. However, this does not seem to bring about a reduction of the drawing force.

### 15.1.3.2 Significance of Lubrication Dependent upon Sheet Metal Thickness, Drawn-part Size and the Efficiency of Deformation

According to Siebel and Panknin the total forming force when deep drawing $F_{ges}$ is divided into the actual forming force $F_U$ (ideal forming force + rebounding force) and friction $F_R$ (mainly the friction in the holder area $F_{RN}$ and the friction on the die rim $F_{RZ}$), i.e.:

$$F_{ges} = F_U + F_R$$

If $F_U$ and $F_R$ are calculated at the maximum drawing force [15.5] and these values are applied dependent on the punch diameter, then with a sheet metal thickness $s_0 = 1$ mm and the material St 14.03 the curves as shown in Fig. 15.5 result [15.6]. The illustration impressively shows the considerable increase in friction and thus also its significance for lubrication over the diameter of the punch, i.e. the size of the drawn-part. With a punch diameter $d_0 = 600$ mm the friction force and forming force are equal. When $d_0 = 1200$ mm the friction force will be approximately three times higher than the forming force.

Figure 15.6 [15.7] shows the change in the efficiency of deformation dependent on the ratio between punch diameter $d_0$ and sheet metal thickness $s_0$ for different coefficients of friction. This calculation was done using an average coefficient of friction. For a constant punch diameter $d_0$ the illustration shows the dependence of the efficiency of deformation on the thickness of the sheet metal. It becomes very clear that low efficiency of deformation is given with thin sheet metal (large $d_0/s_0$ ratios). Here, the forming efficiency is determined to a considerable extent by the proportion of the frictional component of the drawing force (Section 5.1). The influence of the coefficient of friction is critical here. In Fig. 15.6 this can be shown by means of the following example. If a coefficient of friction of 0.2 is given in a forming opera-

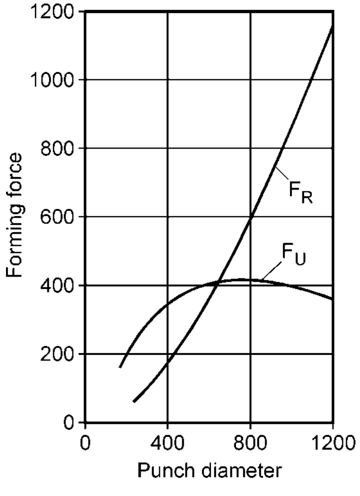

**Fig. 15.5** Dependence of the friction share of the drawing force on the punch diameter. $F_R$ = friction; $F_U$ = forming force; $F_{ges} = F_U + F_R$.

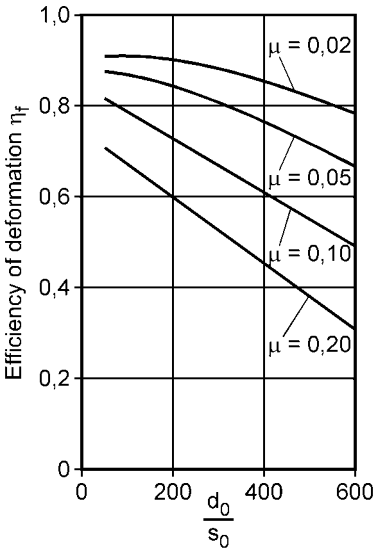

**Fig. 15.6** Influence of friction on the efficiency of deformation, dependent on the ratio between punch diameter and sheet thickness; with a constant punch diameter $d_0$ the curves show dependence on sheet thickness [15.7].

tion where $d_0/s_0 = 200$, which corresponds to a relatively poor lubrication, then the forming efficiency $\eta_F$ is 0.6. If one now wants to obtain the same value when $d_0/s_0 = 400$ then the coefficient of friction must be reduced to 0.1 by considerably improving the lubrication.

This means that, with the $d_0/s_0$ ratio one has a further reference for defining the degree of difficulty when deep drawing as an aid to lubricant selection. As a result one could say that deep drawing operations with $d_0/s_0$ values from 400 to 600 are difficult and values above this are very difficult.

Figure 15.7 shows the proportions of the friction forces $F_{RN}$ and $F_{RZ}$ of the total drawing force at maximum drawing force in cup drawing [15.4], where $d_0/s_0 = 200$

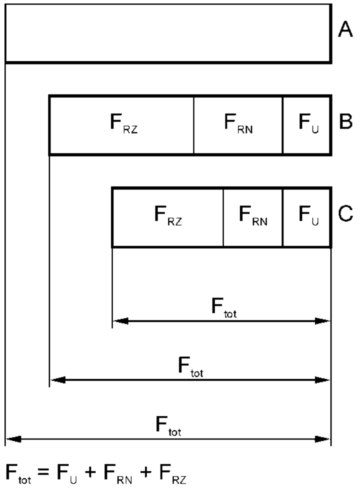

**Fig. 15.7** Friction shares of the total drawing force (maximum drawing force) for different lubrication in cup drawing [15.4]. A, without lubrication; B, low viscous oil–approx. 5 mm$^2$ s$^{-1}$ at 20 °C; C high viscosity oils and drawing pastes. $F_{ges} = F_U + F_{RN} + F_{RZ}$; $F_{ges}$ = total drawing force; $F_U$ = forming force; $F_{RN}$ = friction force in the holder area; $F_{RZ}$ = friction force on the die radius

and the drawing ratio $\beta_0 = 1.8$. A is the maximum drawing force without lubrication. B shows the result with an extremely low viscosity oil, 5 $mm^2\ s^{-1}$ at 20 °C and C is the average result with high viscosity oils and drawing compounds.

### 15.1.3.3 Assessment of the Suitability of Lubricants for Deep Drawing

Most laboratory methods used today for the assessment of lubricants were not developed for lubricant testing but rather for testing the deep drawing properties of materials and the technical process parameters. The most important testing procedure today is still the practical shop trial by which means it is still generally possible to carry out a lubricant test at a reasonable cost. Some laboratory testing methods allow conclusions to be drawn on the deep drawing lubricating performance of lubricants for successful deep drawing, without workpiece fracture and producing an adequate workpiece surface finish quality.

The close-to-practice cup drawing test, for which there are several versions of laboratory devices available, still has great significance among laboratory testing methods. The maximum drawing force and the achievable drawing ratio are particularly important results from the assessment.

#### Maximum Drawing Force

A force–travel diagram can be recorded for the punch force using force measuring instruments which show the maximum drawing force.

#### Maximum Drawing Ratio, $\beta_{0max}$

Details of maximum change in form when deep drawing. This frequently follows from the boundary drawing ratio $\beta_{0\mathrm{max}}$ (as the maximum drawing ratio which can be achieved in the first drawing operation). The drawing ratio is the quotient of shaped blank diameter $D_0$ and punch diameter $d_0$:

$$\beta_0 = D_0/d_0$$

The maximum drawing ratio $\beta_{0\mathrm{max}}$ is reached when the punch diameter is increased to such an extent that, with a constant shaped blank diameter, fracturing occurs. However, it is easier to use a constant punch diameter and vary the diameter of the shaped blank to determine $\beta_{0\mathrm{max}}$. In practical operations the admissible drawing ratio $\beta_{0\mathrm{zul}}$ must be slightly smaller *than* $\beta_{0\mathrm{max}}$. For deep drawing sheet metal (deep drawing quality) $\beta_{0\mathrm{zul}}$ is approx. 1.6 for high $d_0/s_0$ values and 2.3 for smaller $d_0/s_0$ values, so that a change due to the lubricant of ± 0.2 is significant.

The most effective lubricant should result in the highest drawing ratio and the lowest maximum drawing force. Because of the complexity of the friction ratio when deep drawing described earlier there are often exceptions to this statement. This is to some extent understandable, particularly with uniform lubricant coating on both sides.

#### Strip Drawing Test (Fig. 15.8)

A sheet metal strip is drawn between two flat drawing jaws to which a normal force $F_n$ is applied. The strip coefficient of friction is:

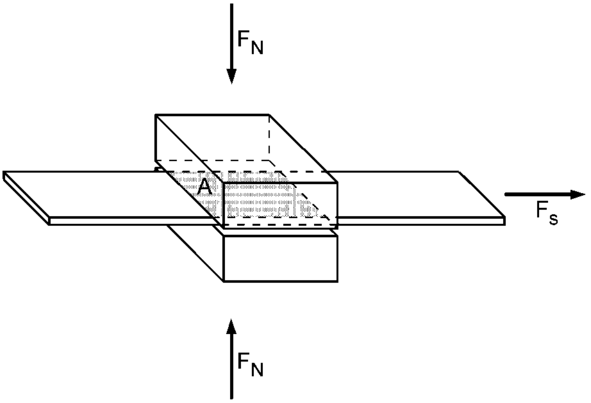

**Fig. 15.8** Strip drawing tester to assess lubricants. $F_n$ = normal force; $F_S$ = strip drawing force; A = stressed surface.

$$\mu_S = F_S/2F_n = F_S/2pA$$

where $A$ is the stressed surface and $p$ the surface pressure. By using different lubricant coatings it is possible to assess a lubricant according to the strip coefficient of friction. But one must take into account that only the conditions in the holder can be simulated. However, these values must be applied with caution and should only be considered relative to the analysis of the different friction areas with varying surface pressures in the region of the blankholder.

#### Deformation Analysis

If the sheet metal blank is marked with a measuring grid comprised of circles prior to a forming operation, then after forming it is possible to determine how the sheet metal has changed. The circles generally deform to produce ellipses. By measuring the axes it is possible to compare the ellipses with the original circles and define the metal deformation. Apart from observing the influence of the material, the tool and possibly even a few process parameters, one is also able to compare lubricants. One can also observe at a critical point how the yield behaves when the lubricant is changed.

### 15.1.4
### Stretch Drawing and a Combination of Stretch and Deep Drawing

Pure stretch drawing takes place during the Erichsen test mentioned in Section 15.1.1. The blank sheet metal clamped firmly on both sides is stretched by tensile stress, generally by means of an upwards traveling stretch drawing punch (Fig. 15.9). Here the thickness of the sheet metal is reduced unlike during deep drawing where the thickness of the sheet metal remains mostly unchanged. The goal is uniform deformation which is favored by the lowest possible friction over the entire stretch area. The reduction in sheet metal thickness leads to a considerable increase in surface area which has to be taken into consideration when choosing the type and amount of lubricant to be applied. On the other hand, when deep drawing the surface of the shaped blank is about the same area as the surface of the drawn-part.

### 15.1.5
### Shear Cutting

According to DIN 8580 shear cutting is a severing process in the separating group. Whereas stamping is a relatively old method of sheet metal working, fineblanking has gained particular significance in the last two decades. Although no particular distinction is made between the two methods in the above-mentioned DIN there are considerable differences in practice. Lubrication has a greater significance in fineblanking than in

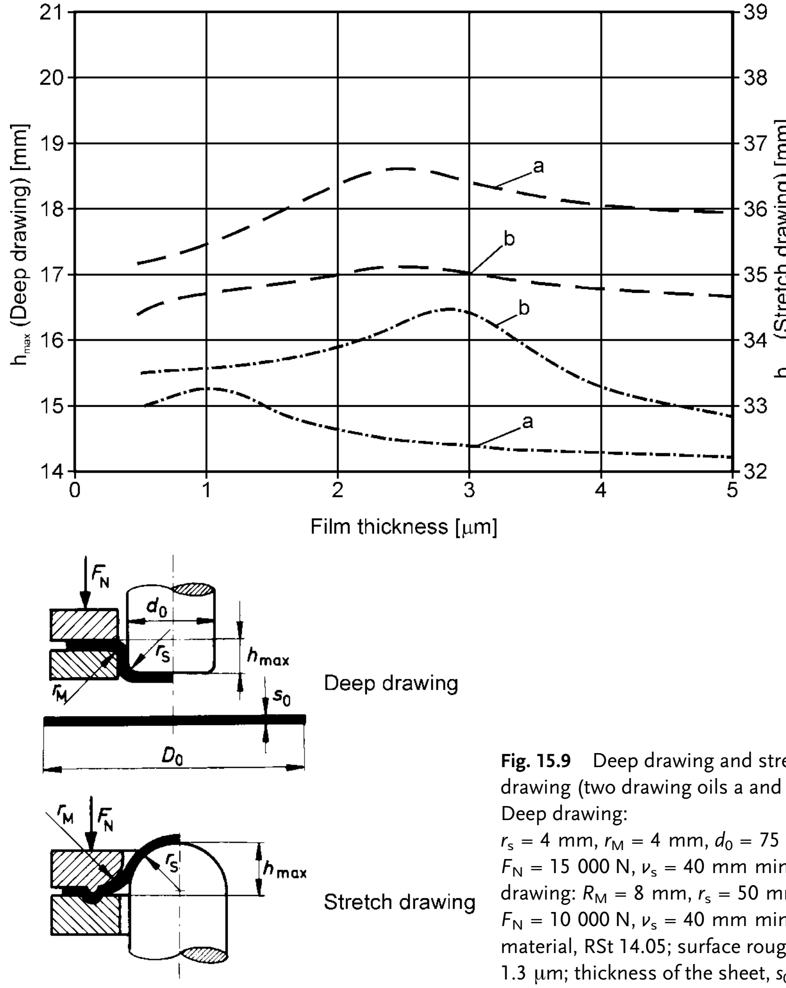

**Fig. 15.9** Deep drawing and stretch drawing (two drawing oils a and b) [15.34]. Deep drawing: $r_s = 4$ mm, $r_M = 4$ mm, $d_0 = 75$ mm, $F_N = 15\,000$ N, $v_s = 40$ mm $\text{min}^{-1}$. Stretch drawing: $R_M = 8$ mm, $r_s = 50$ mm, $F_N = 10\,000$ N, $v_s = 40$ mm $\text{min}^{-1}$. Sheet material, RSt 14.05; surface roughness, $R_a$ 1.3 μm; thickness of the sheet, $s_0$, 1.0 mm.

normal stamping as far as the quality of the workpiece and tool wear are concerned. This normally requires higher performing lubricants. At the same time, lubrication must be given considerable attention in all blanking methods. With difficult to blank materials the significance of the lubricant increases progressively becoming an important component in the operation. Normal or fineblanking parts are normally produced in one single operation. If additional coining, deep drawing or bending operations are necessary, the blanking operation is incorporated into a chain of processes, which can also influence the selection of the lubricant.

#### 15.1.5.1 **Stamping**

A significant characterization of stamping is that one third of the sheet metal thickness is a sheared edge and about two thirds a torn zone. An important stamping method is, for example, making holes for the production of pierced plate for protective grids and for many decorative applications or for the production of screens and filters.

As we can see in Fig. 15.10, the sheared area of stamped parts is subdivided into three areas; the forming area, the cut area and torn area.

If the piercing operation is considered from the lubrication viewpoint, and we assume that a uniformly oiled sheet is present to improve the friction and wear conditions, then the sequence of events will be approximately as follows (Fig. 15.11). The punch moves towards the sheet (a in Fig. 15.11), comes up against the oil and forces the oil radially outwards where it produces a seam of oil which is a lubricant reservoir essential for the further stamping process. An oil film remains between the punch and the surface of the metal sheet. In the forming phase, friction develops between the punch and workpiece close to the cutting edge whereby the punch nose surface area, punch edge and even the punch stem surface are stressed by friction. The influence of the lubricant upon the stamping force reduction is insignificant. The reduction in punch wear, especially punch surface wear is more important. After completion of the cutting phase the scrap breaks away from the material and drops out of the hole. The sheet metal, which is also elastically deformed, springs back radially towards the punch jacket surface and generates a significant surface pressure in the region of the cutting area (c in Fig. 15.11). With the continued downward movement of the punch the lubricant will be transported into the hole and it wets the jacket surface of the punch.

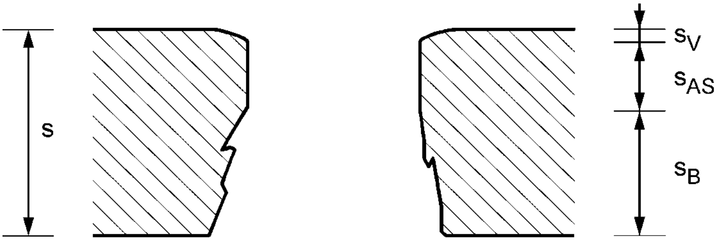

**Fig. 15.10** Breakdown of the sheared zone in stamping.
$s$ = sheet thickness, $s_V$ = deformation area, $s_{AS}$ = initial cut area, $S_B$ = torn area.

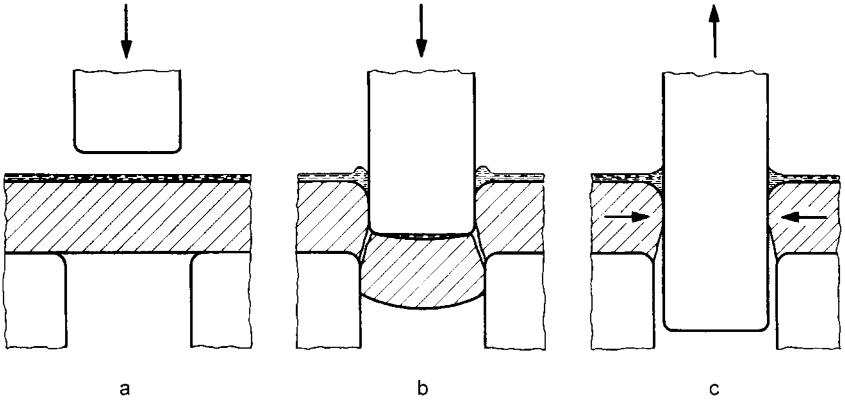

**Fig. 15.11** Stamping. (a) Sheet with uniform film of lubricant before the blanking operation; (b) Stamping operation with the development of a seam of lubricant as lubricant reservoir in the intake area; (c) Retraction with high friction on the punch surface.

This process is critical for lubrication during punch retraction. Since the retraction force is mainly friction, the lubricant becomes particularly significant with regard to reducing the force. Since reducing the return force has been proved in practice to lead to a reduction in wear (punch jacket wear), the measured force when the punch travels backwards is used to assess the lubricant.

Whereas the quality of the workpiece is generally in the foreground for selecting the lubricant for other sheet metal working processes, tool wear is generally the most important factor in stamping lubricant evaluation. Tool wear is also the most significant cost-determining factor. However, very closely associated with the wear, especially punch jacket wear, is the burr height of the workpiece. As far as many stamping processes are concerned, this is the decisive criterion for defining tool life or the number of products produced with the tool. Therefore one also tries to define the cuttability of a material by the number of products produced with the tool. The following definition applies:

$$\mathit{Cuttability} = (\mathit{Number\ of\ products})/(\mathit{Abrasion\ height})\ (\mathrm{mm}^{-1})$$

A change in burr height of 30 μm is set for cutting electrical steel sheet to determine the volume of products produced with a tool [15.8]. As tool wear enters this equation directly through the abrasion height of the tool this can also be used as a criterion for assessing the lubricant, under otherwise constant conditions. In this case the abrasion height can refer to both jacket wear on the punch as well as on the stamping die.

### Lubricants

In many factories efforts are being made to reduce the number of lubricants used for stamping. This reduction in types of lubricant also makes it necessary for the plants to select lubricants based on the most difficult operations, materials and sheet metal thicknesses. Today the majority of companies try to make due with a maximum of three lubricants in the production of stamped parts by combining blanking and forming operations.

Lubrication classification for stamping leads to 6 sheet metal material groups. However, this can only be viewed as a rough sub-division since, particularly in the case of carbon steels, heat treatment has a strong influence on ease of blanking. On top of this, the sheet metals are divided into four thickness categories. The lubricants are divided into 7 or 9 groups and can be described as follows:

- Group 1: oils, 50 to 180 $mm^2\ s^{-1}$, 40 °C; the oils are either completely or to a considerable extent active substances. Used as substances are extreme pressure materials (containing chlorine, sulfur and phosphorous), polar substances such as synthetic esters, natural fatty oils or fatty oil derivatives. Where EP stands for extreme pressure additives and P for polar substances then group 1 is designated EP+++P+++.
- Group 2: oils, 30 to 110 $mm^2\ s^{-1}$, 40 °C; designation EP++P++.
- Group 3: oils, 30 to 110 $mm^2\ s^{-1}$, 40 °C; the oils are either completely or to a considerable extent polar substances, designation P+++.
- Group 4: oils, 30 to 110 $mm^2\ s^{-1}$, 40 °C; apart from considerable proportions of polar substances the oils contain lower concentrations of EP substances, designation EP+P+++.
- Group 5a: oils, 15 to 110 $mm^2\ s^{-1}$, 40 °C; the oils are mixtures of polar substances with mineral oil hydrocarbons, designation P++.
- Group 5b: oil, < 15 $mm^2\ s^{-1}$, 40 °C; these oils are mixtures of polar substances with mineral oil hydrocarbons, designation P+.
- Group 6a: water miscible compounds. Mainly, these are products with a grease consistency, possibly also water miscible oils with considerable concentrations of polar substances and medium to low EP levels, designation EP+P+++. The products are used as undiluted pastes or diluted as lean as 25 %. They are frequently diluted to a ratio of 1:3.
- Group 6b: water miscible compounds. These are simple products without specific combinations of active substances. The products are used undiluted to 15 % but are frequently diluted with water at a ratio of 1:5.
- Group 7: corrosion protection oil. This is a surface protection oil which is already applied by sheet metal producers and is also to serve as a lubricant for further processing (mill oils, slushing oils, prelubes)

Most lubricants in Groups 1 to 5 may be diluted with petroleum hydrocarbon solvents. The solvent serves mainly to facilitate application. If necessary, oils can be applied which have a considerably higher viscosity than those in the first 5 groups. However, these are specific applications which will not be discussed in further detail here. The most important lubricants in the table and their areas of application have been marked in Table 15.1, i.e. the difficult to cut austenitic materials with a sheet thickness up to 1.5 mm require a lubricant from Group 2 and lubricants for thicker sheet metals are in Group 1. Ferritic CrNi sheet metals require lubricants in Group 2 and for thicknesses in excess of 8 mm lubricants in Group 1 are selected. Depending upon thickness, the lubricants in the Groups 4, 2 or 1 are required for carbon steels. Lubricants in Group 5b are suitable for electrical quality sheet whereas the lubricants in Groups 3 to 5b are used for aluminum, non-ferritic metals and their alloys.

**Tab. 15.1** Classification of the lubricant groups for normal blanking by areas of application.

| *Sheet metal thickness* | *Materials* | | | | | |
|---|---|---|---|---|---|---|
| | *Austenitic CrNi-sheet metal > 600 N mm$^{-2}$* | *Ferritic CrNi-sheet metal ~ 450 N mm$^{-2}$* | *Carbon steels* | *Electro sheet metal > 400 N mm$^{-2}$* | *Al + Al alloy* | *Copper + copper alloy* |
| 0.1 to 1.5 mm | 1, 2, 5a, 6a | 1, 2, 5a, 6a | 2, 4, 6a, 6b, 7 | 5a, 5b, 6b, 7 | 3, 4, 5b, 6a | 4, 5a, 6b |
| 1.6 to 3.5 mm | 1, 2 | 1, 2 | 1, 2, 4, 5a | – | 3, 4, 5a | 3, 4, 5a |
| 3.6 to 8.0 mm | 1 | 1, 2 | 1, 2, 4 | – | 3, 4 | 3, 4 |
| > 8.0 mm | 1 | 1 | 1, 2 | – | 3, 4 | – |

A particularly important aspect in addition to the reactivity of the additives (polarity and the effective mechanism of EP components), is the surface phenomena. On the lubricant side, consistency, fast wetting and, as a result, the effective transport to the sheet-tool interface are of particular significance. But also punch and plate surface criteria have to be considered in the generation of effective lubrication films.

#### 15.1.5.2 **Fineblanking**

The most significant difference between a part produced by stamping and a part produced by fineblanking is in the quality of the cut surface, which in many applications serves as gearwheels. Significantly higher shape accuracy is achieved by, among other things, a very small die clearance, which in fineblanking is typically only approx. 0.5 % of the sheet thickness. In stamping this clearance is approx. 10 to 15 % of the sheet thickness. The cutting surfaces have no fracture area and the workpieces have only an insignificant burr.

When fineblanking, unlike stamping, the sheet is clamped on all sides by a vee-ring. The specific tool structure is significant for lubrication. Lubricating chambers are formed by special chamfering on parts of the tool [15.9]. From these lubricating chambers lubricant can even penetrate the die clearance during the operation. Figure 15.12 shows a fineblanking tool with lubricating chambers formed by the above-mentioned chamfering; the black areas are the patches of lubricant. [15.10, 15.11].

The great significance of the quality of the cut surface when fineblanking has led to the surface roughness to be used as an assessment criterion for fineblanking lubricants. Increased surface roughness is an indication of increased tool wear, which in turn leads to increased burring.

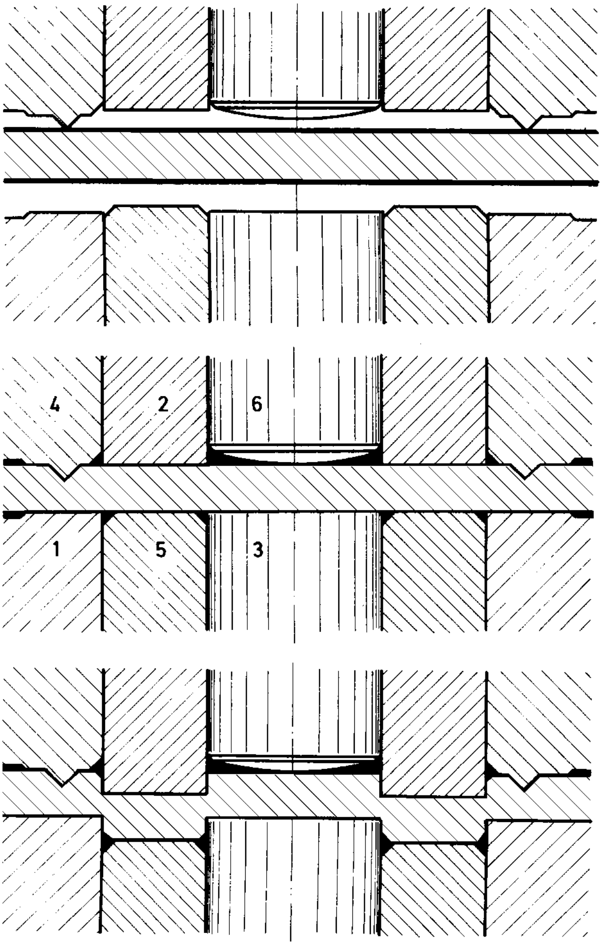

**Fig. 15.12** Constructive measures to improve lubrication for fineblanking (according to F. Birzer [15.9]; the black patches indicate the lubricant film and the accumulation of lubricants. 1, blanking die; 2, cutting punch; 3, inner form punch; 4, pressure plate (guide) with vee-ring (ring hob); 5, injector; 6, thrower; 7, sheet.

### Lubricants

Similar to stamping, Table 15.1 also applies as a rough classification of lubricants [15.12]. However, in general, oils with higher concentrations of additives and oils with reactive EP substances are used because there is a strong tendency towards adhesive wear when fineblanking difficult-to-cut, thick materials with unfavorable structure. Oils with a high chlorine content are frequently used for difficult fineblanking operations [15.9]. However, it must be remembered that not only the amount of chlorine but also the reactivity of the chlorine compound plays a role.

Working with reactive EP substances, especially chlorine compounds, can lead to considerable corrosion problems on fineblanked parts, particularly on the cut surface. Therefore the aim behind formulating a fineblanking oil lies in control of corrosion with adequate reactive substances. Because chlorine compounds cause environmental problems there is a clear trend towards chlorine-free products. The exten-

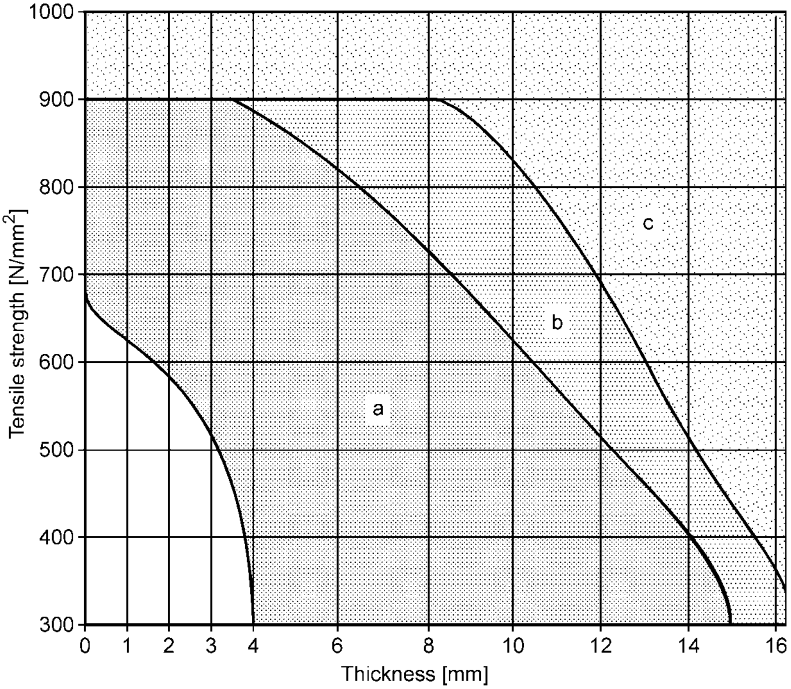

**Fig. 15.13** Application chart of high-efficiency lubricants according to the Feintool specification. (a) main application range; (b) extension of range by use of coatings; (c) application conditionally possible.

sive use is of chlorine-free products is already well established in the area of low and medium thickness sheet metal.

The wetting ability of fineblanking oils also deserves particular attention. This is important when actually blanking but also when applying the lubricant. It is frequently necessary to apply relatively high viscosity oils in a very thin layer. Spraying fineblanking oils is a preferred method of application compared to applying oil by rollers. Uneven sheet metal surfaces are not a problem if spraying. However it must be ensured that the sprayed drops of oil form a complete film and completely and uniformly wet the sheet. To define the area of application for different fineblanking oils, diagrams have been produced in which the fields for sheet thickness and material tensile strength are marked. Figure 15.13 shows one example of such a diagram.

## 15.1.6
## Material and Surface Microstructure

### 15.1.6.1 Material

The most common materials in sheet metal forming processes are unalloyed or alloyed steels, light alloys as well as aluminum, copper and titanium and their alloys. A summary of the commonly used sheet metal materials, the technological specifications and their field of application is given in [15.13]. The alloyed steels comprise of phosphor-alloyed steels, bake-hardening steels with additional hardening after heat treatment, ferritic chrome steels, austenitic chrome nickel steels and micro-alloyed steels with a higher tensile strength. To reduce the sheet metal thickness and thus the weight of components, new cold rolled or hot rolled steel grades such as dual phase steels, transformation induced plasticity steels (TRIP steels) and complex phase steels have been developed [15.14, 15.15].

Because unalloyed or low-alloyed steel is subject to corrosion, steel strips are coated to an increasing extent with protective metal or polymer layers which are electrolytically deposited (electro-galvanized) or hot-dip applied. Zinc and its alloys are by far the most important coatings in sheet metal forming. Zinc and zinc–nickel layers are deposited from chlorine or sulfuric electrolytes by galvanic current. Zinc, zinc–iron diffusion layers (galvanneal) or zinc–aluminum coatings are hot-dip applied. To achieve a better lubricating effect, the sheet metal surface is sometimes phosphated. When the phosphate is applied in the galvanizing line, this material is often referred to as pre-phosphated.

The engineering and construction industries generally use hot-dip galvanized steel with a larger crystal patterns which is additionally chromated for improved corrosion resistance. On the other hand, the automobile industry prefers non-passivated, fine-crystalline surfaces.

Coil-coating methods are gaining acceptance. Such coatings often have a decorative as well as a protective character. Common coating materials include acrylates, epoxides and polyesters. These polymer coatings are often applied to electrolytically or hot-dip galvanized sheet steel. They are primarily used in the construction industry as facades but in recent times, also in the automobile industry. Polymer films which contain zinc dust pigments to improve weldability and which are just a few microns thick have proved successful. Many coated materials display better forming characteristics than the uncoated material. In many cases, the limits to formability are not set by the underlying material but by the peeling-off or cracking of the coating [15.16]

#### 15.1.6.2 **Surface Microstructure [15.17–15.19]**

The surface micro structure of deep drawing sheet steel has a major influence on friction during a forming process. It should transport an sufficient quantity of lubricant to the drawing zone and retain it in spite of surface pressure and relative movement of panel and die. Furthermore, the surface should absorb any abrasion created by wear. In order to fulfil these requirements, the surface of the sheet metal is purposely roughened by skin pass or temper rolling which transfers the structure of a roll to the sheet. The microstructure of the roller's surface is created by a variety of methods. With shot blast texturing, the roller is blasted with steel balls of high kinetic energy which roughen the surface. In electrical discharge texturing, high voltage electrical arcing causes small areas on the surface to vaporize. These processes are irregular and random and create a stochastic surface structure. Newer methods such as electron beam texturing (EBT), galvanic precipitation of hard metal semi-spheres (precision texturing, Pretex) or laser texturing (Lasertex) can be used to create defined and regular structures which are known as deterministic structures. Electron beam texturing is a vacuum process in which the slowly rotating roller is subjected to an electron beam pulsated through a rotating perforated mask. The high energy causes localized melting. Raised-lip craters with a diameter of about 100 microns are formed on the surface of the metal. If these craters overlap, apparently random or pseudo-stochastic structures are created. In laser texturing, a laser beam in inert gas is focussed on the surface of the roller at an angle. Here again, the localized molten metal forms a raised-lip crater.

The aim of all the roller roughening methods is to create the largest possible number of lubricant pockets in the sheet metal surface. Under the effect of contact stress during pressing, the surface is partially smoothed out and the indentations are increasingly isolated from each other. The proportion of isolated void volume forming hydrostatic lubricant pockets which separate panel and die is an important factor for the drawing performance of the sheet metal.

Various methods are used to characterize the topography of sheet metal surfaces. Visual analysis and the feel of the sheet allow a very qualitative evaluation of the surface. The reflection of the metal as well as fine scratches can be seen with the naked eye but are very difficult to determine using quantitative methods. It is possible to guess the expected forming behavior of comparable surface types. Quantitative measuring procedures are necessary for a more exact analysis, in particular if different roughening methods are to be compared. The most commonly used is the contact surface scanning method in which the topography of the surface is recorded along a line with a diamond-tipped needle. The signal generated is usually filtered according to a standardized procedure to eliminate geometric deviations caused by waviness, distortion or twisting in the tested panel. The section height thus obtained is recorded locally. From this, parameters such as average roughness, $R_a$, depth of roughness, $R_t$, and the number of peaks, $P_C$, can be derived and these serve to describe the overall topography. If the surface is measured along a number of parallel lines, three-dimensional topographical charts can be drawn. In addition to the mechanical methods of determining roughness, there are also optical systems which scan the surface (e.g. laser scanners).

While two-dimensional contact surface scanning is a suitable method to evaluate stochastic topographies and is a standardized procedure, deterministic structures can only be adequately defined with three dimensional scans. Only this allows the number and the isolation of the lubricant pockets to be recorded, data which is necessary to make a prediction about the behavior of the sheet metal surface during forming.

### 15.1.7
### Tools Used in Sheet Metal Forming Operations

Interacting with the lubricant itself, the properties of the tool materials and the surface morphology play significant roles with regard to friction and wear as well as workpiece quality. Together with the forming speed, the surface morphology is critical for the build-up of hydrodynamic and hydrostatic lubricant films.

Special tool designs, in particular the surface coatings, have facilitated a clear reduction in the spot lubrication. When using PVD or CVD hard solid layers, tradi-

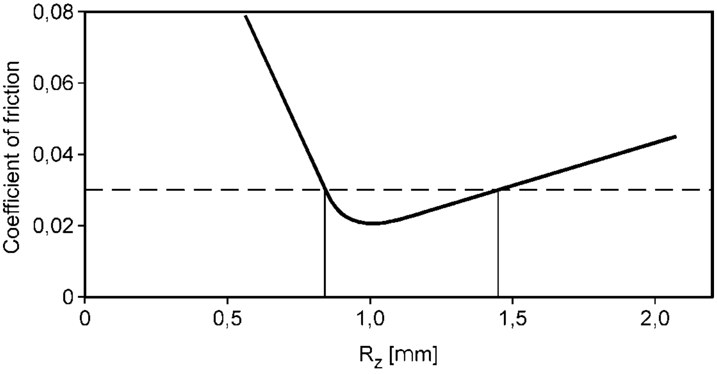

**Fig. 15.14** Dependence of the coefficient of friction on the surface roughness $R_z$ on drawing edges coated with TiC–TiN [15.22].

tional lubricant functions are also transferred into the layer. This applies especially for the lubricants' EP properties.

Today, gray cast iron GG 25 is predominantly used in the production of large-volume tools for sheet metal forming. Surface-hardenable GG 25 CrMo, GGG 60 or GGG 70 are used for the production of large quantities of tools. Apart from the surface hardening, nitriding can also be carried out in the highly stressed surface areas. In specific cases, hard chromium plating methods can also be applied, which, however, require a sufficiently hard substrate in order to ensure a proper layer adhesion. When working under extreme pressure (high plate thickness, high-tensile steels, high drawing ratio), cases made of tool steels are fitted into the gray-cast iron tool (e.g. the materials 1.2379 or 1.2601, hardened and nitrided or hard-solid coated).

Tools with fitted cases cannot be used in the production of parts used in the bodywork of automotive vehicles since they would lead to the formation of visible irregularities in the material surface. Therefore, only monobloc tools are used for high quality surfaces.

Like sheet steels, outer skin parts made of aluminum are drawn from gray cast iron using monobloc tools. In order to prevent adhesion, however, one prefers to use very plain surfaces with lubricant films of > 2 μm. Small tools are chromium plated or coated with PVD (Chromium nitride, TiN). When working with aluminum, PVD layers are preferred to CVD layers.

Figure 15.14 shows the optimal range for the roughness $R_z$ for drawing edges coated with TiC–TiN. A further optimization for the formation of a lubricant layer is brought about by the generation of oxide boundary layers ($TiO_N$–$TiO_2$) [15.22].

The formation of a special surface topography is very important for hard-solid layers which are gaining significance. Optimal roughness rates can be well achieved using CVD technology. In high specific surface pressures, superplain hard solid surfaces (roughness $R_z < 0.1$ μm) lead to the formation of an unfavorable and imperfect lubricant film. On the other hand, low friction values can be obtained with the formation of a thin coherent lubricant film (e.g. 1–2 g $m^{-2}$ prelube without spot lubrication) with roughness rates $R_z$ between 0.5 and 1.5 μm [15.20, 15.21].

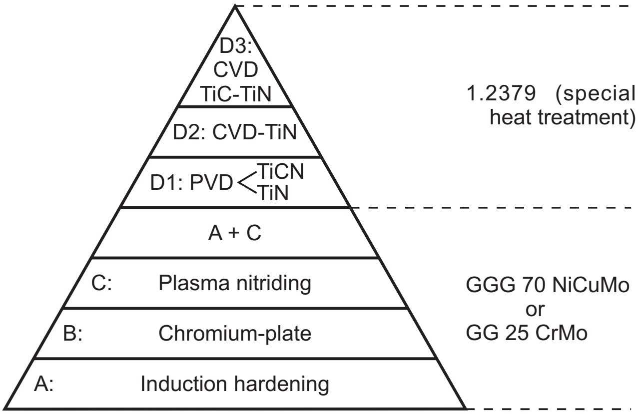

**Fig. 15.15** Methods for the surface treatment of tools used in sheet metal forming operations. Ranking according to wear resistance and basic materials (according to K. Keller and K.-R. Eversberg [15.20, 15.21, 15.23].

Figure 15.15 shows the most important methods in the surface treatment of tools used in sheet metal forming operations, ranking according to wear resistance and basic metals [15.20, 15.21, 15.23].

### 15.1.8
### Lubricants for Sheet Metal Forming

The process improvements and the increased knowledge in this working area have also increased the demands put on the lubricants required for the sheet metal forming. The selection of lubricants and their use depends upon various properties and are, in principle, to be viewed from three different angles:

- state of lubricant and workpiece before forming and practicable application
- behavior during forming
- corrosion protection and removability of the lubricant after forming

Gaining importance today are also economic, ecological, physiological and toxicological considerations.

#### 15.1.8.1 Before Forming

In this field of application a great number of different lubricants are available. An elementary classification without any reference to the performance is given in Table 15.2.

**Tab. 15.2** Variety of forming lubricants.

| *Lubricant* | *Viscosity or consistency* |
|---|---|
| Vanishing oils | 2–8 $mm^2\ s^{-1}$ at 20 °C |
| Mill oils (on sheet metal) | 10–50 $mm^2\ s^{-1}$ at 40 °C |
| Low viscous drawing oils | 10–50 $mm^2\ s^{-1}$ at 40 °C |
| Medium viscous drawing oils | 50–150 $mm^2\ s^{-1}$ at 40 °C |
| High viscous drawing oils | 150–2000 $mm^2\ s^{-1}$ at 40 °C |
| Drawing compounds | flow rate 30–60 s |
| Drawing compounds | flow rate 60–120 s |
| Drawing compounds | penetration 280–450 0.1 mm |
| Bonded film lubricants | solid |
| Drawing foils (plastic) | solid |

Table 15.3 shows different types of forming lubricants and their preferred applications.

**Tab. 15.3** Different types of sheet-metal-forming lubricants.

| *Lubricants* | *Main applications* | *Remarks* |
|---|---|---|
| *Neat lubricants* | | |
| Vanishing oils | – stamping, drawing, pressing<br>– deep drawing of thin sheet metal<br>– punching and calibration | flash point 40–65°C |
| Oils containing polar additives | – lamella and disk plate stamping<br>– embossing, stamping, drawing, pressing<br>– car body manufacturing, deep drawing | low viscous application some lubricants mineral oil free, biodegradable, compatible with prelubes |
| Oils containing EP-additives (chlorine free) | – severe stamping, pressing, deep drawing<br>– fine blanking<br>– drawing and ironing | lubricants can cause copper staining |
| Oils containing EP-additives (chlorine containing) | – severe stamping, pressing, deep drawing<br>– fine blanking<br>– drawing and ironing | high carbon steel, stainless steel, some lubricants do not cause copper corrosion |
| Drawing compounds without solid matter additives | – severe drawing, deep drawing | low to high carbon steel |
| Drawing compounds containing solid matter and/or EP-additives | – most difficult drawing, deep drawing | mostly pasty lubricants |
| *Watermiscible lubricants* | | |
| Oils | – drawing emulsions or solutions<br>– secondary hydraulic fluid for hydroforming | synthetic, semisynthetic or conventionel formulated lubricants |

| *Lubricants* | *Main applications* | *Remarks* |
|---|---|---|
| Drawing compounds without solid lubricants | – severe drawing, deep drawing | sometimes pasty lubricants |
| Drawing compounds containing solid lubricants | – most difficult drawing, deep drawing | often pasty lubricants |

The delivery of lubricants for metal forming depends on the physical properties and the way they are used. Liquid lubricants needed in large volumes are transported in rail tank wagons or road tankers and pumped directly into storage tanks at the user's plant. In other cases delivery is made in drums or canisters with screw caps.

Lubricants with greasy or pasty consistency are delivered in drums with removable covers or hobbocks. Delivery in bulk containers has proven to be advantageous for many users (so called IBC = Intermediate Bulk Containers), generally just called 'containers'.

Wherever possible, products are selected which are flowable as delivered and can be pumped or poured out. Some forming lubricants are used as oil-in-water or water-in-oil emulsions. Water-in-oil emulsions are usually delivered ready for use but oil-in-water emulsions are generally mixed on site. Oil-in-water type emulsions are made up by adding the concentrate to water and the water-in-oil type emulsions are made up by adding water slowly to the provided concentrate while mixing.

As a rule automotive grade strip steel and sheet metal are protected with an oily film, either with mill oil or with prelube (see Section 15.1.12).

When deep drawing, sheet metal lubricants can be applied by manual coating, rolling, immersion, greasing rollers or spraying. Manual greasing is possible with products of any consistency as long as coating by hand does not require too much physical effort.

Application by other methods calls for soft pasty to liquid consistency possibly at relevated temperatures. Suitable for applying by immersion, for example, are emulsions where the immersion temperature is dependent on optimal wetting of the material and the type of pretreatment. In the case of hot immersion with emulsions, most of the water evaporates leaving a thin film of concentrate which, in many cases, does not have to be removed after forming.

Coil and blank washing oils and emulsions usually used to clean automobile body panels before pressing and forming should be compatible with the mill oils and prelubes present on the supplied sheet metal. Ideally they should be out of the same chemistry.

Basically, all the lubricants suitable for immersion lubrication can also be applied by rollers. For a number of lubricants, felt roller application is not suitable when component separation occurs. Moreover, problems can be caused when applying water-in-oil emulsions with felt rollers.

The most favorable type of lubrication is by spraying. As in the case of manual application, it is possible to apply the lubricants specifically on those areas (spots) where it is required, leaving other areas unlubricated. This is where the term 'spot lubricant' comes from. Spot lubrication is being used increasingly for forming operations in sheet metal working. The advantage of this partial lubrication is not only to be seen in the economical use of the lubricant but also in the reduced oil contamination of the presses and the local environment. Moreover, the subsequent cleaning operation is made easier. In principle, all oils, emulsions and the majority of drawing compounds can be sprayed. The sprayability can be adjusted specifically to meet press shop demands.

#### 15.1.8.2 **Lubricant Behavior During Forming**

The success of sheet metal forming depends on three groups of influencing variables:

- on the metallurgical property of the metal and its surface
- on the mechanical forming process and the applied machine and the tools
- on the type and quantity of lubricant applied, from its consistency and on its performance features.

The most significant variable in all forming processes is the formability of the material. The degree of difficulty determined by this is essential for lubricant selection (see Section 15.1.2).

It is expected that a lubricant will facilitate the sliding of the workpiece in the tool by reducing friction and, at the same time, provide an effective separation layer between the workpiece and the tool. Since hydrodynamic lubrication can be expected when forming in only a few cases, the lubricant must contain additives which ensures an effective separation layer during the entire forming process. These additives can be polar in nature or those that change the surface of the metal by chemical reaction. In this way, metal contact between the metal surfaces is avoided.

A similar separation effect can be achieved by adding finely distributed solid substances in the lubricant which function as inert filler or solid matter lubricants.

#### 15.1.8.3 **After Forming**

As mentioned before, steel sheet metal and strip are generally provided with a film of corrosion protective or prelube oil in the steel rolling mill. The range of products have not only proved successful as corrosion protective oil but also for the further processing in light to medium severity drawing operations without an additional lubricant having to be applied.

The remaining film on the parts drawn in this way provides good corrosion protection.

If an additional lubricant (spot lubricant) is applied for forming this can be left in many cases on the workpiece after the operation to provide corrosion protection during intermediate storage or before further processing. However, in the majority of cases, it will be necessary to remove the lubricants immediately after processing or intermediate storage. This is done either by washing or, in the case of heat treatment, during the annealing operation without previous cleaning.

So-called industrial cleaners, generally of an aqueous alkaline type, are used for washing. One differentiates mainly between two groups of cleaning systems, the immersion bath or spray cleaner.

These detergents are comprised of synthetic organic and inorganic components in conjunction with wetting agents and are optimized for the respective form of application. This group of products is presented in detail in Section 15.1.10 Cleaning.

All lubricants for sheet metal forming must be removable with commonly available cleaners. The development of both forming lubricants and industrial cleaners often takes place in the same department. In this way optimal servicing of the user is ensured even in the case of difficult technical applications.

The use of hydrocarbon and halogenated hydrocarbon solvents is restricted to special cleaning machines and is not discussed here.

Deburring, friction grinding or vibratory grinding are further processing methods which are to be carried out with functional working fluids similar to those used when cleaning.

In many cases, cleaning can only be carried out after other operations have been completed, especially in automobile production. This is why the forming lubricants used there have to be compatible with structural adhesives, sealers and other materials to ensure process compatibility (compare flow chart in Section 15.1.12).

Because even small remaining quantities of lubricant found in folds and at similar areas can influence the quality of electro immersion painting (e.g. cataphoretic painting), such lubricants must also be compatible with these paints to a certain extent.

Normally it is required that the forming lubricant does not prevent subsequent welding and that the lubricant does not form welding residues which cannot be removed in common cleaning lines. Thin layers of oil and drawing compounds are generally acceptable. However, synthetic forming lubricants can be advantageous for some specific types of welding operations.

#### 15.1.8.4 Trends in Sheet Metal Forming Lubricants

Generally, and also in this field of application, the consumption of lubricants decreased during the last years. Also the variety of lubricants used in the automobile industry was diminished. On the other hand, simultaneously the complexity of requirements increased. Critical points for selecting lubricants include

- reduction of friction,
- corrosion prevention,
- compatibility with paints and adhesives,
- application procedures,
- effects on CVD/PVD-coated tools.

Because of these trends, great efforts have been made to improve the particular properties of the lubricants, especially concerning the additive levels these include:

- reduced use of chlorinated additives,
- no nitrites, no aromatic hydrocarbons,
- less volatile components,
- unpigmented, low content of solid lubricants.

## 15.1.9
## Corrosion Protection

### 15.1.9.1 Corrosion Mechanisms

One of the principal requirements of lubricants for sheet metalworking is that they provide protection against corrosion. This characteristic, which is often viewed as a secondary requirement, is sometimes the main purpose of the product. This is absolutely the case with corrosion protection oils which are mill-applied to sensitive cold rolled sheet steel. This aspect warrants closer examination because of the importance of corrosion protection.

Corrosion can be classified according to a range of criteria. With regard to its mechanisme, corrosion is most often differentiated by its type and its visual characteristics [15.24].

Oxygen corrosion is the most physicochemically important corrosion mechanism affecting sheet metal. The oxygen in the air at the metal surface is reduced to hydroxide in the presence of water and the metal is oxidized to a cation. A corresponding metal hydroxide rapidly forms and in the case of iron, continues to react to mixed oxide/hydroxide forming rust. In atmospheric corrosion, which is the economically most important form, these steps take place in the ambient atmosphere. The water necessary for a corrosion attack is also airborne and precipitates on the surface of the metal to form an invisibly thin electrolytic film. The thickness of this adsorbed water film and thus the risk of corrosion increases as humidity rises. In general, up to 60 % relative humidity is treated as favorable while 75 % and above is considered to be a critical climate.

If the ambient air cools around a colder metal or as a result of climatic changes, the temperature falls below the dew point. Large amounts of water can condense on the metal surface. Corrosion is also rapidly accelerated by some salts, especially chlorides. As a result of the better solubility of such metal salts, a passivated protective layer cannot be formed. In the case of hygroscopic salts, water is also absorbed from the atmosphere. Ultimately, local corrosion takes place, known as pitting.

Other corrosion mechanisms such as acid corrosion, stress corrosion and the corrosion of the less noble metal caused by contact with metals of differing electrochemical potential are of less significance to sheet metal working. In crevice corrosion, the oxygen concentration gradient caused by diffusion effects also leads to a corrosive difference in electrochemical potential. In the case of copper- or zinc-based materials, dark stains can be caused by chemical corrosion in the presence of sulfur from sulfur-based additives or the atmosphere.

The different types of corrosion are most often classified by their location or appearance rather than by the actual corrosion mechanism. Examples of this are the already-mentioned term crevice corrosion, pitting or the red rust which can affect ferrous metals. White rust forms on zinc and its alloys when a passivating protective layer of zinc carbonate is not formed. This can occur when these materials are stored in the presence of salts which are deposited by metalworking emulsions.

#### 15.1.9.2 **Temporary Corrosion Protection**

To stop the corrosion of metallic materials, their exposure to aggressive media must be interrupted. The lubricant layer acts as such a separator by forming a hydrophobic barrier through which water and salts can only diffuse very slowly towards the metal's surface. Even highly non-polar and hydrophobic mineral oil does not form a complete barrier to water, oxygen and salts. This is why the addition of corrosion inhibitors is necessary. These are adsorbed on the surface of the metal and form a very thin but extremely dense protective layer in molecular terms. In spite of being very thin, the adsorption layer is highly effective. These interactions are illustrated in Fig. 15.16, whereby the adsorption layer has been greatly magnified for graphic reasons. Roughly speaking, the protection against corrosion is a result of the barrier effect (passive) and the adsorption layer (active).

A special case is the inhibiting of the surface by substances adsorbed from the gaseous phase which are known as VCI (volatile corrosion inhibitors) or VPI (vapor phase inhibitors). These inhibitors are special amines, mostly alkanolamines which have a certain vapor pressure as well as being adsorbed on the metal to offer active protection. In order that the gas phase has the necessary inhibitor concentration, the packing around the metal to be protected should be well sealed. To compensate for the unavoidable losses of inhibitor through small leaks in technical packaging, the VCI substance is usually an amine salt. This salt itself does not evaporate but creates an inhibitor reservoir from which free amines can be released until the reservoir is exhausted. When the metal parts are unpacked, the inhibitor desorbs again leaving the metal surface unprotected. The function of VCI products is shown schematically in Fig. 15.17.

Depending on the application, the VCI substance itself can be incorporated into a variety of carriers. Apart from the well-known papers, innovative concepts are gaining in importance. Significantly better protection is offered by the combination of polymer films and VCI. The VCI substance can be added to the inner layer of a

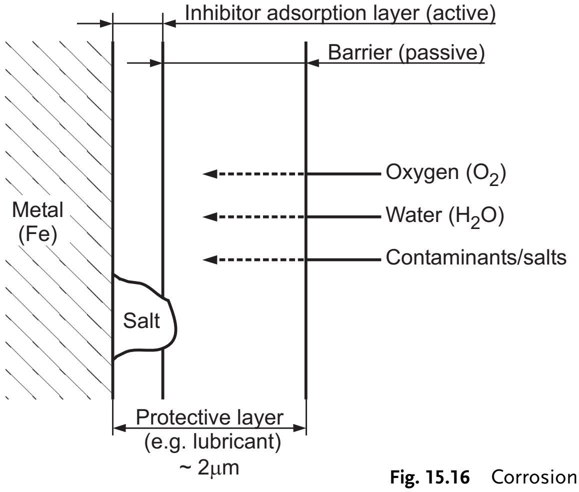

**Fig. 15.16** Corrosion protection by thin organic layers.

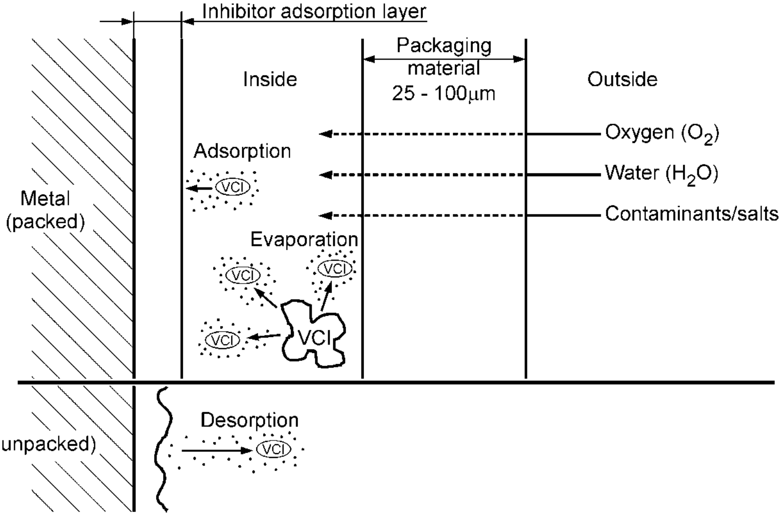

Fig. 15.17 Corrosion protection by volatile corrosion inhibitors.

multi-layer extruded polyethylene film. The benefits of this are minimal water vapor permeability and good VCI protection. The VCI substance can also be added in the form of a powder bag so that normal polymer films can be used. VCI technology is particularly recommended for the protection of car body steel coils or pressed parts under critical transport or storage conditions. Depending on the type of packaging used, the normal thin application of an effective corrosion protection oil is then adequate even for sea-transportation without the need for an additional oil.

As lubricants normally have to be removed after sheet metal forming, only inhibitors which are physically or chemically adsorbed come into question. Substances which chemically react with metals can only be used if they do not interfere with subsequent operations. An example of this is the phosphating of car body panels which is primarily performed to improve the forming characteristics of the sheet metal.

As all fluids, also oils are affected by gravity and runoff metal surfaces. The thicker they are applied and the lower the viscosity, the quicker they run-off. This effect is magnified by the smoothness of galvanized panels. The result is a loss of lubricity and protection but also possible oil-dent problems during pressing and general soiling by the run-off oil. As high viscosity makes the application and removal of the oil more difficult, this parameter has its limits. Run-off can nevertheless be retarded if thixotropic oils are used. These are comparatively low viscosity oils which contain tiny particles and are thus turbid (cloudy). The viscosity of such thixotropic oils increases as the film thickness and flow rate falls. Film thickness lower than the dimensions of the thixotropic agents themselves therefore offer the best retardation. In sheet metal forming, soft, organic components such as soaps or paraffins are often used to retard run-off. If the thixotropic agents are fully dissolved in oil at temperatures between 40–80 °C (temperatures normally encountered during application and removal), the thixotropic characteristics decay. This effectively avoids problems stemming from high oil viscosity during the storage and transport of the panels while offering optimum run-off retardation [15.25].

#### 15.1.9.3 **Corrosion Tests**

The extent of corrosion protection offered can be evaluated by standardized laboratory tests or by practical trials. The standardized chamber tests are reproducible and allow a direct comparison of similarly-determined results. For obvious reasons they only roughly replicate real-life corrosive conditions. Contrary to that, outdoor weathering tests or field trials are performed in the same conditions and are subject to the same corrosion-inducing influences as are all industrial processes. However, because of their poor reproducibility, only products in the same series of tests can be accurately compared. Nevertheless, to at least achieve some degree of comparability with other test series, a well-known reference product is also tested. Table 15.4 shows a summary of the tests frequently used for drawing lubricants.

**Tab. 15.4** Corrosion protection tests for drawing lubricants.

| *Cause of corrosion* | *Method* | *Tests* |
|---|---|---|
| Humidity | Condensation climate | DIN 50 017 KFW/KTW |
| | Stack corrosion test | Various in-house tests |
| | Constant climate test | ASTM D 1748 |
| | | DIN 50 017 KK |
| Atmosphere | Practical trials<br>(Outdoor weathering,<br>Indoor weathering) | Various in-house tests |
| Salts | Salt spray test | ASTM B 117/DIN 50 021 SS |
| Copper-active additives | Copper strip test | DIN EN ISO 2160 |

### 15.1.10
### Removal of Forming Lubricants – Industrial Cleaners

All substances and particles which could negatively affect the production process or the later use of the metal product have to be removed from the surface areas.

During the production process, the surface of a workpiece comes into contact with various materials which, on the one hand, are designed to facilitate the processing but, on the other hand, may produce residues which may lead to disturbances and failures in the subsequent further processing. In addition, the processing will also lead to abrasion. Finally, one has to mention contamination which will unintentionally collect on the metal product's surface during storage and which have to be removed before further use.

In the following the removability of forming lubricants as well as the surface cleaning in general will be considered. According to the application three areas are differentiated:

- intermediate cleaning in the mechanical manufacture
- cleaning prior to heat treatment and surface coating
- cleaning during maintenance.

There is no generally accepted method for determining the 'degreasing' effect of a cleaner. Rather, the success of the cleaner is determined when follow-up operations can be carried out without disturbances. In the automotive industry, it is the water-wettability of the metal surface after the cleaning process which is used as a criterion. In this context it is worth mentioning the test methods according to ASTM F 22 (test method for hydrophobic surface films by the water-break test), ASTM F 21 (test method for hydrophobic surface films by the atomizer test) and the Volkswagen procedure (test instruction 52.02: cleaning test in a slowly moving bath to test the removability of corrosion protection oils and the performance of industrial cleaners).

For a more scientific study of dirt special surface contamination detectors utilize UV light and the subsequent effect of emission. Instruments based on optical stimulated electron emission provide an effective detection on all surfaces that support this kind of emission, such as metals, many paints and polymers, and some ceramics. Inert materials attenuate significantly the electron emission. As a result, these contaminations easily can be detected by this method.

#### 15.1.10.1 **Intermediate Cleaning in Mechanical Manufacture**

Contamination which has developed during mechanical manufacture have often to be removed from the workpieces prior to the dimensional control procedure. Cleaned metal surface areas are sensitive to corrosion. Therefore, it is recommended to apply a temporary corrosion protection to the workpiece during cleaning. This is done by adding passivating active substances to the cleaners or by passivating the metal surface in a separate stage immediately after cleaning. Many cleaners, however, already contain corrosion protection components which offer a sufficient protection during intermediate storage.

Nowadays both neat and water-based cutting fluids are used for intermediate cleaning when suitable.

#### 15.1.10.2 **Cleaning Before Heat Treatment and Surface Coating**

Metal cleaning is an obligatory element in the surface treatment of metals. Only clean and residue-free metal surface areas are suitable for thermochemical processes and ensure a perfect bond of the coating layer. However, differences can be seen between the requirements in the individual thermochemical and coating processes. Enameling processes are especially affected by traces of organic contamination due to the high baking temperature which is used here.

Decomposition will lead to faulty enameling results: 'fish scales', bubbles, pores. Even minor contamination will have a negative impact on the appearance of galvanic coatings and influence the bond of galvanically produced layers.

On the other hand, minor surface contamination can be tolerated in processes like hot-metalizing (galvanizing, tin-coating and lead-coating).

Cleaning metal parts prior to painting is often combined with the application of a thin passivation film (e.g. a phosphate layer) in order to provide a basic coating layer for reliable corrosion protection and adherence of the paint.

#### 15.1.10.3 Cleaning During Maintenance

Machines and plants which become contaminated during operation have to be cleaned frequently. This not only applies especially for vehicles and all types of production machines but also for barrels, tanks and refining plants.

#### 15.1.10.4 Cleaning Methods and Agents

It is seldom possible to remove all contamination from metal surfaces areas in one work stage. Often several cleaning operations have to be combined in one process using physical, chemical and physicochemical methods. Apart from the pure mechanical machining (blasting, grinding), cleaners are also made use of which can either be organic solvents working on a purely physical basis, or chemically active aqueous solutions. Their effect can be enforced by means of physical procedures, e.g. ultrasound.

The cleaning properties of water were known as long ago as the primeval times. Its liquid form allows water to reach ‘everywhere’, dips as well as in cavities and allows it to wet surface areas and the dirt bonded to them. Today, cleaning with aqueous solutions is still the commonly used method and is carried out most frequently in form of aqueous alkaline cleaning.

In this respect, saponification normally is less important. A major role, however, is played by physical and colloidal chemical processes such as adsorption and desorption, particle attraction and repulsion, generation and breakdown of emulsions, whereby wash-active substances, so-called surface- or interface-active substances, take over the control function.

One of the main groups of these substances are ionogenic compounds (bases, acids, soaps, salts) which become active in aqueous solutions. The negative particles in these solutions, the anions (therefore the term anion-active), tend to collect on contaminated surface areas, causing the negative charge of the dirt particles. As the metal surface is also negatively charged, it repels the dirt particles.

The affinity of the anions to the contamination is not the only active factor. Silicates and phosphates can form relatively large polymeric ions (so-called builders) in aqueous solutions. In comparison to smaller ions, they have the advantage that they keep the separated and water-insoluble dirt particles floating in the cleaning solution colloidally, i.e. finely dispersed so that the dirt particles are not able to bond to the cleaned surface again.

Apart from inorganic matrix materials, modern industrial cleaners contain organic compounds which add to the anion-active compounds. These are the non-ionogenic surface-active substances which were discovered in the 1920s. They stand out due to their soap-like properties, however without the disadvantages, e.g. precipitations in hard water. Today, they can be found in nearly all industrial cleaners and detergents.

The molecules of the wash-active substances (generally called tensides) combine characteristics which normally can be individually identified in various substances and apparently rule each other out. Following the principle 'similia similibus solvuntur', a substance will only dissolve in a similar medium, the majority of the substances is soluble either only in water or only in oil. On the other hand, the tensides allow these different molecular components to form one molecule. Tensides consist of a hydrophilic (water-attracting) and a hydrophobic (water-repellent) part. Therefore, they are water and oil-soluble and show a particular 'hybrid' behavior. When mixed with aqueous solutions, even the smallest amounts of tensides develop a very high efficiency. Physically, this can be explained with the tensides' property to collect mainly on interfaces, and this allows high concentrations to be found locally.

Only the addition of these compounds contributes to the high efficiency of today's cleaners. The tensides reduce the water's surface tension and make it in a way more liquid. Therefore, the detergent solution can wet, surround and infiltrate all contamination much better than pure water and penetrate even the first cracks and gaps. The tenside molecules enable a considerable facilitation in the removal of dirt particles while increasing the cleaning effect.

In solutions of certain concentration levels, they will produce molecular groups (the so-called micelles) which serve as back-up resources for the procedures described. They are able to disperse or emulsify (distribute and keep solved) water-insoluble substances, such as oils and greases.

In modern industrial cleaners inorganic, ionic and non-ionic substances establish an optimal and effective combination. The cleaner's efficiency is based on various physical and chemical interrelations coordinated with each other.

### 15.1.10.5 Systematization of Cleaners

Users can choose between various types of cleaner – aqueous cleaners, solvent cleaners, and emulsion cleaners – and between various cleaners within those types. The choice of cleaner type and individual cleaner depends on the requirements of the user.

#### Aqueous Cleaners

Aqueous cleaners are available in pulverized and liquid form, whereby nowadays cleaning liquids are used more and more due to reasons of safety and easier handling. Most cleaners belong to the group of alkaline of neutral cleaners. Acid cleaners play a minor role.

The various possibilities regarding concentration, temperature and application, mean that these cleaners can be used under many different conditions.

The cleaning success depends on the level of contamination. When stored for a longer period prior to the cleaning, some lubricants tend to gum up and harden, e.g. through oxidation or evaporation of individual components. In general, this leads to a decreased removability of the contamination.

Highly pigmented forming lubricants and forming lubricants containing solid matter often cause difficult degreasing problems. A standard solution has not yet been found since the removability of the solid matter from the workpiece's surface structure depends on the geometry and particle size of the solid matter, on specific interactions between solid matter and workpiece surface, on the load during the forming process and on type of solid matter used.

As cleaning residues may have an effect on the layer production during the chemical surface treatment and thermochemical processes, even after profound rinsing, only especially developed cleaners are used in order to avoid failures and disturbances. In addition, the natural character of the basic metal which is to be treated (iron, aluminum, copper, brass, zinc etc.) has also to be taken into consideration.

According to the pH value of the alkaline cleaning solutions, a difference is made between:

- low-level alkaline cleaners (pH between 7.0 and 10.5),
- medium-level alkaline cleaners (pH between 10.5 and 11.5), and
- high-level alkaline cleaners (pH above 11.5).

Low-level alkaline cleaners are often called ‘neutral cleaners’, as they are characterized by a low tendency to saponify natural and synthetic esters. A standard definition of the term ‘neutral cleaners’ does not exist.

Acid cleaners (pH below 7.0) are used for special applications. Apart from the degreasing effect, these cleaners show a caustic effect for the removal of rust, rust flake or other solidly-bonding layers which cannot be removed in alkaline media. Such caustic degreasers consist mainly of hydrochloric acid, sulfurous acid or phosphorous acid added with acid-resistant tensides.

In addition to the degreasing of lubricant films, acid degreasers based on phosphorous acid are also used to produce a thin iron phosphate layer (approximately 0.1–0.3 μm), thus providing a key for a painting layer, e.g. cataphoretic immersion paintings and/or powder coatings.

#### *Application Range for Aqueous Cleaners*

The decisive criterion in the selection of cleaners is the applied cleaning method:

- immersing-bath cleaners (dip cleaners) which are basically emulsifying and
- spray cleaners which are basically demulsifying.

For special applications there are:

- high-pressure cleaners
- steam jet cleaners,
- machine cleaners.

### Immersion Cleaning

Immersion cleaning is an old cleaning method. The cleaners used here dissolve oils and greases easily and disperse dirt particles very well. Immersion baths are recommended especially for small parts or flat parts with large surface areas which may be dipped into the bath in relatively thick layers. They are also suitable when more processes are carried out in immersion baths, e.g. electroplating treatments.

Subsequent to the immersion method, the parts are cleaned in a bath solution at high temperatures for a relatively long treatment period. Often, one stage will not be sufficient and multistage cleaning methods are applied in accordance with the task range.

### *Immersion Cleaning Facilities*

Standard immersion facilities can be constructed simply. The easiest solution is a heatable container made of sheet iron, e.g. the decoction bath which operates at boiling temperature and is nowadays applied in few cases only. Here, the surging of the boiling solution serves as additional mechanical cleaning support. Modern facilities avoid the disadvantages connected with the 'decoction' cleaning: high heat losses, problematic steam swathes, splashes and boiling-over.

The cleaning effect can be mechanically supported by circulating the cleaning solution or by flooding during which the solution is re-pumped and pressed into the bath through numerous valves positioned, staggered, opposite each other. As an alternative, the bath is re-filled and drained several times with a flood pump. By using an additionally installed sprinkler system, creamed grease is rinsed off the bath surface into a spillway, thus preventing that the cleaned parts are re-oiled with demulsified oil when taking them out of the bath. In comparison with the 'decoction' bath, well circulated baths have the advantage that they ensure a perfect cleaning result at a lower temperatures (60–90 °C) and after a shorter period of treatment. Occasionally, the bath solution is circulated using pressurized air instead of the pumping method. Here, cleaners with a particular low foaming tendency are needed.

### **Ultrasound Cleaning**

Another immersion cleaning method is ultrasound cleaning. In some industries, e.g. in the manufacture of jewelry and silver products, cutlery, optic glasses and devices, as well as in the production of high-precision fine-steel tubes, fittings and many structural components, extremely high requirements are made on the cleanliness of the mostly polished surface areas. Residues of the materials used and metal abrasion particles can only be removed completely with great effort because such pigment contamination cling to metal and glass surface very strongly.

Mostly, a treatment with aqueous cleaning solutions alone will not be sufficient if it is not supported by additional mechanical actions. In these cases, the use of ultrasound will have more effect than the flooding of the cleaning solution or a spray treatment. Ultrasound cleaning is based on high-frequency sound vibration which is generated with special vibrator systems.

### *The Effects of Ultrasound*

In the cleaning solution ultrasound is transmitted in longitudinal direction as a longitudinal wave. Due to strong pressure variations, the solution produces numerous small low-pressure bubbles which immediately collapse releasing energy. Here, the micro-roughness of the surface areas and the accumulated particles act as cavitation nuclei. This is particularly true for corners, drill holes, decorative embossings and other areas where conventional cleaning methods will not lead to optimal results.

In clear solutions one can observe after a few seconds, how the dirt particles are removed from the surface areas. This cleaning method is based on the pressure generated by the collapsing bubbles, which can locally exceed 1000 bar.

Sound frequencies of between 20 kHz and 40 kHz, as well as operating temperatures of between 50 °C and 70 °C will lead to the best results. Generally, the minimum cleaning period does not exceed two minutes.

The ultrasound generator of a cleaning system should be set so that a level of the sound energy of between 5 and 20 W $l^{-1}$ solution of the immersion bath is achieved.

#### Spray Cleaning

This modern cleaning method is based on one or more coordinated stages. The intensive mechanical effect of the spray jets has the advantage that in comparison with the immersion bath the period of the treatment is considerably shorter and the operating temperature often can be reduced to 40–70 °C.

Much lower quantities of cleaning solution are needed than in the immersion bath, thus reducing its concentration to 10–30 g $l^{-1}$. This means less use of chemicals, less consumption of heating energy and lower costs for waste-water processing and disposal.

##### *Spray-cleaning Facilities*

Spray cleaning facilities have a complex construction. As a result of this, they stand out due to several important advantages with their closed type of construction. Even at high temperatures, the suction and condensation systems ensure that no disturbing water steam will release into the workshop hall.

Decisive for the intensive cleaning effect are the shape and the order of the nozzles which are positioned in nozzle connections so that during the cleaning the parts are sprayed from all directions, thus avoiding any non-sprayed zones. Normally, the spray pressure is between 0.5 and 10 bar. It will, however, be considerably higher (up to 500 bar) during special cleaning procedures, especially during deburring and high-pressure cleaning. There are one-chamber spraying machines, in which the parts to be cleaned are sprayed from all directions on a flexible grid, as well as multi-chamber continuous spraying facilities. By using these facilities, cleaning, rinsing, pickling and passivating processes can be combined with the drying and applying of anticorrosive agents in a complete pretreatment procedure. The main advantages are a higher throughput than in the immersion bath and an improved process safety.

The number and length of the individual chambers of continuos spraying facilities have to be adjusted to the respective pretreatment problem. The type of nozzle depends on the function of the various chambers. Flat-jet nozzles are used in cleaning, degreasing and pickling chambers, whereas hollow cone nozzles are used in rinsing, passivating and neutralization chambers. The number of nozzles and their order is predominantly determined according to the shape and size of the parts to be treated.

The most important prerequisite for the perfect operation of spray cleaning facilities is the use of quickly emulsifying cleaners. By skimming or filtering, it can be ensured that a ‘clean’ cleaning solution is available over a longer period of time.

#### *Products for Spray Cleaning*

Spray cleaning requires a different construction of the cleaners than in immersion cleaning. The most important characteristics is the different foaming behavior: spray cleaners contain demulsifying tensides with low foaming tendency. It must be observed that the foaming behavior depends on the temperature, i.e. minimum operating temperature has to be reached in order to ensure the self-defoaming effect. This means that the water in the spray cleaners has to be heated while preparing the cleaning solution, prior to adding the cleaner concentrate. The temperature has to be kept on the same level also during down-times to avoid unwelcome foaming during the restarting process.

### Manual Cleaning, Cleaning with Steam Jet and High-pressure Machines using Aqueous Cleaners

#### *The Cleaning Table*

In many workshops and smaller manufacturing companies, metal cleaning is still carried out manually using a brush. Today, however, several developments have been introduced to improve and facilitate this manual work. Instead of using the petroleum tub with brush and cleaning cloth, a cleaning table with a hose-pipe brush for the cleaning solution is used. In many cases, the petroleum can be replaced by high-performance aqueous cleaners which are often better suited for the cleaning of repair parts.

#### *Manual Sprayers*

Often, a spraying process is also used, e.g. in the cleaning of vehicle engines or facilities and machines which can not be transported. The cleaning solution is sprayed onto the surface and allowed to work in. Higher contaminated surface areas are treated with the undiluted cleaning concentrate.

Then the dirt particles are sprayed off with a strong water jet. The surface areas are dried in the air. The drying process is accelerated by blowing pressurized air on the surface.

#### *Steam Jet or High-pressure Machines*

Manual cleaning can also be carried out using steam jet or high-pressure cleaning machines which will normally not work with solvent cleaners but with aqueous cleaning concentrates. The cleaning concentrate is mixed with hot steam or water. This mixture is sprayed onto the respective surface under high pressure. The temperature and jet pressure can be adjusted to the individual application.

### Cleaning with Organic Solvents

The solvent cleaners mainly consist of chloro- and chlorofluorohydrocarbons, and hydrocarbons and solvent cleaners.

### Chloro- and Chlorofluorohydrocarbons

In recent years, the use of organic solvents, especially halogenated hydrocarbons, was made more difficult because of legal measures covering work place protection (Chapter 9). Nevertheless, higher emissions and the immediate exposure of the staff can be avoided with the introduction of corresponding technical measures.

The most important substances in this group are trichloroethylene (trichloroethene), perchloroethylene (tetrachloroethene) methylene chloride (dichloromethane), 1,1,1-trichloroethane and, the chlorofluorohydrocarbons R 11 and R 113, which are also used as refrigerants. The most important criteria for their use are their high ability to dissolve polar substances, the quick drying of the degreased parts, the avoidance of fire hazard in contrast to pure hydrocarbons and the possibility of regeneration by means of distillation. Distillation methods make it possible to separate foreign components. However, it has to be taken into consideration that the boiling point of the compound will increase due to the dissolved lubricant components and that an accelerated decomposition process, accompanied by the formation of hydrogen chloride, starts at about 120 °C for trichloroethylene and at 150 °C for perchloroethylene.

The solvent decomposition accompanied by the formation of hydrogen chloride is one of the largest problems in the area of cleaning with chlorohydrocarbons. This decomposition can also start at temperatures which are clearly below the mentioned figures and can be catalytically accelerated by foreign matter, e.g. water. Conventional solvents for metal cleaning contain effective stabilizers in order to avoid this decomposition which is accompanied by a corrosion process. Therefore, it is important during distillation that the stabilizers mix with the distillate and do not remain in the sump. Normally, however, inhibitors are added for re-stabilizing purposes, thus neutralizing produced hydrogen chloride which has already been produced. A routine test of the halogenated solvents to determine the acid index, the pH of the aqueous extract or the chloride content is useful in order to safely avoid corrosion problems. Test kits are often used for operational monitoring.

### Hydrocarbons and Solvent Cleaners

White spirit, petroleum and isoparaffins are the hydrocarbons most used in metal cleaning. The comparably low prices and ease of disposal are advantageous, however, when using these substances the fire hazard has also to be taken into account.

### Application Range of Solvent Cleaners

The degreasing of pigmented forming-lubricants using solvent cleaners generally constitutes a problem. After dissolving away the lubricant film's soluble organic substances, the solid matter lubricant remains on the surface (see aqueous degreasing).

Normally, solvents are used at room temperature or in closed systems at the boiling point of the solvent – the so-called vapor phase degreasing. The vapor phase, in which the part to be cleaned is immersed, is produced above the boiling solvent bath. It then condenses highly pure solvent matter on the cold workpiece until the temperatures are the same. The condensed solvent rinses the oil film off the workpiece, which will either dry quickly or be immersed for short-term intermediate preservation in a solvent bath to which 1 to 5 % of a corrosion protection concentrate is added.

Solvent cleaners are a product group of their own. They are predominantly used in the cleaning of heavily contaminated or oiled repair parts, e.g. engines, gearing systems and major machines (locomotives). These products consist mainly of hydrocarbons with special additives. They are inflammable.

As a product group, solvent cleaners are related to solvents. They consist of hydrocarbon mixtures or synthetic hydrocarbons and contain further additives in accordance with the respective application, e.g. to improve the corrosion protection or washability.

Today, solvent cleaners which do not contain aromatic components have proven their suitability in many applications.

Solvent cleaners are used either in immersion baths or to spray the parts to be cleaned. According to the composition, they can be used as pure concentrate or diluted with petroleum or, some of them, with water. After it has been allowed to react for a certain time, the solvent cleaner is carefully sprayed off with a strong water jet. When so-called separating solvent cleaners are used, the emulsion generated during water spraying will split very quickly into an oil phase and a water phase so that the oil components are safely retained in the oil separator.

#### Emulsion Cleaners

The concentrates are to some extent analogous to water-miscible cutting fluids and consist of mineral oil, synthetic hydrocarbons and other organic fluids together with emulsifying agents and anti-corrosive compounds. They are mostly used in a concentration of 2 % in water. They do not lead to a cleaning level up to water wettability, which makes them suitable for the cleaning between two operations. Suitable water-mixed coolants with a low foaming behavior are often also used for this purpose. The residual film protects iron materials against corrosion.

##### *Maintenance of Cleaning Baths*

For reasons of cost minimization, the industry focuses more and more on the service life of cleaning baths.

In solvent cleaners, the solvent can be retained and kept fresh through distillation, as already mentioned. More effort is made for the maintenance in case of aqueous cleaning procedures.

Apart from the development of, for example, special spray cleaners with a very good demulsifying behavior, physical methods for the prolongation of the service life of cleaner solutions are also used. These include oil separators, skimmer, filtration systems or centrifuges for solid matter separation and microfiltration. The use of these methods not only ensures the separation of introduced contamination but will also sometimes cause the cleaner's components to be separated which then have to be re-dosed.

The ultrafiltration method facilitates the separation of most organic components from the aqueous phase. Inorganic salts reach the permeate. Thus, this method is also suitable for waste water treatment.

When vacuum-distilling cleaner solutions (e.g. in thin film vaporizers), only the aqueous phase is regained. The non-distilled residue contains the components of the cleaner and the contamination, including the salts.

### 15.1.11
### Testing Tribological Characteristics

Various methods are used to evaluate the deep drawing performance of lubricants. The principals are described in Section 15.1.3. The most common is the strip drawing test which is performed with and without deflection. This method simulates the conditions found under the blank holder and during the flow of material into the die. To examine wear, abrasion and the special behavior of metal coatings (powdering, cracking, smoothing effects) methods with strip deflection, e.g. draw-bead test or Erichsen test are used.

In the straight strip drawing test, a strip of metal is drawn between two flat dies. The drawing force required is measured against the normal force to determine the coefficient of friction. In addition, the time is also recorded. The most important measuring parameters are tool and material dimensions, drawing speed and the maximum drawing force applicable. Apart from the coefficient of friction, the uniformity of the force time-frame is an important criteria for evaluating the tribosystem. Ideally, the material flow between the tool is uniform and free of stick–slip. Finally, the maximum normal force possible until the strip jams is a measure of a lubricant's ability to separate the sheet and the tool under pressure. Evaluation of such data allows lubricants to be developed which are ideal for metal forming operations. An example for a strip testing equipment is shown in Fig. 15.18. The test stand used by the Institute for Production and Forming Technology of the University of Darmstadt, Germany, enables tribological systems to be examined with large tool dimensions, variable drawing speeds and very high normal contact pressures. The various stations are de-coiling, cleaning and oiling. Tools are equipped with sensors to measure forces. Finally, the strip metal is coiled again [15.26].

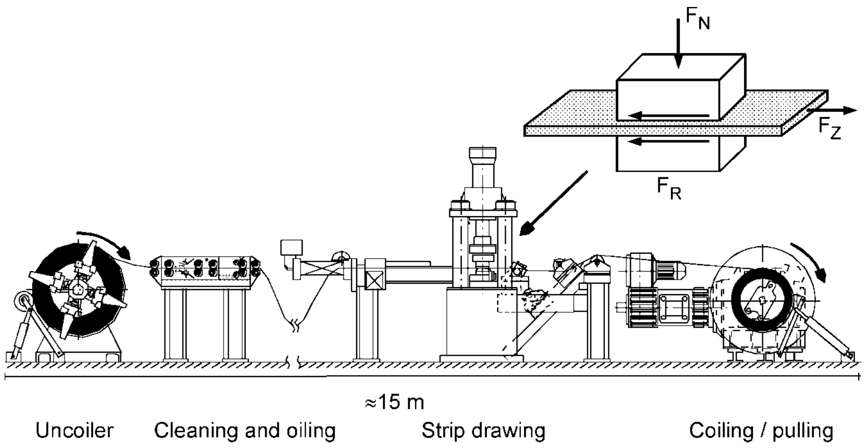

**Fig. 15.18** Strip-drawing tester and tools [15.5, 15.6].

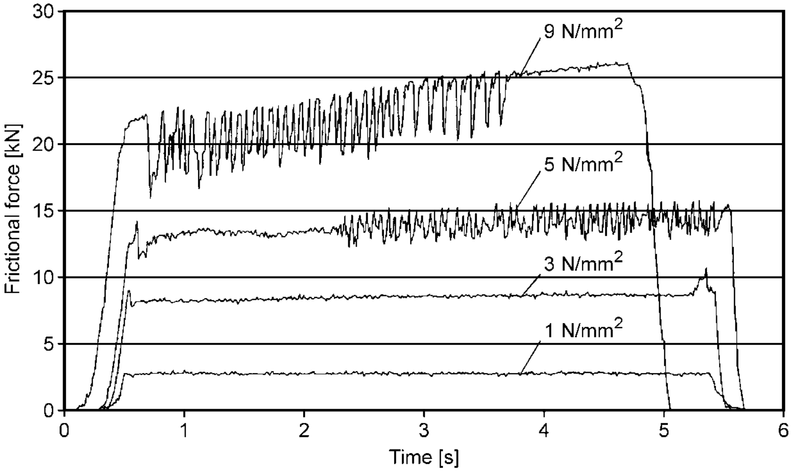

**Fig. 15.19** Frictional force vs time with load as parameter, mill oil/uncoated sheet metal/50 mm $s^{-1}$.

Figure 15.19 shows the results obtained from uncoated cold-rolled steel. The strip drawing test without deflection is reported on at all times. The lubricant is a mill-applied corrosion prevention oil. The forces measured are recorded against time. According to the friction law the frictional force increases with higher loads. From 5 N $mm^{-2}$ onwards stick–slip occurs.

The coefficient of friction as a function of the load is outlined in Fig. 15.20 where the drawing speed is varied. The coefficient of friction falls with increasing speed. The begin of stick–slip is marked. The curves end with the maximum load, when the strip jams and further drawing would cause the metal to tear.

As a rule, lubricants with a higher viscosity have better lubricity. To distinguish the effect due to pure rheology from that caused by additives, some lubricants with identical formula but with differing viscosities are tested. The result is shown in Fig. 15.21 with a corrosion preventive oil as example. The viscosity is chosen

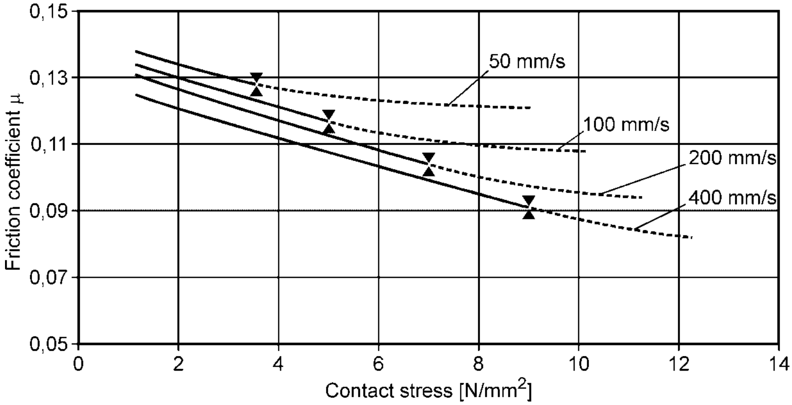

**Fig. 15.20** Friction coefficient vs load with sliding velocity as parameter, mill oil/uncoated sheet metal.

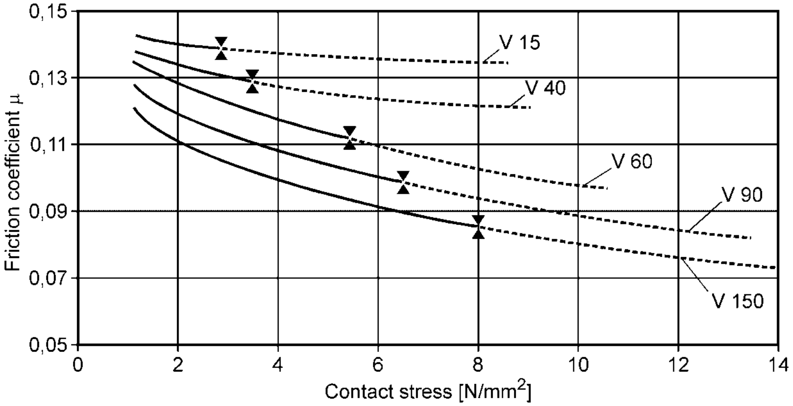

**Fig. 15.21** Friction coefficient vs. load with oil viscosity as parameter, mill oil/uncoated sheet metal/50 mm $s^{-1}$.

between 15 and 150 $mm^2\ s^{-1}$ at 40 °C to cover the most common applications from blank washing to drawing oils applied by spraying.

### 15.1.12
### Sheet Metal Forming in Automobile Manufacturing

The pressing of car body parts is one of the most important sheet metal drawing processes. The corrosion protection oil applied by the steel mills plays a part in every sheet metal forming operation and also makes up the largest proportion of lubricants used. The typical part transfer sequence from the sheet metal to the body-in-white is described in Fig. 15.22. At every stage where oil is applied to the metal surface a full compatibility with upstream and downstream processes is necessary for a high production reliability at minimum overall costs. The single steps of production are discussed in the special sections below [15.27, 15.28].

#### 15.1.12.1 Prelubes

The idea to combine the corrosion protection properties of a corrosion preventive oil with the lubricity of a drawing oil led to the development of the prelubes. Prelubes have existed on the American market for more than 20 years, they were introduced in Europe in the early 90s. Applied at the finishing lines of the steel mills, they serve as the forming lubricant in the press shops. As for the corrosion protection oils, a prerequisite for the suitability of a prelube oil is the absolute compatibility with all single processes from the coil to the body-in-white. The use of prelubes in steel mills reduces the number and quantity of spot lubricants for additional press shop oiling dramatically. But their true benefits can only be fully achieved if the compatibility principle is applied throughout the manufacturing chain. Therefore, modern prelubes systems are modular, even different viscosities can be part of the same concept. This results in a far-reaching multi-functionality for all applications.

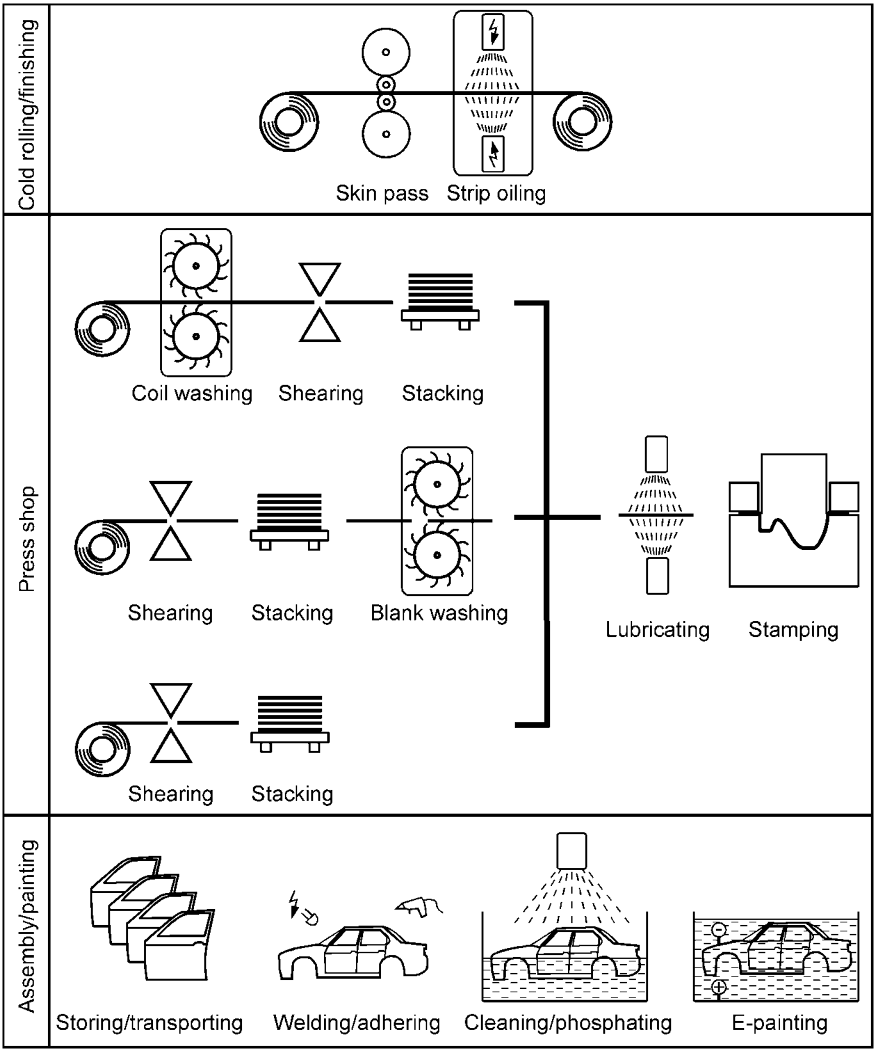

**Fig. 15.22** Process line.

Among the many specifications for mill oils, the comprehensive standard of the Association of German Automobile Manufacturers (VDA) [15.29, 15.30] describes all relevant requirements of a mill-applied lubricant, defines all important properties and lists all test procedures. The results–with the exception of lubricity–are compared with the standard corrosion preventive oil Anticorit RP 4107 S. According to these VDA requirements a prelube needs to be thixotropic for a reduced run-off and is applicable by electrostatic spraying. Of course it should protect the sheet metal against corrosion as effective as the standard oil. Precipitation or other chemical reactions must not occur when mixed with the standard oil. Furthermore, the prelube should be compatible with the cataphoretic paint and the adhesives used in the assembly line. Eventually, the prelube has to be easily removable with industrial cleaners in the preconditioning zone prior to phosphating and e-painting [15.15].

### 15.1.12.2 Skin Passing

To achieve the desired quality, the final surface finish of sheet steel is often rolled-on wet. Water-based solutions are normally used for plain cold rolled steel but low-viscosity oils are used for zinc-coated sheet including electro-galvanized or hot dip galvanized. As a result of the increasing use of coated sheet steel in recent years, the compatibility of skin-pass oils is becoming an increasingly important factor. If the skin-pass and the subsequently applied corrosion protection oil are not compatible, the oil film can rapidly separate in spite of having been evenly applied. The formation of dry islands is detrimental to the tribological system and can cause tearings or oil dents.

### 15.1.12.3 Coil Oiling

A prerequisite for the oil being used by steel mills is that it can be applied easily. The most common method these days is electrostatic spraying. The optimum spraying temperature has been shown to be 50–60 °C while the voltage, depending on the equipment itself, is between 80 and 120 kV. Oils with a viscosity of 30–60 $mm^2\ s^{-1}$ at 40 °C spray easily. If thixotropic oils are used, a full solubility of the dispersed agents at the spray temperature is essential for an optimum spray pattern and a low film thickness. On the other hand, the oil may have to be very finely filtered to remove any impurities in return circuits.

### 15.1.12.4 Transport and Storage of Sheet Metal

The principal objective of oiling coils and stacks in steel mills is to reliably protect against corrosion during transport and storage. Fundamentally, temporary corrosion preventives combine a barrier effect with specific inhibitors. The specific adsorption layer is extremely thin. The effect of inhibitors diminishes rapidly the further they are away from the metal surface. On the other hand, the barrier effect of the oil depends largely on the thickness of the film. Thixotropic oils counter the loss of barrier protection by retarding run-off. The stable film helps to maintain the original level of protection. As this level of protection cannot be achieved with conventional formulations, all of today's prelubes are thixotropic. The retarded run-off characteristics also prevent the soiling of press shop floors with oil as well as build-up of oil in swage beads and cavities which may cause problems with adhesive bonding or paint blisters during curing.

### 15.1.12.5 Washing of Steel Strips and Blanks

Automobile outer body panels often have to be washed because of cleanliness requirements. Advantages, however, are uniform oil film thickness, less die contamination and therefore low die wear. As washing, particularly blank washing, does not generate high costs when compared to the overall manufacturing process, it is economically acceptable.

The steel strips or blanks coated with mill oil are washed with one of various available washing mediums, e.g. water based emulsions, solvent diluted oils or low viscosity oils. When using emulsions, water related problems may occur such as corrosion of stamped parts and tools, wear pick-up and growth of bacteria and fungi. Careful maintenance and special precautions are therefore necessary.

Washing machines do not fully remove the oil from the panels. Depending on the washing fluid, about half of the oil is washed off and is then squeezed off with fleece rollers. The amount of oil remaining on the metal is influenced by the washing fluid and plant-specific parameters such as feed velocity and the number, diameter, material and pressure of the rollers. The residual oil film increases as the fluid viscosity rises. Lower lubricity systems might require additional drawing oils [15.31].

Washing fluids have to fulfil similar requirements to the mill-applied oils because they contribute to the total oil quantity entering the press and paint shops. Apart from offering adequate pressing lubrication, they should be easy to remove, compatible with adhesives and paints and supplement the corrosion protection offered by the underlying oil. As all washing oils obviously settle on top of the mill applied oil layer and are thus relatively far from the metal surface, they tend to run off quickly. For these oils to have thixotropic properties is therefore a significant advantage.

#### 15.1.12.6 **Additional Lubrication**

Many drawing operations require additional spot lubrication which is applied in the press shop. These higher viscosity drawing lubricants lose out when prelubes are applied. Ultimately, they will become unnecessary if the lubricity of the mill applied oil on the panels is adequate. In fact, some enormous savings are reported on by car makers. Long-term plant trials which were exactly documented were evaluated [15.32]. The savings resulting from the elimination of the drawing lubricant itself were only a part of the overall savings potential. The bottom line is improved by additional effects such as lower disposal costs and more stable manufacturing parameters. If in spite of the prelube, difficult deep drawing still requires the use of additional spot lubricants at critical points, these must be process-compatible. As such spot lubricants are again applied on top of the other oil layers, a thixotropic formulation is also advisable to avoid run-off.

#### 15.1.12.7 **Pressing**

The central manufacturing process is that which occurs during pressing. The extremely high investment and running costs of advanced, multistage presses make it essential that the pressing operation is consistent and reliable. Rejects caused by tears, dents or poor dimensional conformity, but also die cleaning, grinding or setting time can soon generate high costs. The large performance reserves offered by prelubes and the prelube-compatible washing oils used for outer skin panels can provide decisive benefits. Figure 15.23 illustrates the advantage of a prelube-type oil in comparison to a corrosion preventive mill oil on uncoated steel strip. Considerably lower friction combined with a smooth flow of material up to higher loads is achieved in the strip drawing test without deflection when the prelube is applied.

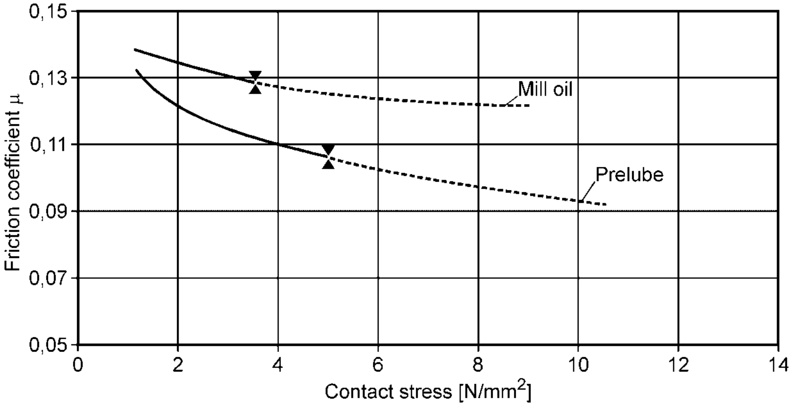

**Fig. 15.23** Friction coefficient vs load with type of oil as parameter, uncoated sheet metal/ 50 mm $s^{-1}$.

Because cold rolled steel strip for car bodies is to an increasing extent zinc-coated, the prelube needs to be as versatile as possible with regard to various metal coatings such as electro-galvanized, hot-dip galvanized, galvannealed or pre-phosphated sheet metal surface. Different lubricants do not necessarily display similar behavior on all coatings. An example is given in Fig. 15.24 where two prelubes are compared on different substrates. While the performance of both lubricants on uncoated sheet metal is quite similar, the coefficients of friction differ dramatically on zinc-plated steel [15.149, 15.150, 15.151].

Lubricants with optimum run-off characteristics are solid films known as dry film lubricants. However, the application of such products in the finishing lines of the rolling mills as well as problems in removing or corrosion protection have turned out to be the major obstacles for their widespread use up to now.

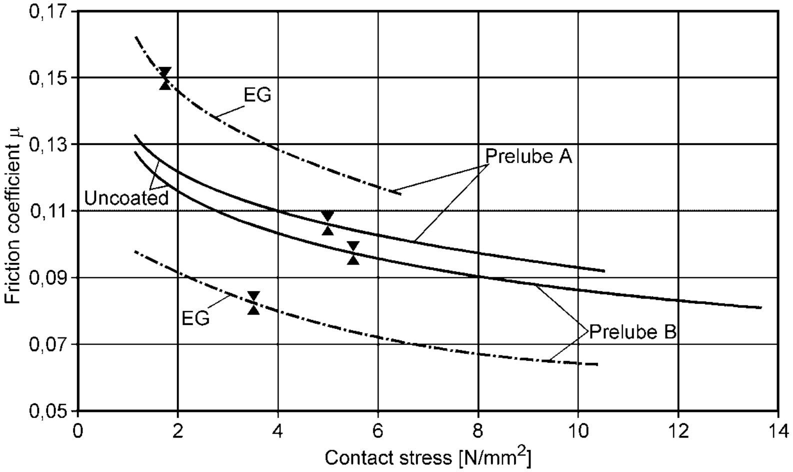

**Fig. 15.24** Friction coefficient vs load with type of prelube and surface coating as parameter, 50 mm $s^{-1}$.

#### 15.1.12.8 Transport and Storage of Pressed Parts

Pressed panels do not usually enter the manufacturing process immediately but are stored for a period of time. In-house storage does not pose any particular demands on the corrosion protection oil used. However, if the pressed panels have to be transported to other plants, overseas, through or into tropical regions, particularly high performance concepts have to be used. A high-performance, thixotropic prelube can make special protection oils unnecessary if it is combined with an intelligent packing concept and vapor phase inhibitors (VCI) [15.19]. The use of run-off-retarding products throughout the manufacturing process is especially worthwhile in the post-pressing stages because oil no longer runs off during transport and storage. This feature is emphasized by the lower film thicknesses of prelubes and prelube-type low-viscosity washing oils.

#### 15.1.12.9 Welding and Bonding [15.33]

Welding is not influenced by today's corrosion protection oils and pressing lubricants and therefore poses no particular requirements. However, this does not apply to the bonding processes used in body building because of the substitution of an increasing number of spot welds by adhesives and the increasing demands on the durability of such bonded joints. Of importance is the strength of the joint – up to its breaking point – but also its corrosion protection and its sealing properties. As oiled panels are bonded, the type and quantity of the oil, physical surface characteristics of the metal and naturally, the type and thickness of the adhesive effect the bonding result. As the automobile manufacturers use a number of different adhesives in their plants for different purposes and all combinations of metals, adhesives and application methods have to be compatible, the corresponding testing procedures are enormous. The formulation of a mill-applied corrosion preventive oil or prelube which has displayed optimum compatibility cannot be constantly adjusted to suit new adhesives, not least because it may be used by a large number of companies. As described, non-thixotropic or over-applied oils can run-off pressed panels and collect in beads and folds. In spite of originally positive compatibility tests, this can lead to poor bonding if the oil bead is too thick. Here again, the specification of run-off-retarding mill oils but also thixotropic washing and drawing oils can eliminate such problems.

#### 15.1.12.10 Cleaning and Phosphating

Oil which is trapped in cavities is difficult to remove with the normal treatment processes. Usually, the cleaner spray does not reach these points. Dip cleaners offer better performance with regard to removing oil from these inaccessible points but they foam excessively when sprayed. In spite of the use of spray and sprayed dip cleaners, components must be degreasable by simple dipping to remove the oil trapped in cavities. The oil must be easily removed from all surface qualities even after long-term storage or temperature induced aging during the pre-curing of body adhesives.

### 15.1.12.11 Cataphoretic Painting

Even though mill-applied oils can generally be excellently removed, small quantities being carried into the electrophoretic paint bath is unavoidable. The potential harm caused by such contamination can be lessened to a certain extent by special additives in the paint tank. The danger of serious, i.e. visible markings in top coats is best minimized by the use of the most compatible oils. A wide variety of contamination tests are performed to establish an oil's compatibility with electro-deposited paints. Usually small quantities of oil are emulsified in a paint bath. After storing, the size and number of craters in the cured paint film are evaluated. Excessive quantities of oil in beads, swages and other cavities resulting from run-off can also be expelled when trapped water and cleaners are boiled off in paint drying ovens. If the oil–water mixture is spattered onto wet and sensitive paint, serious paint irregularities can occur. In the laboratory, this effect is simulated by the oil spray or blow-out test. As opposed to the contamination tests, the results however cannot be quantified with figures but only by a relative evaluation.

### 15.1.12.12 Savings Potential using Prelubes

The use of prelubes in steel mills reduces the number and quantity of spot lubricants required for pressing operations. The savings potential of prelubes is not just the result of one less lubricant required. The true benefits are only available if the compatibility principle is applied throughout the manufacturing chain, beginning in the steel mill and up to the paint shop. It is therefore hardly worthwhile using a highly specialized product for every application at the cost of subsequent manufacturing stages. It is much more important to optimize all the manufacturing stages together. A suitable prelube offers the potential, but the biggest savings result from a more rational overall manufacturing concept. This would result in a far-reaching multifunctionality for the corrosion preventives and lubricants used for different applications, i.e. specific characteristics under the umbrella of overall compatibility. Today's prelube systems are therefore modular so that even different viscosities can be part of the same concept.

Since their introduction, thixotropic, run-off retarding oils have outperformed conventional oils. It remains to be seen if dry film lubricants offer additional advantages.

### 15.1.12.13 Dry-film Lubricants

There is still a growing demand for even higher-performing lubricants as high and highest-strength steels are increasingly used for sheet-metal forming. Complex part design in high-strength steel is a challenge for press shop managers making use of conventional mill oils and drawing lubricants.

As discussed in Section 15.1.12.1, the idea of combining the mill-applied corrosion-protection properties of a corrosion-preventive oil with the lubricity of a drawing oil led to the development of the prelube oils. Nevertheless, the film stability of thixotropic oils is not perfect and may be further improved if the lubricant is enhanced by incorporation of an additive with a semi-solid or waxy consistency. Oil leaking out of coils and running off pressed parts does not occur with firm films. There must, however, be no deterioration of other beneficial properties of the coating.

Automotive press shops have had much longer experience with drylube-coated aluminum strip than with steel strip. Water-based and hot-melt drylubes have been used on aluminum for some years. For reasons of compatibility, however, the members of the Association of German Automobile Manufacturers (VDA) prefer the same drylubes for both steel and aluminum; this precludes use of water-borne systems, because of their lack of corrosion protection. For optimum versatility, the drylube should be suitable for all sheet-metal qualities – uncoated steel or aluminum, hot-dip or electro-galvanized steel, pre-phosphated steel and composite material, for example polymer-coated sheet metal and double-bonded strip. The major requirements for the automotive sheet metal forming process are summarized in the VDA standard VDA 230-202 [15.141].

All drylubes have in common the solid state, or at least a waxy consistency, at ambient temperature. The lubricant must have a fairly low viscosity for ease of application to the metal surface, however. Solids or waxes are difficult to apply and are usually thinned before application [15.148].

For environmental and health reasons and the danger of fire, hydrocarbon solvents are no longer appropriate. Aqueous dispersions of wax or other solid organic compounds, for example polymers, have the desired liquidity. When the water has evaporated a wax or polymer film is formed. To accelerate this process, the freshly coated metal strip must be heated for curing; this is costly and sometimes impossible, because of limited space for the necessary heaters. Emulsifiers are also needed to disperse the product in water. This makes the finished film susceptible to penetration or removal by water; this can cause severe corrosion of steel substrates. Wax dispersions are, therefore, predominantly used for lubrication of aluminum or stainless steel for flat forming or tube hydroforming.

Apart from dilution, if a solid lubricant melts at a reasonably low temperature it may be thinned by heating, to enable spraying. This procedure is used when special waxes are used as bases for hot-melt drylube. The hotmelt is commonly applied by electrostatic spraying at the finishing lines of the rolling mills. Existing equipment can be used but must be adjusted to higher spraying temperatures. The hotmelt can, however, serve just as well as a drawing lubricant in press shops using specially designed spraying equipment for spot-wise application. Squeegee rollers can also be used if an homogeneous film is wanted. In addition to their major technical merits, ease of application is certainly a reason for the popularity of hot-melt drylubes in the sheet-metal-forming industry.

The most obvious requirement for a lubricant is that it be present as a homogeneous layer of adequate thickness in the zone of deformation. A thinly and evenly applied drylube meets this prerequisite almost ideally. The improvement in lubricity can be verified by strip-drawing tests. Figure 15.25 shows the graphs obtained when electro-galvanized (EG) sheet metal is drawn with 90° deflection. The lubricity of the waxy drylube film is a significant improvement even over that of the latest thixotropic prelube-type oils; this often enables cost-saving because of reduction of film thickness.

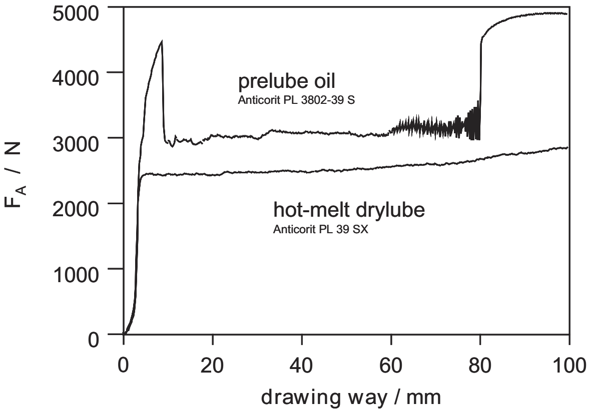

**Fig. 15.25** Results from strip drawing. The lubricant was 1.5 g $m^{-2}$ hot-melt drylube Anticorit PL 39 SX, the substrate was: EG, the die temperature was 40 °C, and the speed 10 mm $s^{-1}$, with deflection.

## 15.2
## Lubricants for Wire, Tube, and Profile Drawing

*Theo Mang and Wolfgang Buss*

### 15.2.1
### Friction and Lubrication, Tools, and Machines

#### 15.2.1.1 Forming Classification

According to DIN 8584 these forming processes comes under the manufacturing processes with forming under a combination of tensile and compressive conditions, or in closer sense to drawing by sliding action processes. Wire and rod drawing is applied to produce semi-finished products with smooth surfaces and close tolerances for the most varied of application purposes (Table 15.5).

**Tab. 15.5** Areas of application.

| *Material* | *Application* |
|---|---|
| Low-carbon steels | Wires, wire netting, nails, rivets, pins, screws |
| High-carbon steels | Wires and rods for the massive forming and free-cutting machining, wire ropes |
| Alloyed steels | Springs, welding rods, special components, steel cord for tires |
| Copper and copper alloys | Wires, pipes and components for the electronic industry, wire netting, screws and moldings |
| Aluminum and aluminum alloys | electric transmission lines, screws, moldings |

Solid or hollow bodies are generally drawn through a stationary drawing tool (drawing die, drawing nozzle, drawing ring) to reduce the cross section or change the cross section geometrically.

The drawing hole is the opening in these drawing tools.

The drawing of wire, in theory, is comparable with the drawing of round rod. When drawing both, rod with non-circular cross section and pipe, the basic knowledge of friction, lubrication and wear is employed for better defined round cross sections.

In the case of wire and profile drawing the sliding friction between the surface of the workpiece and the drawing hole surface is reduced by lubrication, but when drawing pipe with an inner tool the inner wall and inner tool also have to be lubricated.

Wire drawing in the case of the slip-type drawing process has outstanding economic significance. Typical lubricants and their application are also used for drawing pipe and profile and as a result are to be studied more closely in the following.

#### 15.2.1.2 **Friction and Lubrication, Machines and Tools when Wire Drawing**

In no other forming process the lubrication, the machine and tool technology are so closely associated with each other as in the case of wire drawing. We differentiate between dry and wet drawing, immersion and extrusion drawing machines and drawing dies for dry and wet drawing; three different types of lubrication technology are applied:

- *Dry drawing*: (in wire drawing this term does not mean the absence of lubricant but the use of solid, not liquid lubricants) with dry drawing soaps in the drawing box. These lubricants only lubricate the drawing die and hardly ever have a cooling function.
- *Lubricated drawing with pasty or high viscosity lubricants*: which means drawing greases, drawing oils or drawing emulsions.
- *Wet drawing*: Wet drawing is generally done with oil-in-water emulsions, seldom with non-water miscible oils, however, quite recently, also with special, highly viscous, degradable ester oils.

In wet drawing the task of the lubricant is to lubricate and cool the drawing tools and keep the machines clean. A further criteria for lubricant selection is the thickness of the wire.

##### Classification of Wire Drawing by Wire Thickness

National or international standards such as ISO DIS 22034-2 or ADDMA 8911 are applied for wire dimensions. Trivial classifications are also still in use. Where steel wire is concerned we differentiate between the following dimensions, for example in Table 15.6.

**Tab. 15.6** Dimensions of steel wire.

| *Wire* | *Diameter, d (mm)* |
|---|---|
| Coarse wire (special dimensions) | 40–15 |
| Coarse wire | 16–4.2 |
| Medium wire | 4.2–1.6 |
| Fine wire | 1.6–0.7 |
| Finest wire | < 0.7 |

A different classification is applied for copper wire as is shown, for example, in Table 15.7.

**Tab. 15.7** Dimensions of copper wire.

| *Wire* | *Diameter, d (mm)* |
|---|---|
| Coarse wire (special dimensions) | 20–12 |
| Coarse wire (rolled wire) | 12/8–3.5/1.5 |
| Medium wire | 3.5–0.4 |
| Fine wire | 2.1–0.15 |
| Finest wire | 1.0–0.05 |
| Super fine wire | 0.4–0.01 |

#### 15.2.1.3 **Drawing Force and Tension**

The cross section is reduced in the drawing hole (Fig. 15.26).

If, for example, a wire 1 mm$^2$ thick is drawn through a hole 0.8 mm$^2$ then $\varepsilon$ is 0.2 (or 20 %). If a section of wire is stretched from 1 mm to 1.2 mm, then $\lambda$ is 0.2 (or 20 %). At the same time this reducing operation means a considerable increase in the surface area. Accordingly, the sliding friction in the drawing hole takes place under these difficult lubricating conditions.

In the wire drawing machine the drawing force $F_Z$ is applied by a drawing disc (best known as cone) which causes the reduction in the drawing hole and overcomes the counter pull on the wire infeed side. Frequently the proportion of the drawing force related to the reduction in cross section is stated namely the drawing tension $\sigma_Z$. It can be well understood that the drawing tension must not be greater than the yield stress or the tensile strength of the wire so that the wire does not tear. To be able to work reliably at high drawing speeds the drawing tension must be clearly lower than the yield stress so that fluctuations in material properties and the coefficient of friction can be compensated. The drawing tension relationship has been defined to indicate the necessary safety allowance [15.35].

The Siebel formula is to be mentioned as a further theoretical issue, according to which the drawing tension is broken down into one part for forming without loss, one part for friction and one part for displacement (or shearing) [15.36, 15.37]. The

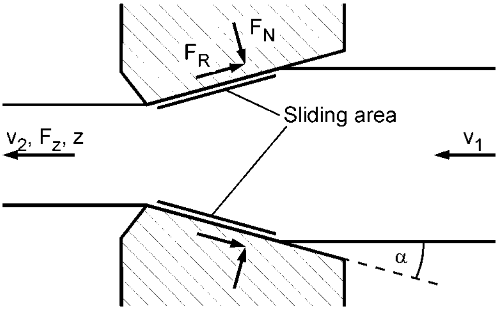

**Fig. 15.26** Friction and drawing tension when drawing wire. $v_1$ = initial speed; $v_2$ = final speed; $F_Z$ = drawing force; $F_N$ = normal force; $\sigma_Z$ = drawing tension; $F_R$ = friction force; $k_f$ = average deformation resistance; $\varepsilon$ = reduction of cross section; $\alpha$ = half reduction angle (drawing angle); $\mu$ = average friction coefficient.

$$\sigma_Z = k_f\left(\underbrace{\ln\frac{1}{1-\varepsilon}}_{\text{Forming}} + \underbrace{\boxed{\frac{\mu}{\alpha}\cdot\ln\frac{1}{1-\varepsilon}}}_{\text{Friction}} + \underbrace{\frac{2}{3}\alpha}_{\text{Shearing}}\right)$$

Siebel equation is given by ignoring the back pull. If the amount of friction is considered then this increases with the coefficient of friction μ and the reduction in cross section ε and declines with the drawing angle α. Since the drawing tension for proportion of displacement increases with the drawing angle, it can be seen that there must be an optimal angle α for the drawing tension. Not only the significance of the drawing hole geometry can be seen from this but also that a minimum in drawing tension over the drawing angle does not indicate a minimum of friction. The course of the drawing tension over the drawing angle is given by taking a brass material (Fig. 15.27) with the assumed prerequisites whereby the minimum is approx. 20°, i.e. the hydrodynamics are favored at this angle.

In the area of wire drawing, empirical values have had a greater influence on the optimization of the drawing hole geometry than theoretical issues. On the other hand in practice it is not the drawing force but the surface finish and especially the drawing die wear that is the prime consideration.

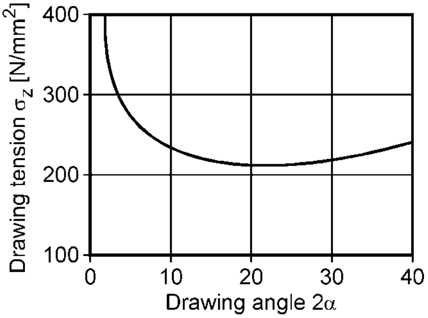

**Fig. 15.27** Dependence of drawing tension on drawing angle.

### 15.2.1.4 Drawing Tool and Wear

Drawing tools (drawing dies and drawing rings) are made of steel [ISO 1684], hard metal [ISO 1973], diamond single crystals or polycrystalline diamonds (PCD) [DIN 1546]. PCD layers consist of randomly oriented diamond particles sintered together in composite materials backed with cemented tungsten carbide.

Steel drawing dies are used especially for drawing steel rod and pipe drawing. Hard metal drawing dies are used mainly in the steel wire drawing sector. Smaller diameter wire is still almost only drawn with drawing dies made of sintered hard metal. Diamonds are used in the medium wire sector but above all for drawing fine and finest wires in copper, steel, molybdenum and tungsten. Pressure sintered hard metal alloys have proved particularly successful for wet drawing in the hard metal drawing die sector.

The porosity of normal-sintered drawing dies, as usually used in dry drawing, is detrimental to drawing and leads to unfavorable wear behavior. Pressure-sintered dies are a further advantage because they can be polished better.

Of decisive significance for the wear of natural diamonds, alongside the degree of inhomogeneity, is the crystallographic orientation [15.38]. Polycrystalline sinter diamonds [15.39] have gained significance for the profile drawing, for the medium wire drawing sector, and to some extent, also for drawing fine wire. Compared with single crystals these have the advantage of orientation-independent wear properties, are more resistant to impact and have higher heat conductivity.

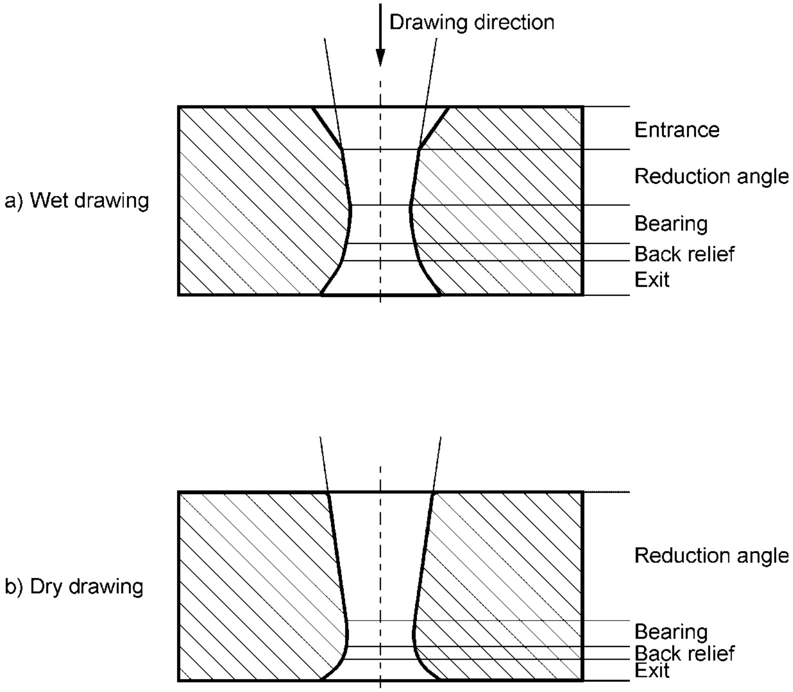

**Fig. 15.28** Drawing hole geometries.

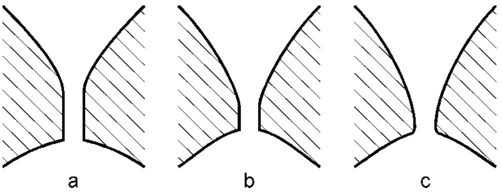

**Fig. 15.29** Drawing hole, shape of diamond dies. (a) shape used for hard wires (high carbon steel, stainless steel, tungsten); (b) shape used for copper wire; (c) shape used for soft wires (gold, silver, aluminum).

When designing the drawing hole geometry, the theoretical and empirical values of lubrication film development have to be considered as well as the specific criteria of different wire materials. The different drawing hole geometries of hard metal drawing dies (Fig. 15.28a) and diamond drawing dies (Fig. 15.28b) are generally used. In the case of hard metal dies a longer infeed entrance favors the hydrodynamic lubrication when using dry drawing soaps, even in the case of low drawing speeds (Fig. 15.29). Limit analysis of flow-through conical converging dies is known in detail [15.142]. General drawing die geometry details are found in DIN 1547.

In the case of thin wire, drawing die wear is used to assess the drawing lubricant as well as the number of wire cracks. The causes of drawing die wear are very complex in nature and depend on the influence of the machine engineering and on foreign substances which are carried in the lubricant or on the surface of the wire Fig. 15.30). Different forms of drawing die wear, which correspond with the different causes, are known, the extent of which also depends on the nature of the drawn material.

The possibility of the lubricant reducing wear is especially given in the area of boundary friction and the prevention of cavitation.

The service life of drawing dies is a significant factor for economic wire drawing. Giving the wire weight as a reference variable has become established practice in the coarse and medium wire sector despite comparable details only being available with exactly the same wire thicknesses. Typical figures for this are, for example, 70 tons copper wire with a final diameter of 2.50 mm and a drawing speed of 17 m $s^{-1}$, and the figure is 50 tons in the case of final diameter of 1.60 mm and 30 m $s^{-1}$ drawing speed. The expansion of the final drawing die is in this case max. 0.04 mm.

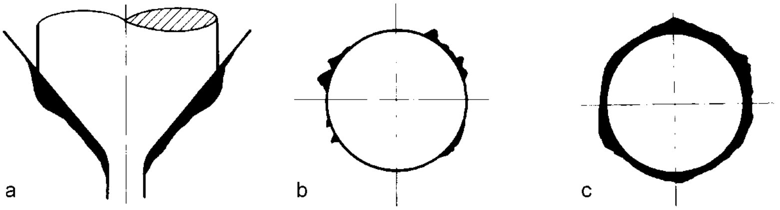

**Fig. 15.30** Modes of die wear. (a) annular wear, especially from soft materials; (b) grooving caused by hard particles in the drawn material or in the lubricant, cross section; (c) expansion of the die, roughened; in the case of diamond, single crystals change of shape, cross section.

A clearly better service life is achieved through the use of polycrystalline sinter diamonds, with re-polishing being necessary in each case after drawing 50 tons without widening occurring.

A more expedient reference value, namely the wire run-through length (that is to say the wear path) for the drawing die service life, is very seldom used. With good basic wire material, well set-up machines and good lubricating conditions, drawing die control and change, can be determined independent of diameter, for example, 12 000 km [15.40]. With a final diameter of 0.50 mm this means a drawing die capacity of 21.6 ton wire but only 1.92 ton wire with 0.15 mm dia.

#### 15.2.1.5 **Wire Cracks**

To assess the suitability of lubricants in the case of wet drawing of fine and finest wire, besides the wear phenomenon on the drawing dies and the quality of the wire surface, the frequency of wire cracks is also applied. There is a large number of causes of wire breaking of which only a few can be associated directly with the lubricant or the lubrication system such as, for example, deficient lubrication (applying too little lubricant or inappropriate lubricant performance) or the development of residue (inadequate rinsing and washing effect of the lubricant) in the drawing cone of the tool. On the other hand overrolling (rolling faults) or wire inclusions (impurities from the melts) cannot be influenced by lubrication.

#### 15.2.1.6 **Hydrodynamic Drawing**

There has been no lack of studies on the influence of hydrodynamic conditions on wire drawing. It can be assumed that the hydrodynamic friction in the drawing hole plays an essential role at high drawing speeds (Fig. 15.31). Nevertheless, the effect of polar and wear protection additives is of greater significance for the boundary lubrication conditions and to avoid stick–slip [15.41].

A number of changes has been made directly on the drawing tool or to the lubricating system to completely separate the wire from the drawing hole wall. However, as is also the case with other forming processes, inadequate or no contact between the workpiece surface and tool leads to a mat or even rough surface finish. As a result, the use of hydrodynamic drawing by sliding action is restricted to preliminary or interim drawing.

In the case of wet drawing the pressure of the lubricant can be applied in principle by pumps [15.42], which, in turn, results in considerable expense. In the case of liquid lubricants special wire infeed devices can be used while for dry drawing multiple pressure drawing dies can be used. Well-known infeed processes are the Christopherson method for wet drawing and Bisra for dry drawing soaps [15.43]. Figure 15.31 shows a pressure drawing die with infeed pipe for drawing under hydrodynamic conditions.

#### 15.2.1.7 **Wire Friction on Cone**

In single drawing machines the wire is drawn from the drawing drum through the drawing hole, but only sliding friction occurs in this case. As far as multiple drawing machines are concerned there is a cone between two drawing dies which, due to the

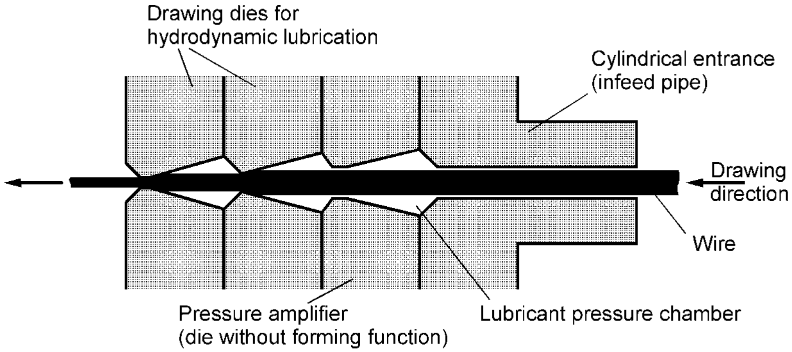

**Fig. 15.31** Triple stage pressure drawing die. Drawing under hydrodynamic conditions.

stretching of the wire has to turn faster than the cone in the proceeding drawing stage. The wire stretch can alter as a result of drawing die wear. This means that the speed of the cone has to be adjusted while drawing, or the changed drawing speed has to be taken up on the cone as collection wire.

As an alternative, the changing length of wire can be compensated by wire slip on the cone on so-called slip-type drawing machines. The slip occurs with the sliding friction between the wire and cone (Fig. 15.32). As a general rule, the greatest slip is on the first cone (up to 30 %) whereas slip no longer occurs on the last disc.

In the case of wet drawing the lubricant lubricates, cools and keeps the cone clean. The friction between wire and cone is taken up by the cone torque and as a result is concerted into drawing tension. The force available for pulling through the drawing hole is reduced by this, which is why the deformation per drawing stage by slip-type wire drawing is less then when compared with the non-slip method.

The transfer of the force to the cone is in accordance with the rope friction law (Fig. 15.33). The coefficient of friction must be adequate in size to be able to transmit force. No force can be transferred in the case of $\mu = 0$, i.e. $F_1 = F_2$. The smaller $\mu$ is the larger is $\alpha$. However, the number of wrappings is limited to ensure trouble-free running. With smooth coarse wires the number of windings can be as high as 7. In the case of super finest drawing, the numbers of coils can even be less than a full wrapping and the wrapping angle $\alpha$ must be set by special jigs, for example, to 300°.

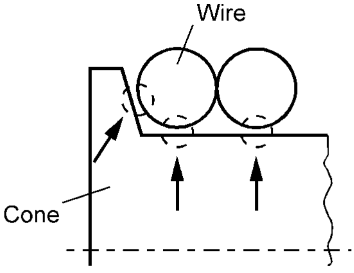

**Fig. 15.32** Contact between wire and cone–sliding friction on slip-type drawing machines.

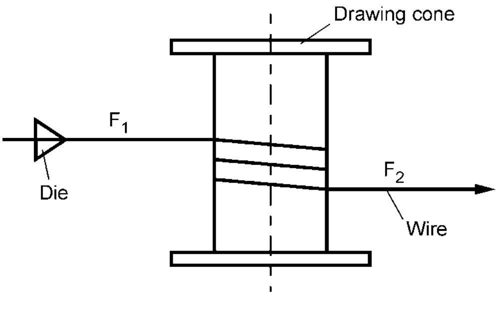

**Fig. 15.33** Chained forces before ($F_1$) and after ($F_2$) cone. The equation of rope friction is applied: $F_2 = F_1 e^{\mu\alpha}$, where $\mu$ is the coefficient of friction and $\alpha$ the wrapping angle.

Cone wear can quickly lead to disc failure if the same zones are continuously stressed. To avoid wear grooves the wire can be moved on the cone by pushing the drawing die to and fro. This results in a more even wear and a considerably longer service life for the cone.

The running surfaces of the cones, the so-called drawing rings (also called drawing cones) can be made of different materials, depending on the application. Metal drawing rings, for example, made of flame plated materials are used frequently when drawing copper wire on coarse wire machines. Generally, ceramic drawing cones are used for medium, fine and finest wire. Even compound constructions made of ceramic and steel play a role, especially with thicker medium wire. In the case of ceramic materials these are often aluminum oxide or zircon oxide with low average roughness ($R_a$ between 0.3 and 0.1 μm).

Slip-type wire drawing machine can be different in design and are not so complicated in construction. Non-slip type wire drawing machines can be operated without sliding friction on the cones but have to have a sensitive speed control system for the cones.

As a general rule, in the case of tandem arrangements the individual discs are mounted next to each other. In the case of cones of uniform size the speeds (or the angular speeds, $\omega$) are stepped according to the wire length. With machines working on the cone principle these are very compact in construction. On a third type, cones of the same size are mounted on a shaft but run via gears at different speeds. Compared with the cone system this type of machine has large cones which offer advantages for the surface of the wire, especially in the coarse wire sector because of the lower bending stress of the wire.

Modern wire drawing machines on the tandem or cone principle are designed as multi-wire machines, especially in the fine wire sector for copper drawing where up to 40 wires can be drawn in parallel. Thus a 24 wire drawing machine with 31 dies each wire holds 744 dies at the same time. In this case the application of the lubricants calls for the lubricant to be directed accurately to the required spot [15.44]. To be also considered are the increasing demands put on lubricants (for example, stability to aging, abrade carrying capacity, filterability, etc.). Moreover, an adequately large volume of lubricant must be available and provisions must be made for cooling where necessary. In the case of copper wire drawing, for example, the working volume of emulsion can fluctuate between 0.5 and 2.0 $m^3$ for each wire.

#### 15.2.1.8 Lubricant Feed in Wet Drawing

Depending on the type of machine, a choice (Fig. 15.34) is given between two basic principles, the immersion machines and the spraying machines, which are used more frequently today.

There are no consistent rules for lubricant delivery. The users proceed in many varied ways. Either the lubricant is supplied to a circulating central lubrication system or a separate circulation system is used for each individual machine.

Immersion machines provide advantages for thicker wire drawing or for particularly difficult to form materials with high heat development. Hollow draft can occur at higher drawing speeds over approx. 20 m $s^{-1}$. This can cause inadequate lubrication, especially when using lubricants which contain finest dispersed or dissolved air.

The cones are only partially immersed on special types of immersion machines (Fig. 15.34b). Only the drawing dies are flooded in other applications (for example with super-finest wire) not the cone (Fig. 15.34e).

#### 15.2.1.9 Dry Drawing

Dry drawing is applied most frequently for drawing steel wire. Dry drawing lubricants (also called drawing soaps, drawing stearates or drawing powder) are applied

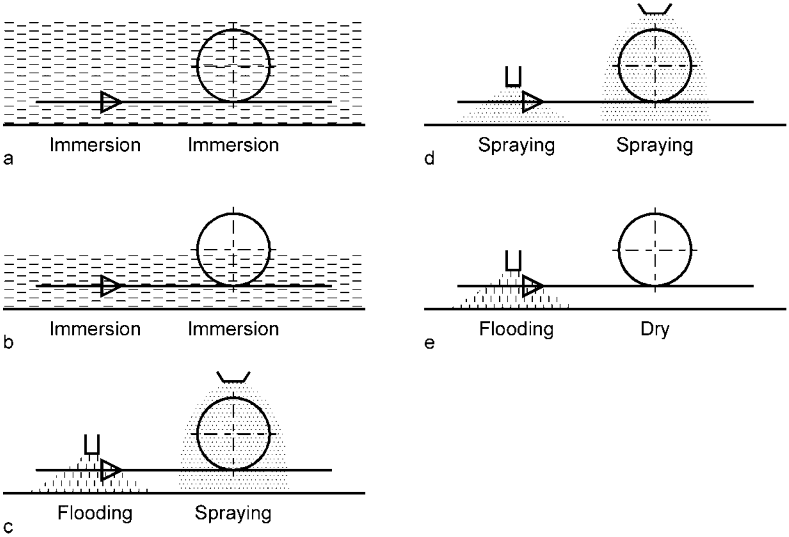

**Fig. 15.34** Lubrication feed in wet drawing–lubrication, cooling, and cleaning of drawing die and cone. (a) immersion–immersion, (b) immersion–immersion, (c) flooding–spraying, (d) spraying–spraying, (e) flooding–dry.

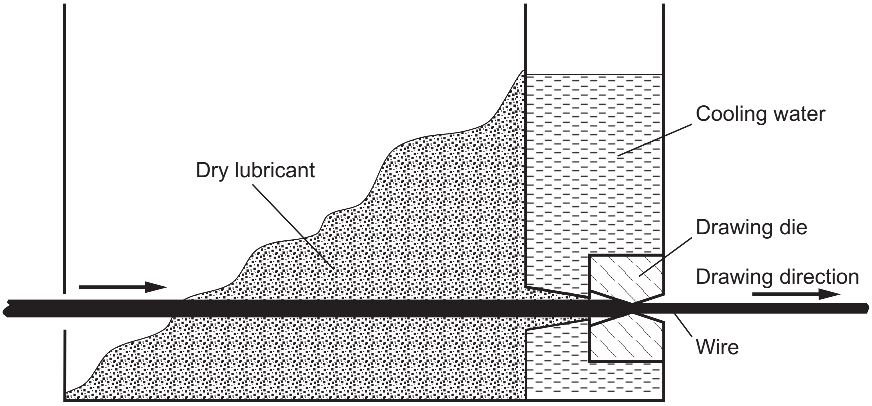

**Fig. 15.35** Dry drawing using a drawing box with die cooling.

as a general rule through drawing boxes (Fig. 15.35), which are located immediately in front of the drawing die. The applied lubricants must have the necessary powder fineness for the respective wire, which means it must be neither too coarse nor too fine in order to enable sufficient quantities to get into the drawing hole.

The granulation must be selected so that there is no tunnel forming. The drawing soap must neither melt during the drawing operation nor be baked but must allow easy layering and remain in motion and an optimal heaping angle must set in (Fig. 15.35).

Frequently, it is advisable for the drawing die, which is not cooled by the dry drawing lubricant, to be cooled by special devices, for example, by water cooling (Fig. 15.35).

#### 15.2.1.10 **Applying Lubricant as Pastes or High-viscosity Products**

The application of lubricants as pastes or as very viscous oils or emulsions follows, in circulating systems in so far as this is ensured by flowability and suitability for pumping. The drawing machine also has to be appropriately designed for this. Frequently, a drawing box is used for the lubricated drawing if no other devices are available, which causes sealing problems, in particular at the wire intake of the drawing box.

### 15.2.2
### Drawing Copper Wire

In the wire drawing of non-ferrous metals, copper wire drawing still plays an important role, especially because of its outstanding significance in electrical engineering – although copper wire has lost some of its significance in the wake of the introduction of glass fiber cable and has been replaced by aluminum wire in other sectors.

Copper wires are produced in final thicknesses between 20 mm and 10 μm. It is not possible to work with the same cross section reduction $\varepsilon$: coarse wire $\varepsilon \approx 25\%$, super finest wire $\varepsilon \approx 9\%$. This corresponds to wire lengthening $\lambda \approx 33\%$ for coarse wire and $\lambda \approx 10\%$ for super finest wire.

The production of copper wire with different tensile strengths is only possible through the forming process. It is not possible to quench and temper later as is the case with steel. This is why working with very different yield stresses is necessary in the case of copper, which means very different pressures in the lubrication gap. The tensile strength of hard or spring hard wire is > 370 N mm$^{-2}$ and 200 to 250 N mm$^{-2}$ with soft copper.

The basic wire material and its production have a great effect on friction, wear and lubrication when producing wire. An old, widespread method of producing rolled wire is based on cast billets. These are welded together and subsequently hot rolled. In the case of the Southwire method the molten copper solidifies in the groove of a casting wheel. The resulting copper rod is hot rolled directly afterwards (conti-process). In the case of the dip forming process a peeled copper wire is drawn through a copper belt and hot rolled afterwards, whereby the immersion and the rolling process is carried out in an inert gas atmosphere. Produced are wires with an oxygen content of less than 20 mg kg$^{-1}$.

Hot rolling leads to scaling of the wire surface with copper oxides, except in the case of dip formed wire. This oxide layer must be removed by pickling before the following drawing operations. Mixed acid is used for pickling. Wire with a mat surface is obtained, with a characteristic pickled profile and typical porosity. Smooth surfaces free of pores can only be reduced by wire peeling. Dip-formed wire on the other hand has a clean, smooth surface almost without pores right from the outset. It is used in an unpickled and unpeeled state for wire drawing and is suitable especially for further processing to produce fine and finest wire.

Because of the different basic materials there are also various application technology consequences concerning the lubrication of the drawn wire. As a result, the working requirements for the lubricant and lubricating system depend not least on the main qualities of the wire. Dip-forming wire, for example, demands a higher quality lubricant as a rule, or at least a higher application concentration.

#### 15.2.2.1 **Lubricants**

Water-mixed lubricants are used exclusively. Just in the first draft of coarse wire, a special lubricant of high viscosity is frequently used in the lubricated drawing or, alternatively, the concentrate used in a following wet drawing is used in a pre-drawing block to ensure more favorable lubricating conditions at the given low drawing speed. The lubricant used for this application must be compatible with the lubricant used afterwards.

Emulsions are the most important of all the lubricants in wet drawing. Apart from these, tenside solutions, free of mineral oil and fatty oil, are significant. To a small extent other synthetic solutions are also used. The three groups of lubricants (see Table 15.8) can be characterized on the basis of the most important ingredients.

**Tab. 15.8** Lubricants for wet drawing copper wire.

| *Type of lubricant* | *Ingredients* |
|---|---|
| Emulsions | Hydrocarbons, mineral oil |
| | Natural fatty oils |
| | Synthetic esters |
| | Nonionic surfactants |
| | Anionic surfactants |
| | Stabilizers, inhibitors |
| | Antifoam agents |
| | Other additives |
| Tenside solutions | Alkali soaps |
| | Alkali salt of sulfated fatty oils |
| | Nonionic surfactants |
| | Other additives |
| Other synthetic solutions | Polymers |
| | Organic salts |
| | Inorganic salts |
| | Other additives |

In practice these differ mainly through their degree of dispersion or their solubility properties in water.

The emulsions can vary from relatively coarsely dispersed emulsions with an average drop size of approx. 5 μm to the finely dispersed emulsions of an average drop size under 1 μm. These are colloidal solutions where soap and tenside solutions are concerned. The other synthetic solutions can be real solutions or partial colloidal solutions [15.45, 15.46].

The planned use of the drawn wire has an influence on the choice of lubricant, depending on wire thickness and material quality. The use of higher quality lubricants was a parallel development to higher drawing speeds and the greater demand for surface quality. In addition, greatest importance is being attached to cleanliness and enamel adhesion, particularly in the enameled wire sector.

Some 100 drawing stages, which have to be lubricated, have to be run through to produce 10 μm super finest wire from 8 mm thick coarse wire, whereby drawing speeds up to 50 $m\ s^{-1}$ are achieved. The mean drawing speed comes to 20–25 $m\ s^{-1}$.

Working with a standard lubricant for all stages is just as impossible as using a different lubricant for each stage. As a result, present day drawing plants only generally differentiate between two groups of wet drawing lubricants: lubricants for the coarse and upper medium wire sector and those for the medium and fine wire sector. Occasionally special lubricants are also used for finest and super finest wire.

### 15.2.2.2 Lubricant Concentration

Despite the many different types of lubricant, specific concentrations are applied according to the wire sector (Table 15.9). These concentrations are differentiated by their fat content in the conventional way.

**Tab. 15.9** Concentrations of drawing emulsions

| *Emulsion* | *Concentration (%)* |
|---|---|
| Coarse drawing | 12–18 |
| Medium drawing | 5–10 |
| Fine drawing | 3–7 |
| Finest drawing | 2–5 |
| Super fine drawing | 0.5–2 |

Very often one has to compromise when using central lubrication circulation systems with neighboring drawing thickness sectors, in order to keep the costs within limits. At least one tries to incorporate those drawing machines to a central supply unit on which certain dimensions are to be drawn despite different quality wire (peeled, pickled and dip-formed wire).

#### 15.2.2.3 **Solubility of Copper Reaction Products**

Monovalent and bivalent ions of copper are developed during the drawing operation which react with specific substances from the lubricant. Numbering amongst these are especially the oxidation products of hydrocarbon materials, fat substances and other ingredients. It is mainly copper soaps that are formed. Apart from this anionic surfactants react and copper reaction products are formed, likewise mainly soaps. The study of the emulsions used has revealed that the main quantity of copper reaction products are given in undissolved state and react hydrophobically and, as a result, are found in the oil phase of the emulsion. That is why the oil phase frequently is green in color. Reaction products also form in the water phase, which are generally blue in color and come under the copper amine complex salts. In all the copper reaction products only contribute to a very low extent to the conductivity of the drawing emulsion.

#### 15.2.2.4 **Water Quality and Electrolyte Stability**

Very frequently copper drawing emulsions are mixed with water of low hardness to avoid initial foaming problems with the fresh emulsion. Demineralized (deionized) water is used only in machines or central systems which generate very little foam in operation. A key value to assess the salt content and the degree to which salt can be added to the drawing emulsion is the electrical conductivity generally given in $\mu$S cm$^{-1}$ (micro-Siemens) or mS cm$^{-1}$ (milli-Siemens).

By making up the evaporation losses with the soft water the conductivity will not increases as quick as it does with hard water, while diluting the used emulsion with demineralized (fully deionized) water only a very slow increase of the conductivity is found. When the so-called limit conductivity is achieved the emulsion starts to become unstable. This value varies from lubricant to lubricant and depends, on the one hand, on the type and volume of electrolytes and, on the other hand, on the composition and the ingredients contained in the lubricant. Experience has shown that bivalent cations (mainly earth alkalis) destabilize more strongly than the monovalent alkalis. Where high electrolyte load and high emulsion temperature are concerned, a water-in-oil emulsion can form as a result of the floating oil, and this is irreversible. Together with copper abrasion a pasty mass is produced which contaminates the machines and circulation system and can only be removed with difficulty. Due to these facts lubricant manufacturers recommend fully demineralized water to compensate at least the evaporation losses in drawing emulsions. As a general rule, fully demineralized water is used for this application because of the necessary cleanliness of the wire surface for enamel wire production.

#### 15.2.2.5 Laboratory Testing Methods

There are no practice-relevant laboratory tests to assess lubricants in respect of their lubrication effect and protection against wear. Common test methods as listed in Table 15.10 are applied in principle to assess fresh emulsions and monitoring used emulsions. Also additional testing methods have been discussed without being observed generally [15.47].

**Tab. 15.10** Testing methods for copper drawing emulsions.

| *Property* | *Fresh* | *Used* |
|---|---|---|
| Concentration (fat content) | x | x |
| Emulsion stability after standing | x | x |
| Thermal stability | x | |
| pH value | x | x |
| Alkali reserve | x | x |
| Stability against electrolytes | x | |
| Electrical conductivity | x | x |
| Limit of electrical conductivity | x | |
| Foaming tendency | x | |
| Corrosion protection on iron | x | x |
| Staining on copper | x | |
| Dissolved copper content | | x |
| Growth of bacteria, fungi and yeasts | | x |

Generally the pH is between 7.5 and 9.0. Lower values lead to an unstable emulsion. On the other hand corrosion can occur on machines and pipelines, (at the worst pittings) because of the electrochemical difference in potential between copper and iron.

When assessing used drawing emulsions, the significance of the copper in the solution is disputed. Values between 500 and 2000 mg $l^{-1}$ Cu depending upon the lubricant, type of application and the drawn material can appear without drawing difficulties occurring. Even the conductivity of the emulsion is influenced to only a negligible extent by the dissolved copper content.

#### 15.2.2.6 Lubricant Temperature

Special cooling facilities are only required when the forming heat is not dissipated to an adequate extent by the environment, as is often the case when drawing coarse wire. Modern drawing emulsions are frequently used at temperatures from 30 to about 45 °C. Higher feeding-in temperatures lead to increased aging of the emulsion. Because of the influence of the drawing temperature on the friction conditions in the drawing hole and on the cones, many drawing plants have tempering facilities which control this process parameter and heat up the emulsion to application temperature after standstill periods.

#### 15.2.2.7 Influence of the Lubricant on Wire Enameling

Lubricant components must lead neither to wetting and running irregularities nor reduced adhesion. Whether a lubricant is suitable for the respective enameling process depends on gases of the thermal process, the temperature program and the type of enamel and cannot be laid down generally because of the large number of process parameters used. There are no laboratory testing methods which allow a reliable statement to be made. In other words, only a practical test can prove the suitability of a lubricant.

#### 15.2.2.8 Circulation Systems, Cleaning and Disposal of Drawing Emulsions

As already mentioned above, the available volume of emulsion per drawn wire has a great influence on the behavior and service life of the drawing emulsion. Low circulation counts (i.e. circulation of the total volume of emulsion per unit of time) permits effective cleaning of the emulsion because the major part of the copper wear particles is separated by sedimentation. In many cases hydrocyclones [15.48] or paper filters are also used. The limit to the separation of abrasion particles by sedimentation and in cyclones is between 25 and 10 µm. Paper filters have an effective filtration fineness between 10 and 5 µm. Also flotation became a particular method of separation of the copper particles out of emulsions [15.49]. Electrolysis of the drawing emulsion in bypass was applied in trial to gain a lower copper content by removing dissolved copper which was got back in metallic form [15.50].

The copper content makes the disposal of copper drawing emulsions difficult [15.143]. The copper contents have to be minimized to the stipulated extent by taking suitable measures before treatment or emptying into the waste water collection network, both in the separation oil phase as well as in the water phase. Evaporation, chemical or electrochemical separation methods can offer advantages, depending on location. Ultrafiltration allows the oil to be separated but cannot be applied as a general rule to ensure the limit value applicable for waste water. This is why expensive treatment is necessary in this case.

## 15.2.3
## Drawing of Steel Wire

### 15.2.3.1 Requirements

There are so many different demands put on finished wires and their production conditions that it is not possible to draw up a system which will observe all criteria. Used are all types of lubricant and lubricant materials as shown in Section 15.2.2: dry drawing, lubrication drawing and wet drawing. The selection depends upon very different criteria as can be seen in Table 15.11.

**Tab. 15.11** Criteria for the selection of steel wire lubricants.

Basic material (according to type and tensile strength)
Drawing machine
Finished wire thickness ranges
Wire pretreatment
Intermediate or final annealing
Required wire surface finish
Required tensile strength of finished wire
Further processing (coating, enameling, galvanizing)

Most steel wires are differentiated according to carbon content (low carbon content < 0.15 % C, high carbon content mainly between 0.4 and 0.7 % C).

The tensile strength of steel wire varies over a wide range depending upon application purpose and is, for example, 350 N mm$^{-2}$ for final annealed binding wire and 800 N mm$^{-2}$ in the case of pin wire. Drawn cold wire with high carbon content [DIN 668 K] for forming processes is annealed on spherical formed cementite to again achieve the original forming capacity. The tensile strength of rolled wire is between 130 and 260 N mm$^{-2}$.

Dry drawing calls for more extensive pretreatment to produce the carrying layer for the lubricant. Very often 0.5 mm finished wire diameter is used as the border between dry and wet drawing. In the case of special requirements (surface, tensile strength, suitability for galvanizing) this limit very often clearly moves upwards. There is also only a very unclear border between lubricated drawing and wet drawing.

In the case of finished wire diameters between 40 and 10 mm, very frequently single drawing machines are used, below that generally multiple drawing machines. For finished wire < 5 mm, for example, one starts with 5.5 mm thick rolled wire which is drawn over eight drawing dies, to 2.0 mm. After this it is possible, for example, to draw down to 0.7 mm in a dry drawing operation over 9 further drawing stages. The required wire tensile strength is achieved – depending on the initial heat treatment – by cold solidification when drawing. The required fine lamella perlite structure is achieved by a special heat treatment, the so called patenting.

If the strain hardening is too great, especially in the fine wire sector, intermediate annealing will be necessary.

The pretreatment of rolled wire after heat treatment involves the removal of scale and the application of a lubricant carrier layer. The scale is removed mechanically (for example by brushing) or chemically (by pickling). Chemically descaled wire has clearly more favorable wear behavior in the drawing die.

#### 15.2.3.2 Lubricant Carrier Layers

Applied lubricant carriers improve the adhesion of the lubricant, but, over and above this, have a real lubricating and separating function. These can comprise loosely absorbed or chemically firmly grown so-called conversion layers [15.51, 15.52]. The type of carrier and how it is applied depends upon the material and how difficult the wire drawing operation is [15.144]. Lubricant carrier layers are expensive to produce and are an essential cost factor, which is why they are only applied when this is absolutely essential.

#### 15.2.3.3 Lime as a Lubricant Carrier

Lime is applied as calcium hydroxide ($Ca(OH)_2$) from hot watery suspensions by immersing the pickled (or also phosphatized) wire coils. Apart from its tribological function, lime neutralizes the remains of acid from the pickle. Depending upon the required layer, the concentration is from 5 to 30 g $l^{-1}$ $Ca(OH)_2$. The applied layer thickness – which is frequently only achieved after immersing and drying several times – depends on the drawing stages to be run through and for thick wire is generally higher than for thin wire. To be noted at this point is that chlorides can increase in the lime baths as a result of pickling with hydrochloric acid while sulfates occur when pickling with sulfuric acid. There is a risk of corrosion in the case of a chloride content of > 80 mg $l^{-1}$ or a sulfate content > 150 mg $l^{-1}$ because the soap film remaining on the wire after drawing affords inadequate corrosion protection.

#### 15.2.3.4 Borax as Lubricant Carrier

The wire is immersed in solutions with 120 to 350 g $l^{-1}$ $Na_2B_4O_7 \cdot 10H_2O$, the decahydrate of borax [15.53], at 80 to 90 °C. The wire is then dried so that the pentahydrate $Na_2B_4O_7 \cdot 5H_2O$ is given, which has the best lubricating properties. In the case of steels with high carbon content, better results are achieved with borax than with lime. Since they are more difficult to apply, borax coatings are only used if possible phosphatizing can be replaced by adding additional lime.

#### 15.2.3.5 Phosphate as Lubricant Carrier

Generally zinc phosphate layers from approx. 3 to 7 g $m^{-2}$ are applied under the especially difficult conditions given when drawing steel wire. The phosphate layer is formed in a multi-stage reaction which can be shown in a simplified form as follows [15.54]:

Pickle attack: $Fe + 2H_3PO_4 \rightarrow Fe(H_2PO_4)_2 + H_2\uparrow$

layer formation in the absence of bivalent Fe ions (hopeite is produced):

$$3Zn(H_2PO_4)_2 \rightarrow Zn_3(PO_4)_2\downarrow + 4H_3PO_4$$

layer formation in the presence of bivalent Fe ions (phosphophyllite is produced):

$$2ZnHPO_4 + Fe^{2+} + 2H_20 \rightarrow Zn_2Fe(PO_4)_2\downarrow + 2H_3O^+$$

The classical combination of zinc phosphate coating, lime coating followed by dry drawing soap when drawing steel wire still plays a significant role today.

### 15.2.3.6 Oxalate Coatings and Silicates

Stainless steel cannot be phosphatized, only oxalatized. The reaction solution contains oxalic acid instead of phosphoric acid. Other main parts are ferrous salts, which serve as activators and accelerators. In simple form the reaction can be shown as follows [15.54]:

Pickle attack: $Fe + 2H^+ \rightarrow Fe^{2+} + H_2\uparrow$

Layer forming: $Fe^{2+} + (COO)_2^- \rightarrow Fe\ (COO)_2\downarrow$

The forming layers of oxalates not only contain ferrous oxalate but also the oxalates of other alloy partners of steel, such as nickel and chrome. The coatings from 3 to 20 g $m^{-2}$ thick are gray–white, green–yellow or dark green to black depending on the alloy and the thickness and are rather more fine crystalline. They are only suitable for cold forming.

Silicates (for example, water-glass) are seldom used mainly because of the possible formation of abrasive substances which cause problems in the following wet drawing operation.

#### Metal Coatings

Very often the decorative effect is the main consideration. Over and above this copper in particular makes a contribution towards the tribology of the wire drawing operation in that it functions as an effective lubricant carrier layer together with the liming. The coating is applied to copper by immersing the wire in an acidic copper salt solution. In the case of stainless steels the copper coating is applied by electrolysis.

### 15.2.3.7 Lubricants for Steel Wire Drawing

The same basic technical requirements apply for wet drawing as is the case for copper wire drawing. The thicker the wire is for drawing, the greater the need for effective lubricating polar and EP substances. This applies for the emulsions used in the fine wire sector as well as for the drawing oil in the medium wire section.

Occasionally pasty drawing greases containing fillers are used in the lubricated drawing before wet drawing. The combination of semi-plastic lubricant carrier layers with circulating oil lubrication is also a type of lubricated drawing [15.55]. However, the major volume of steel wire in coarse and medium draft are drawn with dry drawing soaps.

Chemical structure of dry drawing soaps: here one always speaks about stearates, despite the actual composition. However, the percentage of soap is not based solely on stearic acid as the basic material but can contain all possible vegetable and animal fats in saponified form.

Depending on the type of soap, alkali, earth alkali and earth metal soaps are the contents whereby sodium, calcium and aluminum soaps have the most significance. The soap content of the stearates can be between 20 to 80 %. Used, of course, as further substances in the content are natural and synthetic (under certain circumstances also chlorinated) waxes, polymers, inorganic solid substances such as lime, borax, soda, talcum, titanium oxide as well as molybdenum disulfide, graphite and sulfur. Generally earth alkali and earth metal soaps are not dissolvable in water, but on the other hand sodium soaps are very soluble in water. This is why alkali stearates can be also applied by immersing the wire coil in the hot watery soap solution with subsequent drying, besides being used in the drawing box.

The properties of the soaps and the other substances [15.56] are also decisive for the behavior of the lubricant residue on the wire when annealing and degreasing. This is why the lubricants for wire drawing are selected not least with this in mind. For these reasons drawings soaps free of ash in combination with substances free of annealing residue are already used in individual applications [15.57].

Dry drawing at high speed calls for lubricants with high melting soaps due to the high development of local heat. Generally, stearates have a higher melting point than oleates (Table 15.12).

**Tab. 15.12** Melting points of fatty acid salts.

| *Stearate* | °C | *Oleate* | °C |
|---|---|---|---|
| Calcium | 167 | Calcium | 123 |
| Barium | 212 | Barium | 145 |
| Sodium | 253 | Sodium | 169 |

Nevertheless, technical drawing soaps are not only selected according to their melting point. More important are generally the lubricating properties, the matching grain size distribution, the flowability in the drawing box, the protection against wear for the drawing dies, the further processing of the semi-finished products and the corrosion protection, etc.

Hydrodynamic lubrication is gaining more and more significance. An apparatus can be used for measuring the rheological behavior by which the dry drawing soap is stressed in reverse flow extrusion [15.58]. An apparent viscosity of 3000 Pa s is given for calcium stearate under the conditions provided by this method of testing, but only 200 Pa s for sodium stearate. Even extrusionmeter measurements confirm the experience made with calcium stearate and determine that it is more suitable for wire drawing. Drawing force measurements are also used as assessment criteria for lubricating properties [15.59].

In practice, it is the grain distribution and flowability of the dry drawing soap which are decisive for lubrication. Coarse grains flow better than fine grains but are not so easily picked up by the wire. In Germany one classifies grain distribution into coarse (> 0.8 mm), medium (0.8 to 0.2 mm) and fine (< 0.2 mm). The degree can be determined by screen analysis. The resistance of a drawing soap to abrasion is also important. It depends upon the composition, production process and the milling and must not be too high so that the wire can take enough lubricant with it. The incoming wire has a grinding effect as a result of which the drawing soap circulating in the drawing box (Fig. 15.34) becomes continuously finer in the course of time and has to be punctually replaced before tunneling occurs.

### 15.2.4
### Drawing Aluminum Wire

The focus in aluminum wire production is on conductor wire for cable and overhead power transmission lines. For this purpose wire is produced in coarse draft between 5.0 and 1.66 mm in diameter. However, aluminum has been able to replace copper wire to some extent in other sectors over the past three decades. Available as basic materials are cast rolled wire, for example, produced by the Properzi method or continuously extruded wire, with initial diameters of 15, 12 or 9.5 mm. The tensile strength of the starting wire is between 70 and 130 N $mm^{-2}$. Wire faults at the beginning, such as rough surface overrolling and foreign inclusions, have a negative effect on the tribological processes in the drawing hole and can cause wire cracks and increased drawing die wear.

#### 15.2.4.1 Drawing Machines and Lubrication

Single drawing machines are now only used for special dimensions for smallest batches. The main volume of aluminum wire is produced today by slip-type wet drawing machines with non-water miscible drawing oils. These can be designed as immersion or spraying machines (Fig. 15.33). The drawing speeds, depending upon the final size, are between about 25 and 50 m $min^{-1}$ and, experimentally, at much higher speeds. For example, a rolled wire 9.5 mm in diameter is drawn in eleven drafts (with declining cross section reduction of 32 to 22 % per drawing stage) to 1.6 mm.

Pressure sintered hard metals, especially tungsten carbide, are generally used as drawing dies.

The drawing angle $2\alpha$ is between 18 and 24°. In the case of aluminum wire drawing the problem of heat development is greater than for copper because non-water lubricants are used. A drawing machine working in the most important diameter range for aluminum uses for example 60 $m^3$ $h^{-1}$ drawing oil which, with a circulation factor of 5 $h^{-1}$, means a lubricant volume of 12 $m^3$.

An oil temperature of 40 to 45 °C is considered to be optimal which is why heating (at start up after longer standstill period) and cooling of the lubricant are necessary to maintain a constant oil temperature.

#### 15.2.4.2 Lubricants for Aluminum Wire Drawing

To some extent, drawing compounds with a high content of drawing effective (polar) substances is still found in the non-slip single draft. Otherwise almost only very age-stable non-water drawing oils in a wide range of viscosities are used. Drawing coarse wire the oil viscosities can be between 300 and 1000 $mm^2\ s^{-1}$ at 40 °C, in medium wire drawing between 50 and 100 $mm^2\ s^{-1}$ and in finest and super-finest wire drawing oils with very low viscosity are frequently used, for example, 2 to 10 $mm^2\ s^{-1}$ at 20 °C. The higher the viscosity, the more important preheating the oil becomes at start-up.

In the case of aluminum wire drawing a very fine abrasion particles occur. Normal oil filtration, as it is used in other applications, faces great difficulties in the case of aluminum wire drawing so that the particles can normally only be removed by sedimentation at stand-still. To this must be added that the drawing-effective additives in the lubricant develop aluminum soaps which increases the viscosity of the oil. Therefore, the consumed lubricant is often supplemented by lower viscosity oils. Altogether in this way very long oil service lives (as a general rule several years) are achieved.

### 15.2.5
### Wire from Other Materials

By volume steel, aluminum and copper clearly play the most essential role in wire drawing. Special conditions are necessary for wire made of other elements and alloys, depending on the possibilities given in the wire drawing shop, the material-specific conditions in pretreatment and the availability of special lubricants. This is why this subject cannot be reviewed systematically.

#### 15.2.5.1 Stainless Steel

In the case of this material, which is becoming increasingly important, oxalate coating and also plastic films, for example from chlorinated polymers or lead layers are applied as a lubricant carrier instead of the oxalation or liming for coarse wire drawing. Lubrication in the drawing dies can be provided by dry drawing soaps or with EP oils (highly additivated, generally containing chlorine) [15.55].

Medium and fine wire is drawn with EP oils, finest wire also with water-miscible lubricants.

Chlorine and lead compounds cause considerable environmental problems. This is why EP oils, free of chlorine, have been used successfully in recent times, for example, in combination with an electrolysis-applied copper plating.

#### 15.2.5.2 Nickel

This material behaves in a similar way to stainless steel. Coarse wire with sodium stearate is drawn, for example, over seven slipless drawing stages from 5.5 to 2.0 mm, medium and fine wire from 2.0 to 0.6 mm and from 0.6 to 0.1 mm with non-water miscible EP oils which must be free of corrosive sulfur. During the annealing (> 800 °C in reducing atmospheres) the lubricant residue must not have a detrimental effect on the nickel surface.

#### 15.2.5.3 Tungsten

Tungsten wire, which is required for the production of incandescent bulbs, is produced by a special method. First, a 3 mm thick wire is produced from sintered tungsten or a single crystal tungsten by round forging. This wire is then subsequently drawn through hard metal drawing dies at a temperature between 800 and 950 °C, or at lower temperatures in the fine wire sector. Fine wire, approx. 60 μm in diameter, is treated at about 180 °C in the oxidizing milieu and finally drawn to approx. 20 μm. Watery dispersions of colloidal graphite [15.60] are used as lubricants. In the case of tungsten wire drawing the oxide layer serves as a lubricant carrier and improves the adhesion of the graphite [15.61, 15.62].

### 15.2.6
### Profile Drawing

In the case of wire and rod drawing the preliminary materials will normally have a circular cross section. Where profile drawing is concerned, an extrusion molded, generally hot rolled initial material is available which already has a similar profile to that of the finished material.

As for wire drawing, the method comes under push–pull forming and from a tribological point of view is to be treated very much in the same way.

Profile drawing is dominant in all kinds of steel working but has much less significance by volume in non-ferrous metals. The different tribological degrees of difficulty in the case of profile drawing is found on the one hand in the cross section reduction, in the difference between the initial and final geometry of the profiles, and last but not least, in the forming of corners and edges with very close radii. Accordingly the tribological most difficult and manifold area is the drawing of special profiles. Figure 15.36 shows cross sections suitable for lubricant assessment by analytical description of the geometries [15.63].

The profiles are drawn on drawing benches, to some extent at > 150 m min$^{-1}$, with drawing carriages which are moved by chains, ropes, toothed racks or hydraulic plungers. There are single and multiple drawing carriages available. Drawing benches have been developed for continuous drawing with drawing carriages arranged one after the other with clamping jaws which take over the drawing function one after the other.

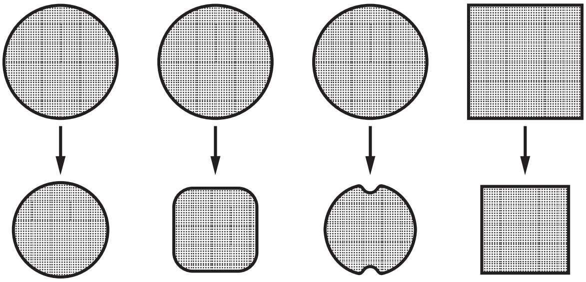

**Fig. 15.36** Profile drawing – drawing steps for lubricant assessment.

#### 15.2.6.1 **Lubricating Tasks in Profile Drawing**

During cold profile drawing, a high precision workpiece has to be produced from a geometrically inaccurate initial material. As far as the majority of special profiles is concerned, the aim is to produce surfaces with specific set functional properties, for example, which favor the technical flow properties of turbine propellers. To achieve the required quality of the profile the main task in this case the function of the lubricant used is not to reduce tool wear but to meet the profile form and surface criteria. Moreover, the lubricant must contribute to achieving a high drawing speed and very frequently it must lead to the drawing force being reduced in order to achieve the required result in as few drawing stages as possible. However, it must be considered that as the lubricating film thickness increases, the drawing force decreases, but, on the other hand, the quality of the surface finish also decreases [15.64].

The drawing tools for profiles for high batch production frequently consist of low wear hard metal materials although tool materials, less resistant to wear can be used for small batch production.

#### 15.2.6.2 **Pretreatment and the Use of Lubricant when Profile Drawing Steel**

The basic materials must enable chemical descaling (pickling) or mechanical descaling (blasting, descaling, brushing). The same pretreatment methods are applied as is the case with steel wire drawing: liming, phosphatizing, boraxizing, oxalatizing, etc. (see Section 15.2.3). Phosphatizing is frequently not used, should the surface have to be treated later, in order to save the dephosphatizing operation. Dry drawing soaps in drawing boxes or even water soluble drawing soaps (applied by immersing the preliminary material in sodium stearate solution, for example) are used for lubrication. However, oil circulation lubrication has the greatest significance for completely flooding the intake side of the drawing tool – not least because of the easier insertion of the workpiece in the drawing hole during discontinuous working. The drawing oils have a viscosity of 10–200 $mm^2\ s^{-1}$ at 40 °C depending upon the application but most frequently between 50 and 120 $mm^2\ s^{-1}$. To be noted in this respect is the thickening frequently observed, for example, by lime entering from lime coatings.

Drawing oils with a high percentage of additives are used depending upon the degree of difficulty. In this case the same selection criteria are valid as in the case of wire drawing. Drawing oils containing chlorine are generally removed before intermediate annealing because of the development of corrosive gases. Special emulsions containing solid film lubricants and drawing grease are suitable for particularly polished drawn surfaces.

### 15.2.7
### Tube Drawing

The great significance of tube drawing becomes clear when one considers that about one seventh of the steel produced worldwide is used for the production of seamless or welded tube. Moreover, the production of tube made of stainless steel, titanium, aluminum and especially in copper has considerable significance both, commercially and technically.

Generally tubes are used to conduct liquids, vapors, gases and phase mixes. Steel tube and quite recently even aluminum pipe has gained considerable importance as a construction element and, for example, are processed by means of internal high pressure forming [15.65] (hydroforming) to make automobile components (axles, cradles, engine supports, frame parts et cetera).

#### 15.2.7.1 **Tube-drawing Methods**

Wherever possible tube is produced by casting or forming methods in the required dimensions and tube quality. Precision tube with defined, closer tolerances, especially with defined surface finishes, can only be produced by cold forming. Cold drawing of tube, apart from other cold forming methods such as cold pilger rolling or cold rolling, are the last link in the production chain and, as a result, is to be covered in this section. As is the case with profile drawing, tube drawing is performed out on drawing benches (see Section 15.2.8). Long lengths of tube, especially copper tube, are drawn on drum drawing machines which in principle work in the same way as single wire drawing machines. Figure 15.37a shows the working principle of a drawing bench and Fig. 15.37b that of a drum drawing machine. The permissible form changing limits are given by the required drawing force to be transferred after the change in form to the given tube cross section. This must not exceed the tensile strength force of the cross section.

#### 15.2.7.2 **Tools and Tool Coatings**

With regards to the tribological demands, the drawing method is the primary factor [15.67]. Shown in Fig. 15.38 are the most usual methods: in the case of hollow drawing without an inner tool, only external friction occurs and as a result only lubrica-

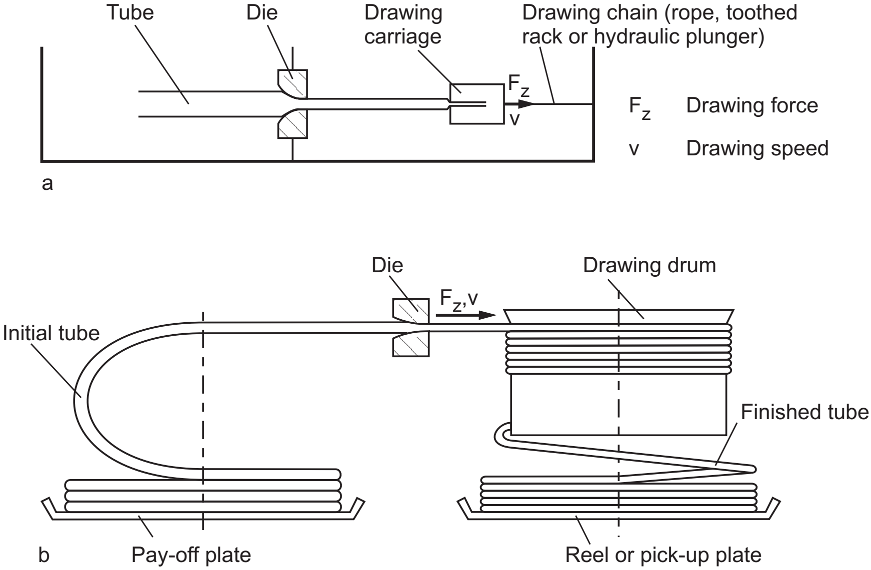

**Fig. 15.37** Working principles of tube drawing [15.66]. (a) drawing bench, (b) drawing machine.

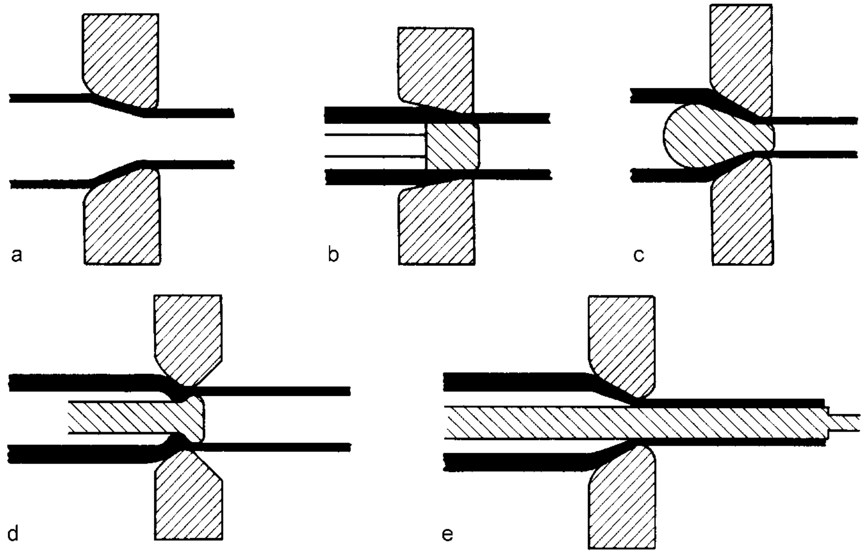

**Fig. 15.38** Drawing procedures for tube manufacture. (a) hollow drawing: no inner tool, friction and lubrication from outside only; (b) mandrel drawing using stationary mandrel (fixed plug): friction and lubrication on the outside and inside of the die; application of lubricant can take place through the tubular mandrel rod (see Fig. 15.39); (c) drawing using flying mandrels: used in particular in long tube length on drum drawing machines; a defined amount of lubricant is applied manually before drawing; (d) drawing using crown mandrels: high surface pressure resulting in difficult friction and lubrication conditions; (e) rod drawing: the inner tool (rod) moves with the same speed as the finished pipe; a large amount of friction is needed to move the pipe; the highest speed between the pipe and the rod is given by $v_P = v_R(A_0 - A_1)/A_0$, where $v_P$ is the pipe speed, $v_R$ the rod speed, $A_0$ the cross section of the finished tube wall, and $A_1$ the cross section of the initial tube wall.

tion from the outside is necessary. In the case of thin walled tubes, the walls become thicker but where thick walled tube is concerned, the thickness is reduced by up to 35 % per draft. In this case the inner surface loses its initial quality. In the case of various mandrel drawing processes a considerable improvement in the inner surface finish and a defined wall thickness are achieved with a cross section reduction per draft of up to 50 %.

As a general rule flying mandrels are only used in conjunction with drum drawing machines, especially with non-ferrous metals. The continuous application of lubricant for the inside is possible by means of the mandrel rod [15.68] where mandrel drawing is concerned (Fig. 15.39) The most difficult situation is given, from a tribological point of view, when drawing with crown mandrels, because of the high surface pressure. On the other hand, very smooth surface finishes are achieved. The drawing tools used for pipe drafting are mainly made of hard metal and are similar in structure to those for wire drawing [drawing dies pursuant to DIN 1547-2, drawing mandrels pursuant to DIN 8099-1 and DIN 8099-2].

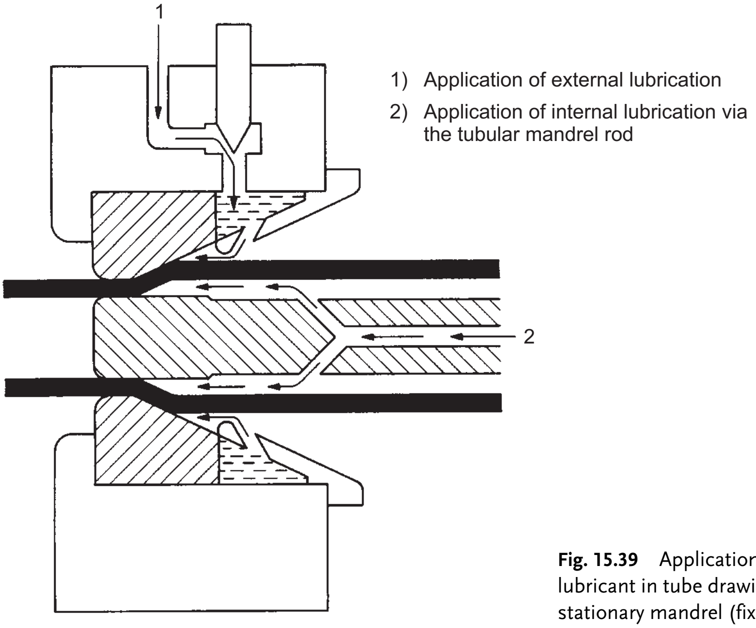

**Fig. 15.39** Application of lubricant in tube drawing with a stationary mandrel (fixed plug).

The advantage of the high reduction in cross section given when rod drawing (> 60 %) is set off by the disadvantage that the inner tool (rod) has to be removed again from the pipe.

There have been similar advances in the development of tool technology [15.69] as in other sectors over the past years. In modern production engineering the introduction of CVD and PVD coating techniques and the introduction of new tool material have contributed most to this development due to their extraordinary tribological aspects, especially concerning heavy duty forming dies [15.70].

#### 15.2.7.3 **Lubricants and Surface Pretreatment for Tube Drawing**

The pretreatment methods are comparable in principle with those used for wire and profile drawing.

**Steel tube**

The scale is removed in all drawing processes, except occasionally in the case of hollow drawing, for example, when drawing thin-walled scaled tube where little importance is attached to surface finish. However, mechanical scale removal in tube drawing is not as important as is the case with wire, rod or profile drawing because of the undesirable surface strengthening. Generally, descaling is by means of sulfuric acid (pickling) or annealing under inert gas by phosphatizing or zinc phosphatizing and even oxalatizing in the case of high alloyed high-grade stainless steels (cf. Section 15.2.3).

Wet drawing with EP oils does play a role for less difficult forming operations, as is the case with profile drawing to produce blank drawing.

A special form of wet drawing is carried out with so-called reactive forming lubricants. These oils contain organic and inorganic derivatives of phosphoric acid applied initially in a heated immersion bath. For example, bundles of tubes are immersed after pickling or inert gas annealing for some 10 to 15 minutes in heated reactive oil at 40 to 70 °C where they are left a certain period of time before being drawn with the residual oil film. The phosphate layer remains as a rule on the pipes. Although the ferrous phosphate coating produced in this way cannot be stressed to the same degree, tribologically, as zinc phosphate coatings, or even saponified zinc phosphate coatings, the method has gained very strongly in importance for cost reasons. The ferrous phosphate coating also provides good corrosion protection. High alloyed materials as specified for rod and profile drawing are drawn with special highly viscous oils with chlorine additive (300 to 1000 $mm^2 s^{-1}$, with a Cl content from 30 to 70 %). In this case the inner lubrication frequently turns out to be a problem. Apart from these, certain polymer and cellulose ether films have proved to be suitable.

Emulsion and water mixed drawing greases have only certain significance when blank drawing with a low degree of deformation.

### Titanium Tube

These are important as capillary tubes and for pipeline construction in the aircraft industry. Lubricants containing chlorine have good drawing properties which, however cannot be used for applications in aircraft construction because of the risk of crack corrosion. The suitability of titanium for phosphatizing is considerably less than that of steel. Tribologically effective coating calls for multi-stage treatment with acid. In this case there is a risk of hydrogen embrittlement.

### Aluminum Tube

The same working principles apply as for drawing aluminum wire. Strong polar forming oils are used mainly for this.

Working with phosphate coatings is also possible when drawing tube made of aluminum alloys which, for example, are used as a basic material in cold extrusion. In this way it is also possible to draw difficult aluminum alloys. The phosphatizing baths for aluminum are similar in construction to those for ferrous materials, but contain a certain amount of fluoride ions which are required for masking troublesome aluminum ions. The following simplified reaction equations [15.71] are valid:

Pickle attack: $4Al + 4H_3PO_4 \rightarrow 4AlPO_4\downarrow + 6H_2\uparrow$

Layer formation: $3Zn(H_2PO_4)_2 \rightarrow Zn_3(PO_4)_2\downarrow + 4H_3PO_4$

The pretreatment of the aluminum surface before phosphatizing calls for a high degree of cleanliness. As opposed to the case with ferrous materials, cleaning is frequently in alkaline media which attacks the aluminum specifically so that the oxide skin is carefully removed. The remaining oxide skin is then removed during the pickling operation.

**Copper Tube**

Copper tube has gained outstanding significance in the refrigeration and air conditioning installation branch. Particularly rational drum drawing machines (see Fig. 15.36b) with high drawing speeds (up to 35 mm $s^{-1}$) are used with flying mandrels. The required quantity of lubricant is supplied by means of grease filler for inner lubricating. Oil circulation lubrication is used for the outside, if necessary, also with emulsions (frequently 30 to 40 %).

The following single steps are an example of the working conditions for the production of bulk pipe for air conditioning technology with the most essential final diameter of 9 mm:

- rolling a continuously extruded tube with an outer diameter of 40 mm with 1.9 mm wall thickness with flying inner tool, internal grease lubrication and external emulsion lubrication (10 %).
- drawing in seven to eight stages on drum drawing machines at 9 mm diameter and 0.3 mm wall thickness without intermediate annealing. Inner lubrication with strong polar drawing grease, external lubrication with a high viscous synthetic drawing oil comprising polar and non-polar proportions and a viscosity of > 500 $mm^2$ $s^{-1}$ at 40 °C; 300 l oil tank, oil circulation rate 25 l $min^{-1}$.
- final annealing at 600 to 650 °C over a period of 20 to 30 min. The drawing oil must be evaporated so that no residue is left (thermal degreasing).

### 15.2.8
### Hydroforming

The full name of 'internal high pressure forming' commonly is replaced by the term 'hydroforming'. This process covers a very innovative technological field and comes under the manufacturing processes using fluids as forming media.

Generally the internal high pressure processes can be divided in internal high pressure forming, internal high pressure cutting and internal high pressure jointing. The installation of hydroforming process facilities require specific engineering [15.72].

First applications using internal pressure are known since more than 100 years for the bending of tubes. In the sixties the production of T-fittings became a typical application. Then exhaust components, motor cradles and other structural components for automobiles were manufactured. In the past ten years the hydroforming technology grew considerably and it will gain more and more importance due to the general demands in saving energy and raw material cost, due to improved mechanical equipment and process control, due to replacement of complex structural components manufactured in short process chains (often with additional operations integrated in the hydroforming tools) and due to the contributions of an intensive research [15.73]. The application area for the time being was based on standard tubular pre-products. Meanwhile even tailored tubes having different materials or wall thicknesses can be processed [15.74].

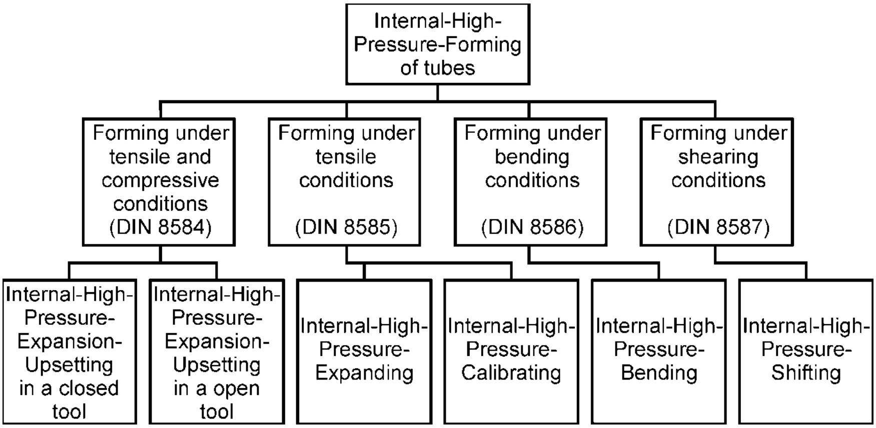

**Fig. 15.40** Process variants of internal high-pressure forming. (Source: VDI-guideline 3146 [15.77].)

In more recent time another application field is developing, the hydroforming of sheet metal: Two blanks are being formed simultaneously [15.75, 15.76, 15.145] where the pressure fluid is applied through a docking system – a special type of fluid-media-based deep-drawing.

A classification of the hydroforming process-variants is given in Fig. 15.40 [15.77]. The different variants show the steps or stages of the hydroforming process. Of course whenever possible several variants are combined in one process step.

The application of the hydroforming technology caused new constructive solutions in several areas of metalworking industry.

Favored objects of research and development for example were flow-optimized prefabricated components for the pipeline engineering, weight-optimized components for lightweight construction of motor vehicles and strain-optimized structural parts with adjusted dimensions of cross section for the space frame of motor vehicles. Worth mentioning are both advantages in design and engineering such as reduction of process steps or saving of welding seams. Moreover the fact is important that generally all kind of ductile materials (steel and aluminum alloys as well as copper and brass) can be processed in hydroforming.

### 15.2.8.1 **Process Principle**

Hydroforming is regularly made on tubular components where straight or preformed pre-products are expanded in a split tool by applying hydrostatic pressure [15.78]. The stages of this operation are as follows. The workpiece is placed into the die, the die halves close, the mandrels (rams or sealing plungers) move in a close position to the workpiece and the pressure fluid is filled into the workpiece while air escapes. The mandrels now seal perfectly and the internal high pressure is built up. The pressure increases and the workpiece is shifted and formed in axial and/or radial direction. Finally when the workpiece is already very close to the shape of the dies a calibration pressure of about 2000 to 2500 bar is applied by which the workpiece is accurately adapted to the contours of the dies. Recently the calibration pressures have been increased up to 4000 bar and higher to match also the requirements e.g. in case of thicker material walls.

The main operations of the hydroforming process are indicated in Figs. 15.42–15.44.

### 15.2.8.2 **Process Configuration**

The features of the hydroforming process follow the same principles of tensile and compressive conditions as explained initially in this chapter. Due to strain hardening hydroforming allows the production of complex and light weight components of high strength which cannot be obtained by other production processes.

Preconditions for an uninterrupted course of manufacture are several requirements which have to be observed. The parameters of the internal pressure and the axial force determine most basically the hydroforming process. The work diagram (Fig. 15.41) schematically presents the forming limits and the possible progression of the operating parameters. The combination of the process parameters and characteristic values of the materials effects reasonable results only within the process window. Operating parameters outside the process window cause typical failures such as folding, buckling and bursting.

### 15.2.8.3 **Tribological Aspects of Hydroforming**

Besides the formability of the material and the geometrical aspects of the workpiece the friction conditions between workpiece and tool are most important for the hydroforming process.

Neither the friction conditions, however, nor the factors influencing the friction have been studied sufficiently. In the sheet metal forming the friction coefficient is known to depend generally on different parameters such as contact normal stress

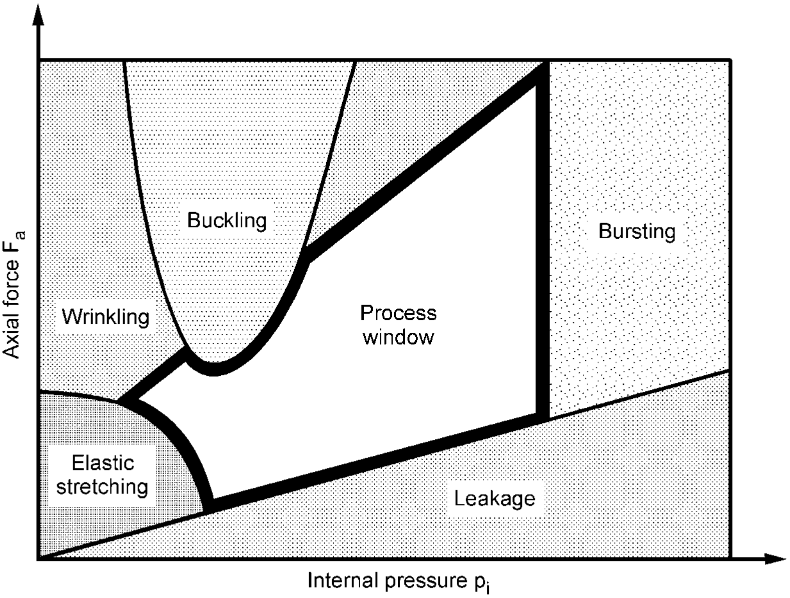

**Fig. 15.41** Work diagram of internal high pressure forming. (Source: VDI-guideline 3146 [15.77]).

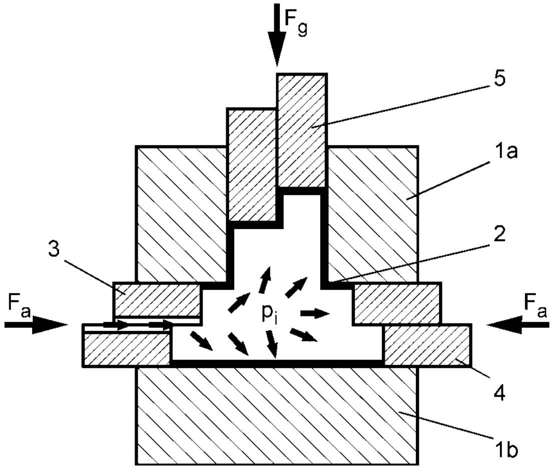

**Fig. 15.42** Hydroforming: operation step–expansion. $F_a$, closing force; $F_g$, counter force; $p_i$, internal pressure; 1a, upper die half; 1b, lower die half; 2, workpiece (tube); 3, mandrel (with inlet for the secondary hydraulic fluid); 4, mandrel; 5, counter pressure punch.

[15.146], tool and workpiece surface texture, viscosity and composition of the lubricant, sliding speed, temperature and so on. In hydroforming the situation is even more complex, the contact normal stress is influenced additionally e.g. by the internal pressure and the axial force. All these technological parameters change constantly during the hydroforming process. Different research projects have been started to study the influence of the friction coefficient under clearly arranged and simplified conditions.

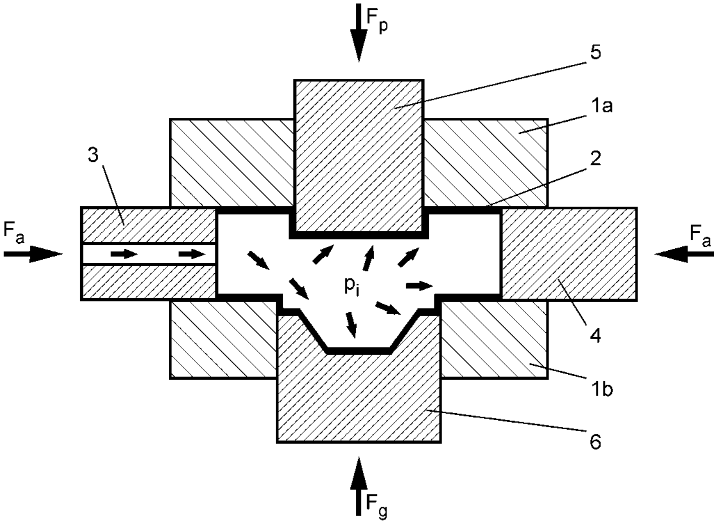

**Fig. 15.43** Hydroforming: operation step–displacement. $F_a$, closing force; $F_g$, counter force; $F_p$, punch force; $p_i$, internal pressure; 1a, upper die half; 1b, lower die half; 2, workpiece (tube); 3, mandrel (with inlet for the secondary hydraulic fluid); 4, mandrel; 5, punch; 6, counter pressure punch.

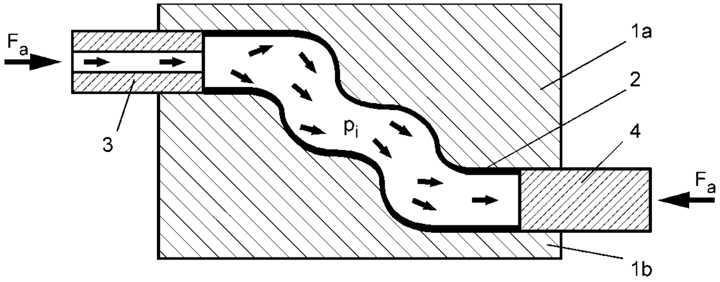

**Fig. 15.4** Hydroforming: operation step–calibration. $F_a$, closing force; $p_i$, internal pressure; 1a, upper die half; 1b, lower die half; 2, workpiece (tube); 3, mandrel (with inlet for the secondary hydraulic fluid); 4, mandrel.

#### 15.2.8.4 **Lubricants for Hydroforming**

Lubricants are used in operations before hydroforming, e.g. for the bending of the tube and the preforming of the workpiece. The remaining lubricant sometimes suits also for the hydroforming process itself. Additional lubrication (sometimes called ‘external lubrication’) is necessary in many other cases depending on the shape and the geometry of the workpiece, especially when little radii of curvature and difficult formable materials have to be processed. Thus the range of lubricants is wide but intricate. It includes drawing oils and compounds as well as wax-films and solid film lubricants [15.79]. In respect to the pressure conditions lubricants of high polarity in combination with anti-wear or EP-additives have to be used. The selection is preferably done in accordance with the application conditions. It is clear that the removability of the residues of the lubricant becomes more and more essential since this is the relevant parameter of the general process compatibility.

Another essential parameter is the secondary hydraulic fluid. This functional fluid is the pressure medium in hydroforming while the primary hydraulic fluid is used in bed and slide and to move the sealing plungers as well as the counter-pressure cylinders. As secondary hydraulic fluids aqueous solutions or emulsions are in use. The solubility of the hydroforming lubricant in the secondary hydraulic fluid is one of the fundamental features of the hydroforming process.

Where oils are used as external lubricants two alternatives were successfully established in the practice depending both on the constructive conditions of the tank-side equipment and the degreasing facilities after hydroforming: the use of rather soluble and more often the use of insoluble (demulsifying) lubricants.

Additional operating steps are necessary when solid film lubricants are used. The particular application properties of this type of lubricants and the subsequent degreasing possibilities must be adequately arranged.

## 15.3
## Lubricants for Rolling

*Theo Mang and Wolfgang Buss*

### 15.3.1
### General

Forming processes are classified according to the main deformation stress, pursuant to the standard DIN 8582. Accordingly, rolls are classified as ʹforming under compressive conditions'. Based on the quantitative production of semi-finished goods, rolling is one of the most significant processes in forming technology. Cast slabs, blooms, billets and wire can be processed to semi-finished products through rolling.

The main forming takes place in the hot-forming temperature range, to be able to utilize firstly, the advantages of lower rolling forces and, secondly, to obtain semi-finished products with soft structures suitable for further processing. Semi-hot forming has only been used to an insignificant degree in the rolling sector. On the other hand, cold rolling is a process with surprising significance, more especially in view of the increasing demands being put on forming accuracy and the quality of the surface finish.

Of all the products manufactured by rolling, in type and quantity the flat products have the greatest significance, in both the hot and cold rolling sectors. These processes are followed by the important hot rolling processes for tube, section and wire production and last but not least the cold rolling process for producing taps and spiral drills. Apart from this, teeth and thread are rolled – even on one-off work pieces – and there are a number of smooth rolling methods for surface finishing. For the production of sheet and strip is made up to > 90 % from ferrous metals, aluminum sheet accounts for only approx. 5 to 8 %, with copper materials in the region of 2 to 3 %.

Strip widths from ≥ 650 mm are generally termed 'wide strip' [in accordance with DIN EN 10130 (previously DIN 1623)] and ≤ 650 mm as 'narrow strip' [DIN E EN 10139 (previously DIN 1624) or ISO 6932].

In sheet steel processing there has been an increasing trend towards both high-strength steel [15.147] and hot strip with improved surface quality in recent years. Wherever possible today, hot rolled strip is used instead of cold rolled sheet because of the lower price. For example, hot processed sheet down to a thickness of approx. 1.5 mm is used in automobile production.

The use of high quality hot rolling oils in the forming process instead of water cooling has contributed to a large degree to improving the surface quality and the formability of hot strip.

In the past years there has been continuous further development of lubricants. Also in other forming processes under compression. Although the lubricants for the various processes are fundamentally different they have many common features for the technology and tribology of rolls, especially for the behavior of the lubricant in the roll gap. Some criteria have decisively influenced the development of lubricants, especially rolling emulsions, and this will be explained briefly here.

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#### 15.3.1.1 **Rolling Speed**

Up to the 1930s, the standard roll stand for the cold rolling of sheet metal was the four-high reversing roll-stand. This was followed by the building of tandem roll stands, in the initial stages predominately three four-high roll stands, followed by four four-high roll stands in the fifties and, finally, from the sixties onwards five and six four-high roll tandem lines [15.80].

Although the maximum rolling speed of a reversing roll stand or a three-stand tandem line around 1940 was only about 500 m min$^{-1}$, the first four-stand tandem line installed ran at 1000 m min$^{-1}$ and today's five or six-stand tandems at 1500 to 2000 m min$^{-1}$. Above all, modern roll stands can be run in all working phases at a higher speed with high mechanical safety. The reason for this is the enormous progress made in measuring, testing and control technology. Contributing to this are a number of measuring and regulating facilities incorporated in the cold-rolling mill line which are supplemented by systematically linked regulation of the applied lubricant:

- in the case of the work roll: speed and torque, rolling force and adjustment,
- in the case of strip: thickness and thickness profile, speed, surface testing, width measurement and edge positioning, inner fault testing, temperature measurement
- in the case of the reel tensioner on the coil: strip traction, flatness.

#### 15.3.1.2 **Rationalization**

Not only the output increased with the introduction of tandem lines and an increase in the number of roll stands. The demands increased to the same extent as production costs dropped. This resulted in the need to improve the lubricants which are now supplied by large central lubrication systems and have to be adapted to the new demands. For example, it became absolutely essential to adjust the degree of rolling emulsion dispersion to take into account the operating requirements for roll performance and peripheral volume.

#### 15.3.1.3 **Surface and Material Quality**

The new tandem lines allowed the surface quality of less expensive flat steel products to be increased to a previously unknown extent. At the same time the material parameters were being continually met. It became necessary to push ahead with the improvement of rolling lubricants, especially because of the need for freedom of annealing residue.

#### 15.3.1.4 **Hygienic Commercial Requirements**

Standard lubricants (blank oil) used in earlier years generated enormous lubricant aerosol at higher rolling speeds and had to be replaced by easy to handle lubricants, generally water mixed, to reduce the exposure and risk for workers.

### 15.3.2
### Friction and Lubrication when Rolling

The rolling of sheet and strip, as the most important process, is to be reviewed below. Unlike other forming processes the friction between tool (work roll) and workpiece (rolled material) when rolling must be neither too high nor too low, so that the friction necessary for feeding can be applied. This is complicated when, as in the case of sheet rolling, the full rolling force is transferred by the work rolls, whereby in the case of strip rolling, the large share of the rolling force is applied through the reel tensioner.

Figure 15.45 shows the rolling process as a simple model. The sheet to be rolled runs at the speed $v_1$ into the roll gap of the rolls turning at a peripheral speed $v_3$. During this operation the thickness of the sheet is reduced from $s_1$ to $s_2$.

The sheet reduction $\varepsilon$ is usually given as a percentage:

$$\varepsilon = [(s_1 - s_2) \times 100]/s_1$$

Reducing in the roll gap leads mainly to an extension of the rolling stock length but not to an increase in width. Accordingly, the initial speed of the sheet must be lower, and the final speed higher, than the peripheral speed of the rolls. As a result sliding friction occurs. The difference in speed between $v_1$ (run-in) and $v_3$ (rolls) given as a percentage, is called backward slip,

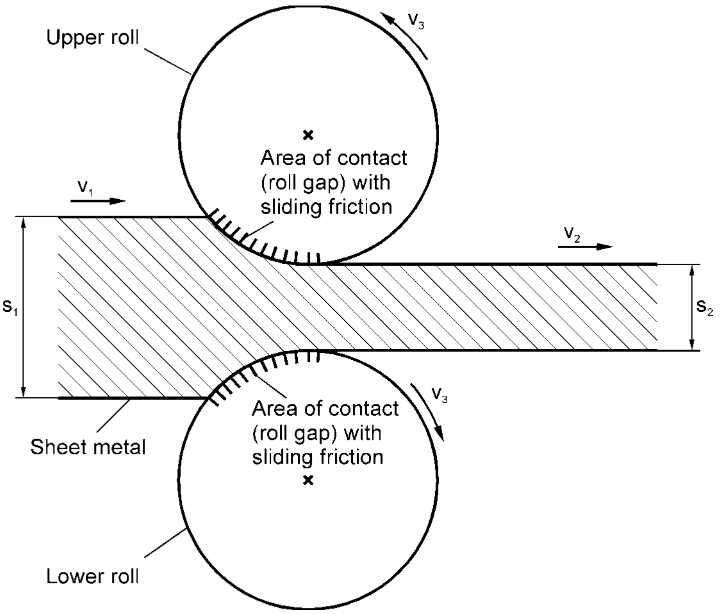


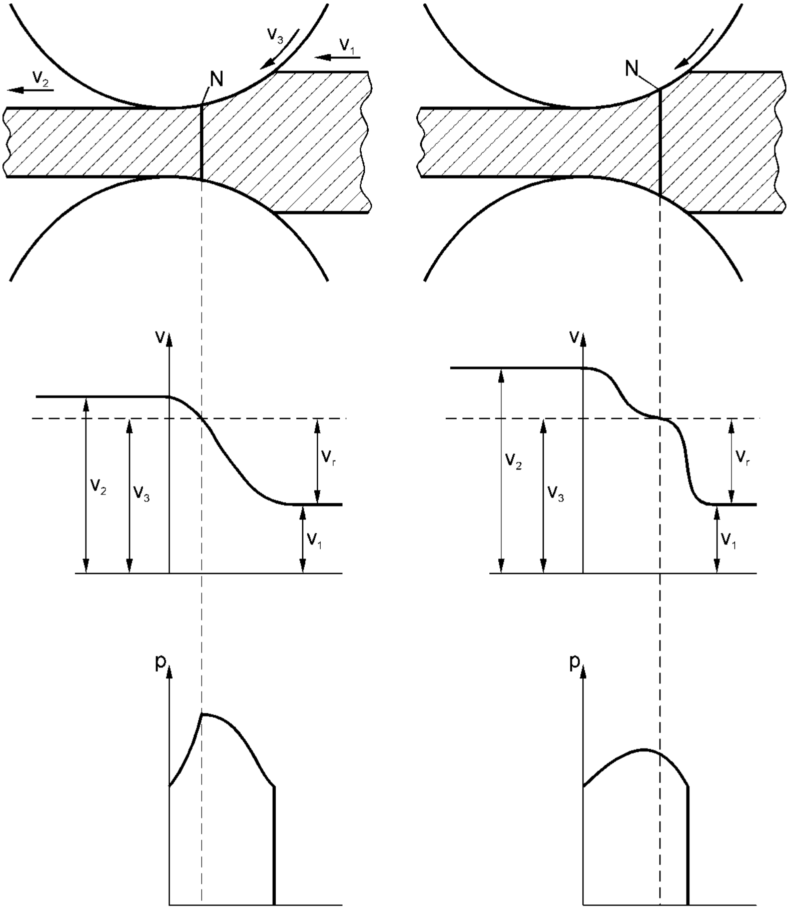
**Fig. 15.45** Principle of the rolling process. $v_1$ and $v_2$ are, respectively, the initial and final speeds of the sheet, $v_3$ is the peripheral speed of the sheet, $s_1$ is the initial sheet metal thickness and $s_2$ the sheet metal thickness after rolling.

$[(\nu_3 - \nu_1) \times 100]/\nu_1$

and the difference between $\nu_2$ (run-out) and $\nu_3$ (rolls) is called forward slip.

$[(\nu_2 - \nu_3) \times 100]/\nu_3$

In the contact area of the friction arc there is a point in which the speed of the rolls and sheet is the same, the neutral point N (no-slip point), at which there is no slip. As a result of varying friction, for example, caused by different lubrication, the N position within the friction arc can be moved. Sheet speed and pressure distribu-



Example 1

Example 2

**Fig. 15.46** Different states of speed and pressure in the roll gap (cf. Schey [15.81]). $\nu_1$ and $\nu_2$ are, respectively, the initial and final speeds of the sheet metal, $\nu_3$ is the speed of the rollers, N is the neutral point (non-slip point), $\nu_r$ is the speed difference between the velocity of the rollers and the. sheet metal in the roll gap (friction caused by sliding speed), and $p$ is the pressure. Example 1 is a neutral point with marked pressure maximum; example 2 is an adhesive area without marked (or with minor) sliding speed and sliding friction, and no pressure peak.

tion must not be uniform as a matter of principle but can vary depending upon the position of N (Fig. 15.46). In example 1, N is positioned nearer to the run-out than in example 2. Moreover, in example 2 there is a larger section given with relatively low slip. This leads to an increased proportion of static friction (sticking) instead of slip friction and, as a result, to an increase in the friction in total, i.e. more energy must be expended on compensation for friction. Moreover, stronger pick-up takes place on the work rolls because of unfavorable pressure conditions. By improving the lubrication, N can be moved towards the roll gap exit. It is possible to reduce the pressure tension through an analog-running tensile stress, namely by involving reel tension. The tribological ratios are improved in general through this measure, which increases the degree of deformation efficiency so that the undesirable widening of the strip can be almost completely avoided.

More theoretical papers have been written on the friction process covered briefly here than on all other known processes in metal working. However, all these theoretical papers for describing the processes in the roll gap have not led to any direct explanations which could be used for the further development of roll lubricant.

The significance of hydrodynamic lubrication in the roll gap has also been studied in manifold ways, likewise the elastohydrodynamic theory and the plastohydrodynamic model. The geometric relationship when the strip enters the roll gap also benefits hydrodynamics, which also explains, in comparison with other metal working fluids, the unusually high influence of the emulsion viscosity on the friction ratio. The specific property of emulsified lubricant comes into effect under higher load and at high speed, the so-called plate out. To be understood under this is the capacity, under these conditions, to form a continuous oil phase in the roll gap and build this up for an extremely short period of time to take advantage of an effective lubricating film in the border area between work rolls and roll bite.

Lower friction has the effect of lower roll wear and prevents the occurrence of heat stripes and similar rolling faults. However, as a matter of principle it must be made clear

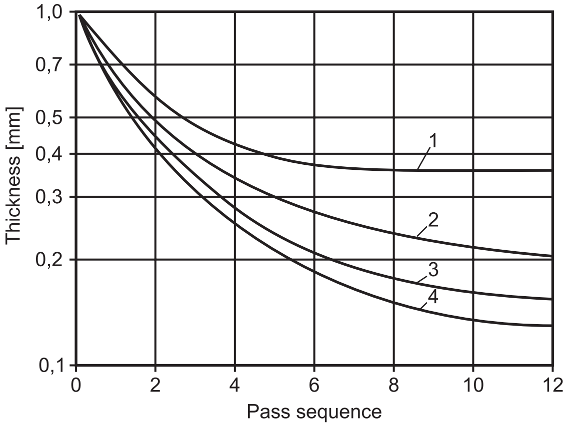


**Fig. 15.47** Lubricity of rolling emulsions according to the sheet metal thickness as a function of pass sequence [15.82]. 1, water; 2, emulsion, based on rapeseed oil; 3, emulsion, based on mineral oil with EP additives; 4, emulsion, based on palm oil.

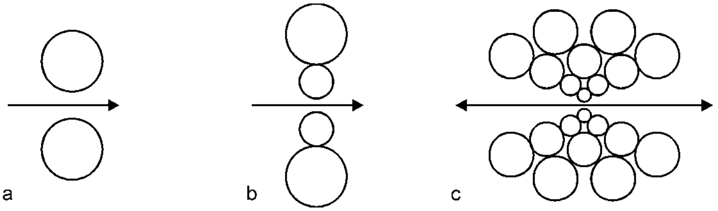


**Fig. 15.48** Typical design of roll stands. (a) duo roll stand; (b) quarto roll stand; (c) multi roll stand (construction Sundwig or Sendzimir).

that the rolling operation is only possible with a minimum of friction, to safely avoid firstly, so-called refusals when starting the rolling operation, and secondly, to prevent roll slipping (and, as a result, further rolling faults such as, for example, uneven surface roughness, rubble marks, etc.) during the rolling operation.

A practical method for measuring the influence of lubricant on the rolling result is, for example, measuring the rolling forces on trial roll stands. The reduction in thickness in a specific random sample program can be also studied (Fig. 15.47) and, above all, the achieved surface can be assessed. Testing methods on a laboratory scale generally allows no practice-relevant statements to be made on the efficiency of roll lubricants.

The type of roll stand has great applied engineering significance for the use of lubricant. The most common types of roll arrangements are shown in Fig. 15.48. In the case of the multiroll stands the forming lubricant not only lubricates the action joint between the rolling stock and work rolls but also the contact lines between work and back-up rolls.

Two-high stands have the disadvantage that they cannot produce ideal rectangular strip cross sections because of the roll sag under load. The conditions are better where stands with back-up rolls are concerned. The small work rolls of multiroll stands such as the Sendzimir type ensure a considerably extended degree deformation efficiency. The disadvantage is that a quite uneven strip is produced despite accurate control of the up to 18 back-up rolls. Generally this strip has to be leveled by stretching.

### 15.3.3
### Rolling Steel Sheet

#### 15.3.3.1 Hot Rolling

Slab produced by means of continuous casting or continuous casting are produced on hot roll stands. As already mentioned, special hot rolling oils, are used in the production of high quality strip on hot wide band lines (Fig. 15.49).

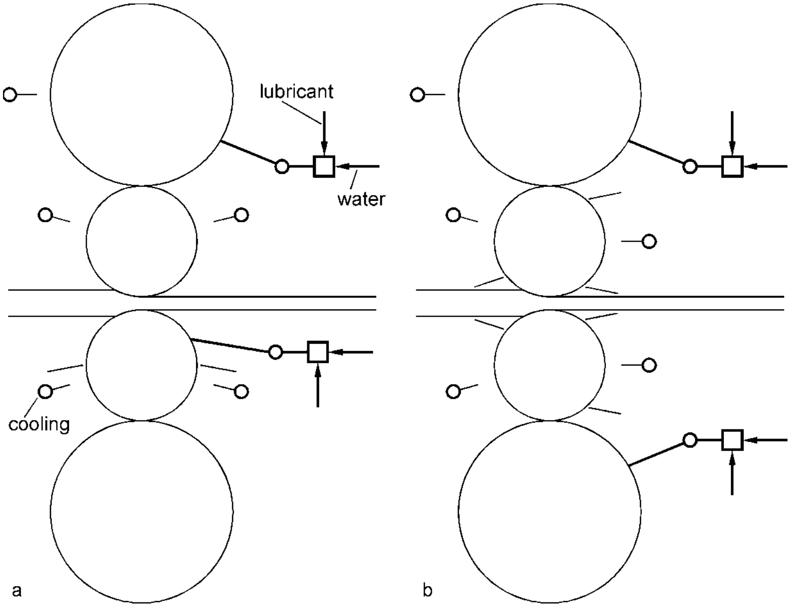


**Fig. 15.49** Arrangement of spray-nozzles for cooling and lubrication during hot rolling of (a) thin and (b) thick hot strip [15.83].

These lubricants comprise thermally stable basic materials and are different to the usual emulsified rolling oils for cold rolling in that they cannot be mixed with water. Hot rolling oils are generally, together with the necessary cooling water, applied to the work rolls by means of a separate spray system and reduce the friction between work rolls and roll bite but also between work and back-up rolls. In this way the tribological relationships are improved in the long-term and lead, more especially, to reduced roll wear. Parallel to this, an effective reduction in thickness can be achieved by lowering the rolling force, in other words, the forming performance increases despite lower consumption of drive energy.

The most significant reason for the use of hot rolling oils is, however, the improved quality of the surface finish down the entire length of the strip.

Hot rolling oils contain, besides mineral basic oils, polar and EP active substances, which form a wear-resistant 'protective skin' on the work rolls. Moreover, it must be ensured that the drops of oil are firmly held during the brief period they are stressed on the roll surface before they are washed away by cooling water or burn away when coming into contact with the glowing surface of the rolling stock. The consumption of hot rolling oil for steel is between 15 and 80 g $t^{-1}$.

The use of hot rolling oils presupposes, apart from a sophisticated application system, an effective extraction of the vapors arising as well as a satisfactory cooling water treatment.

### 15.3.3.2 Sheet Cold Rolling

To be understood under sheet is a thickness range between 3.0 and 0.35 mm (finished sheet). The strip produced for automobile production is particularly significant in this case. The final thicknesses, which are frequently between approx. 1.1 and 0.4 mm put highest demands on the cooling oil emulsions, particularly in this sector. The rolled strip widths are between 600 and 1800 mm.

The rolling speeds are between 600 to 2000 m $\mathrm{min}^{-1}$, depending upon the design of the tandem line, and even higher in individual cases.

The hot strip used for cold rolling is generally 6.0 mm thick. Before cold rolling the hot strip surface is descaled by means of hydrochloric acid or sulfuric acid and washed down to remove the acid. Hot strip, which is not scheduled for subsequent cold rolling is frequently coated with a standard corrosion protective oil and traded as a semi-finished product. Otherwise a so-called pickler oil or precoat oil is applied which primarily provides protection against corrosion and prevents the strip from being scratched when coiling. The choice of pickler oil has a great bearing on the later cold rolling process [15.84]; used to some extent is the concentrate of the rolling oil mixed in the emulsion or a closely associated lubricant. There is no coiling where continuous processing is concerned. In this case either rolling oil or emulsion is applied immediately before the strip enters the cold roll stand.

The oil applied after pickling or immediately before cold rolling is to be only slowly emulsified during the rolling process in order to ensure, even in the case of multistand tandem lines, sufficient basic lubrication of the strip down to the last pass.

After rolling, the strip must be annealed under inert gas. In a continuous process, annealing can follow straight after rolling or by annealing the round coils in a bell-type annealing furnace. Should it not be possible to wash off the remaining oil or emulsion before annealing, the rolling oil must be free of annealing residue (mill-clean process). If the strip is washed before annealing, the rolling lubricant can be designed to produce a stronger lubricating effect without having to consider freedom from annealing residue.

#### Rolling Emulsions

Where rolling is concerned, apart from the tribological tasks, the emulsion has to take over the very important function of heat transfer. The composition of this lubricant for rolling depends to a great extent on the circulation system and the other conditions and parameters for the rolling operation.

Rolling oil bases are mineral oil hydrocarbon substances or synthetic hydrocarbon material in combination with polar and EP active substances and more especially, a specifically matched emulsifier system. The saponification number of the emulsion concentrate gives an indication as to the share of polar substances [15.85] it contains. Rolling oil saponification numbers for rolling sheet are frequently between 60 and 180 mg KOH $\mathrm{g}^{-1}$. Different synthetic esters are used, frequently polyolester, but also vegetable and animal fat substances [15.85].

Free fatty acids indeed increase the lubricating effect but they must not be too high in proportion because of possible reaction with soluble soaps. The hydrolysis resistance of ester components must be high in order to keep the development of soaps low. The acid value (or neutralization number) gives an indication as to the quantity of free acids. Values of 3 mg KOH $g^{-1}$ are equivalent to approx. 1.5 % acids.

Rolling emulsions are mixed with fully softened water, generally between 1 and 3 %. The emulsion temperature is frequently between 35 and 55 °C. The consumption of precoat oil and emulsion concentrate is together between 0.25 and 1.00 kg $t^{-1}$ sheet.

The emulsion stability is important for the rolling process. Stabile emulsions are generally used in high volume circulating systems, and metastable emulsions in smaller circulating systems. In the case of metastable emulsions one advantage is the easier flotation of leakage oil, abrasion and dirt; stable emulsions are generally easier to control.

The stability of fresh emulsions is determined, for example, in accordance with ASTM D 3342 and is frequently given as stability index. This testing method is suitable as a characteristic specification value for incoming lubricant control. During operational use it is better to use the degree of dispersion of the emulsion (particle size distribution) instead of emulsion stability. The degree of dispersion can be determined by photometric or diffractometric methods. The results also allow conclusions to be drawn as to the share of foreign oil and the correct ratio between emulsifiers and polar components.

Table 15.13 shows frequently measured key data of rolling emulsions.

**Tab. 15.13** Frequently measured rolling emulsion data.

| *Property* | *Fresh* | *Used* |
|---|---|---|
| Concentration (fat content) | x | x |
| Foreign oil (leakage oil) | x | |
| Emulsion stability after standing | x | x |
| Particle size distribution (degree of dispersion) | x | x |
| Heat stability | x | |
| pH value | x | x |
| Electrolyte stability | x | |
| Electrical conductivity | x | x |
| Foaming properties | x | |
| Corrosion protection against iron | x | x |
| Iron content | | x |
| Chloride content | | x |
| Sulfate content | | x |

As a general rule, the emulsion is checked several times a day during operation and particular attention is paid to the degree of dispersion, the cleanness of the sheet and the risk of corrosion. The pH values are frequently between 6.0 and 7.0, the chloride content is not to exceed 20, the sulfate content 40 or the iron content 100 mg $l^{-1}$.

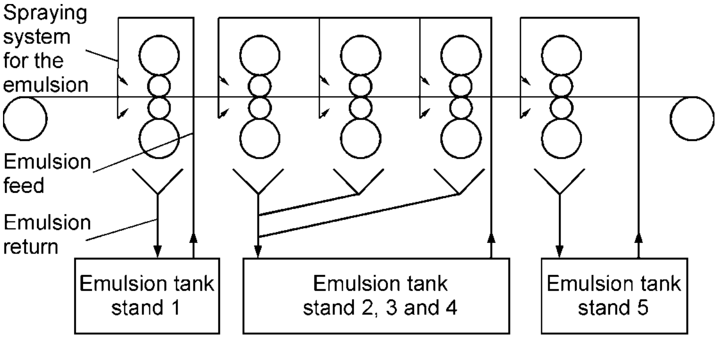


**Fig. 15.50** Emulsion systems in a five-stand four-high rolling mill for sheet metal.

### Circulation Systems for Emulsions

The dimensioning of an emulsion system is orientated on the number of stands, the rolling capacity and the various possibilities of types of emulsions. There are multi-stand lines with their own emulsion system of < 100 $m^3$. However, generally there are considerably greater quantities in circulation. Figure 15.50 is a schematic drawing of a very flexible facility: stand 1 has own tank with approximately 100 to 150 $m^3$, stands 2, 3 and 4 are supplied from a large tank with a capacity of approximately 600 $m^3$ and stand 5 has its own system with a capacity of approx. 100 $m^3$. Each individual system can be provided with skimmer and filtration system (for example, with paper and magnet filters). With such an arrangement it is possible to run different concentrations of emulsion in each individual basin, for example, at the last stand with a cleaner for better cleanliness.

Adequately large circulation systems enable foreign oil and contamination to be skimmed during operation, a process which does not function perfectly in the case of small systems because of the high circulation rate. In this case a facility is occasionally used where a second tank is set up and serves as a retention basin.

Small plants for narrow width roll stands are usually run with stabile emulsions in a pH range from 8.5 to 9.3. The emulsion must provide considerably higher corrosion protection.

### Strip Cleanliness and Annealing

Annealing is always undertaken under reduced inert gas.

In all applications where there is no washing before annealing, conditions for strip cleanliness after annealing have to be provided by using an especially well cleaned emulsion free of ignition residue substances(base oil, ester, emulsifiers). Besides this principle measure, further factors such as state of coil (tight or loose), influence the annealing result [15.86].

On the other hand, where continuous annealing is concerned the conditions are clearly more favorable because evaporated or cracked substances can escape easier.

Both spectroscopic processes and even conventional testing methods are applied to determine the cleanliness of the strip, for example, the residue hydrocarbon.

Compared with the conventional annealing process which takes several days and comprises electrolytic cleaning (only facultative, not compulsory), dwell type annealing, coil cooling, temper rolling (skin pass) and finally strip inspection, all the processing stages of the continuous process, (including cleaning) take place in one plant so that the run-through time only takes about 10 minutes. The continuous annealed material stands out due to its very clean surface, good flatness and the consistent mechanical properties over the length and width of the strip.

The difference between the continuous annealing processes is primarily the achievable cooling speeds involving gas-jet, roller quenching, water spray and/or hot water quenching [15.87]. The known annealing processes are given in Table 15.14.

**Tab. 15.14** Annealing processes.

| | |
|---|---|
| CAPL (Continuous annealing and processing line) | Design: Nippon Steel Corporation (Japan) |
| NKK-CAL (Nippon Kokan continuous annealing line) | Design: Nippon Kokan (Japan) |
| KM-CAL (Kawasaki multipurpose continuous annealing line) | Design: Kawasaki Steel Corporation (Japan) |
| HOWAQ (hot water quenching) | Design: Centre de Recherches Métallurgiques (Belgium) |

By varying the temperature program and more especially by controlled cooling speed it is possible, during continuous annealing, to set the system for completely different qualities depending on the annealing cycle, for example, strip for drawing and deep drawing, high tensile strength steels, dual phase steels and bake-hardening steels.

### Re-rolling

In so-called skin pass rolling mills the strip must be re-rolled after annealing to improve the flatness and achieve the required material properties (forming changed tensile strength, tensile yield strength etc.). Narrow strip is very often skin passed with two-high stands but wide strip only on four-high stands. The reduction in thickness is only about 1 to 3 %. The strip is rolled dry or wet with special temper rolling oils, which, in particular, increase the strip cleanliness. Skin pass fluids have also proved to be successful for providing a uniform mat finish, because the surface of the temper rolling roll closes more slowly and does not pick up dirt or friction particles.

Skin pass fluids are synthetic watery solutions and mainly contain tenside as well as organic and inorganic acid derivatives as corrosion protection components. Normally these are free of sodium nitrite and must be compatible with the subsequently applied corrosion protection oils and prelubes.

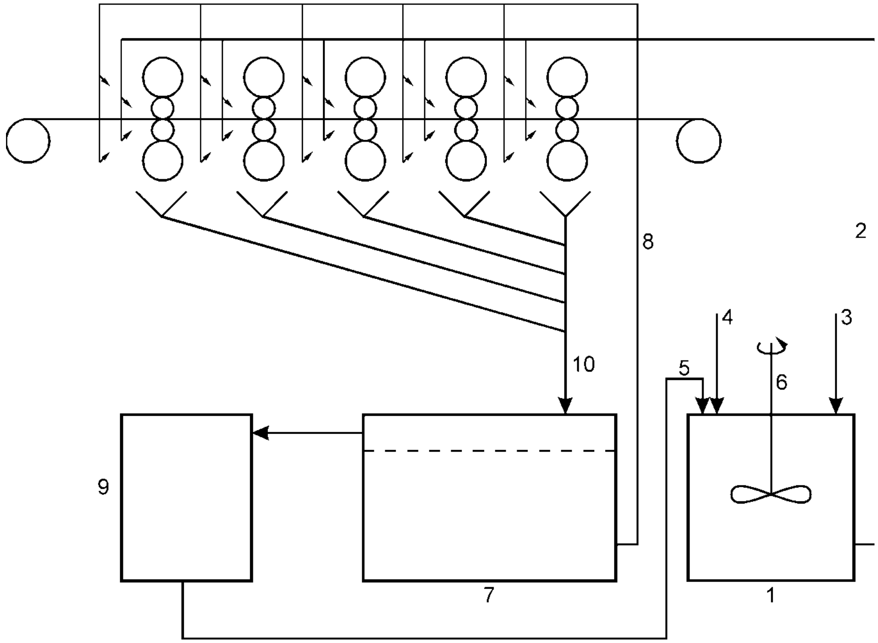


**Fig. 15.51** Finest sheet cold rolling on a five-stand four-high tandem mill; principle of the so-called direct application. 1, preparation of an dispersion out of water and fatty oil (15–20 %); 2, dispersion feed to the rolling mill; 3, addition of water to the mixing tank; 4, addition of fresh fatty oil to the mixing tank; 5, addition of reconditioned fatty oil to the mixing tank; 6, mixing tank; 7, return tank (separation of fatty oil and water); 8, water feed (containing max. 1 % fat) to the rolling mill; 9, reconditioning of the fatty oil; 10 return of cooling water and dispersion.

### 15.3.3.3 **Finest Sheet Cold Rolling**

Finest sheet has a thickness range of 0.15 to 0.35 mm and is rolled as wide strip on five or six-high stand tandem lines and galvanized afterwards to provide so-called tin sheet. This is why this thickness range is generally referred to as tin plate.

Rolling is carried out with semi-stable high fat content emulsions by the so-called direct application of fat oil dispersions (Fig. 15.51). Before tin plating the remains of lubricants are removed in an electrolytic degreasing plant. In the case of direct application, natural fats such as palm oil or animal tallow are often prepared as a 10 to 20 % dispersion in completely softened water in large mixers. The drops of fat oil are approx. 50 μm in size. The fat and water are separated during the return run, treated and used again. Recovered fat can be used for rolling until an acid number of approx. 20 mg KOH $g^{-1}$ is given.

Where semi-stable emulsions are used one works with initial concentrations of 3 to 6 % in fully softened water (Fig. 15.52). The size of the drops in such emulsions is between 10 and 50 μm. The treatment of semi-stable emulsions is less expensive than dispersions used in direct application.

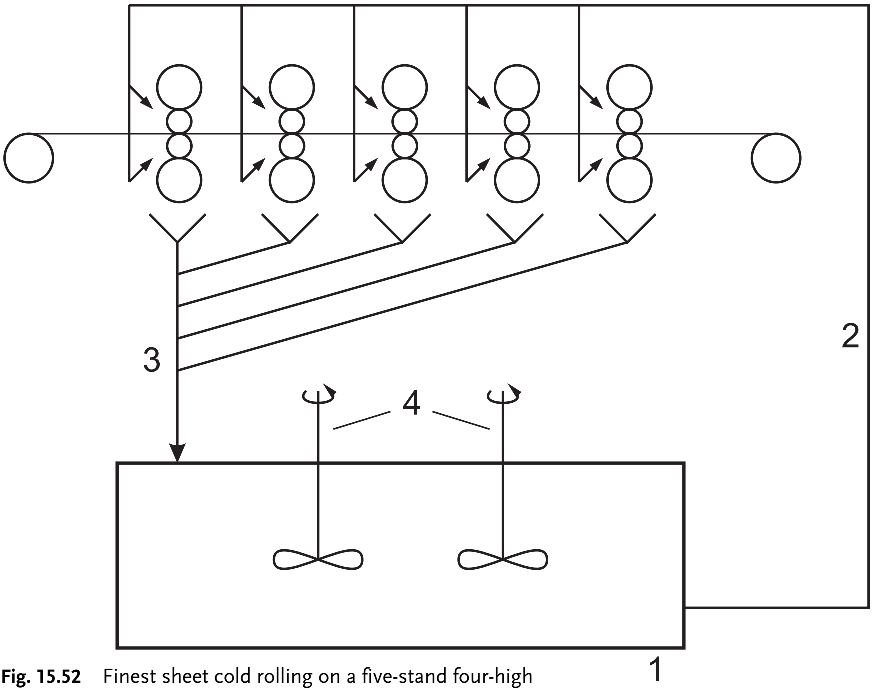


**Fig. 15.52** Finest sheet cold rolling on a five-stand four-high tandem mill using a semi-stable emulsion (without filtering station). 1, emulsion tank (3–6 %); 2, emulsion feed; 3, emulsion return; 4, stirrer.

### 15.3.3.4 Cold Rolling of High Alloy Steel Sheet

Higher alloyed steels, more especially high grade stainless steels, are rolled on multiroll stands with a preference for lubricants which do not mix with water. Although high grade stainless steels generally produce scale to a considerably lesser extent, the steel is pickled before cold rolling to keep roll wear low. Austenitic steels are generally pickled with a mixed acid without hydrochloric acid content, but ferritic steel is pickled with hydrochloric acid.

The rolling speeds, which depend upon the alloy and material thickness, can be up to 500 m $\text{min}^{-1}$, in which case, for example, high grade stainless steel hot strip be reduced by up to 85 % in eleven passes without intermediate annealing. Frequently, however, annealing, pickling and re-rolling have to take place in order to achieve the required strip quality.

The selection of the rolling oils has a decisive influence on the surface finish of the sheet. Mat surfaces are produced when the viscosity is too high even when using ground or polished work rolls. This is almost always undesirable in the case of high grade stainless steels. This is why in practice preference is given to mineral oils at approximately 15 to 20 $\text{mm}^2\ \text{s}^{-1}$ at 20 °C which have the closest possible boiling range [15.88]. Oils with lower viscosities often cause problems by vapor bubbles being developed through the reduced heat dissipation.

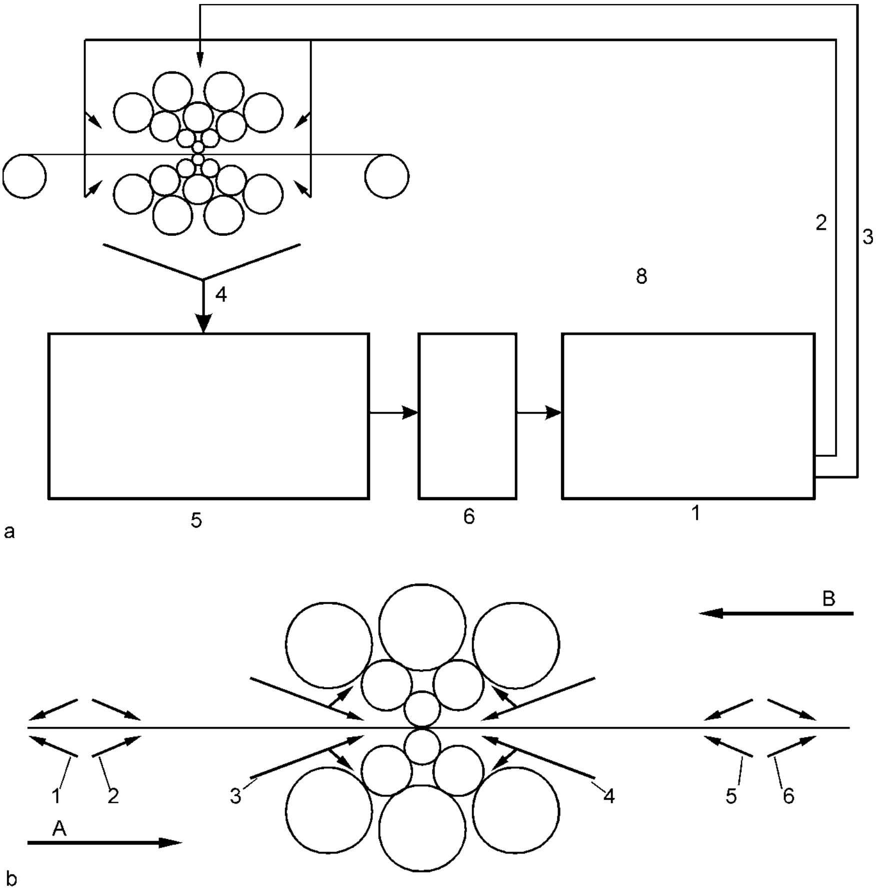


**Fig. 15.53** Rolling of stainless steel sheet on a multi-roll stand using rolling oil. (a) oil circulation: 1, oil tank with clean oil (e.g. 100 $m^3$); 2, oil feed to lubricate the strip (12 bar); 3, oil feed for the lubrication of the roller bearings (6 bar); 4, oil return; 5, return tank with uncleaned oil (e.g. 169 $m^3$); 6, oil reconditioning by hyperfine filtration (0.5–2 μm fineness, < 10 mg $L^{-1}$ load of solid matter. (b) arrangement of spray-nozzles at the rolling stand [15.88]: direction A, nozzles 1, 2, 3, 4, 5 in action; direction B, nozzles 2, 3, 4, 5, 6 in action.

Because rolling oils used on multiroll stands also take over the lubrication of the roll bearings, the additives must cater for both operations.

In the case of a twenty-high roll stand the flow rate for strip lubrication is, for example, 600 to 1000 $m^3$ $h^{-1}$ and for bearing lubrication 200 to 250 $m^3$ $h^{-1}$ with an oil temperature of approx. 40 °C. The required oil pressure for strip lubrication is approx. 12 bar and for bearing lubrication 6 bar. Oil cleaning is carried out generally by floating filtration because of the high quality surface finish required. The achievable filter fineness is generally < 2 μm and the residue contamination content < 10 mg $l^{-1}$ (solid foreign matter). Figure 15.53 shows a multi roll stand, type Sendzimir.

### 15.3.4
### Rolling Aluminum Sheet

Cast blocks from 200 to 650 mm thick are used which are rolled down on hot roll stands to 10 to 20 mm after milling off the oxide skin and heating by reversing to be subsequently reduced to 6 to 2 mm on two to five-high stand tandem lines. Reversing stands can be two-high or four-high in construction. Hot rolling is only carried out with emulsions.

Sheet and strip are rolled down from 3.0 to 0.1 mm and foil down to 5 μm by cold rolling. Cold rolling is almost exclusively carried out with low viscous, water-immiscible oil.

### 15.3.5
### Aluminum Hot Rolling

Depending upon the alloy and required reduction in thickness the blocks are preheated to 450 to 580 °C before hot rolling. With a rolling speed between 180 and 300 m $\text{min}^{-1}$ in the reversing process and appropriately higher speeds in tandem stands, the material is rolled with stable emulsions in an initial concentration between 2 to 6 % and an upstream temperature of 35 to 60 °C. The temperature in the last pass, depending upon the alloy, is still 280 to 230 °C.

The concentrates comprise mineral oil hydrocarbons or synthetic hydrocarbons with polar additives (ester, fatty acids, fatty alcohols), tensides and further anti-wear substances, where needed.

Fatty acids form aluminum soap which can cause too high slip and tarnishing. This is why it is only to be used in low amounts.

Decisive for the lubrication and quality of the surface finish is the development of a uniform, fine coating of aluminum pick-up (roll coating) on the work rolls. If the coating is too thin the friction conditions are unfavorable and frequently unstable. On the other hand a roll coating which is too thick causes uncontrolled release of parts of this coating which, in turn, leads to pick-up rolling and surface faults. To prevent this, excessive pick-up is removed from the work rolls during the rolling process [15.89] by means of brush rolls. Exact control of the temperature of the work and back-up rolls is accomplished by means of specific emulsion dosing to obtain a favorable hot strip profile.

Emulsion analysis, emulsion care and cleaning is carried out in the same way as in other applications. Here again the particle size distribution is considered as well as the stability tests. In the case of aluminum rolling, even greater attention has to be paid to the compatibility of the machine lubricants and hydraulic fluids with the emulsion in order to avoid detrimental patches.

### 15.3.6
### Aluminum Cold Rolling

After soft annealing at temperatures from 370 to 430 °C, finish rolling is carried out on cold wide strip lines at speeds up to 2700 m $\text{min}^{-1}$. The sheet thickness is between 0.1 and 3.0 mm but the major amount of rolled strip is about 0.6 mm thick and achieved in two to seven passes.

Despite the advantages of better heat dissipation, lower fire risk and less expensive health protection water mixable rolling oils [15.90] have been unable to establish themselves for this application , because of, for example, patch forming as a result of residue moisture in the round coil, and hydrogen embrittlement of the work rolls can only be controlled to a deficient degree. The demands put on cold rolling oils are summarized in Table 15.15 according to their importance.

**Tab. 15.15** Demands on cold rolling oils.

High surface quality finish
Large reduction in thickness
No spotting
High roll service life
No tendency to gum
Good filtering ability
Low tendency to burn
Fulfillment of hygienic working requirements

When making up strip rolling oils, the main attention has to be paid to the selection of the base oil. Particularly suitable are paraffinic hydrocarbon substances with a viscosity which is not too high, in order to avoid mat surface finishes. Consequently, closely cut base oils with flash points above 85 °C and a viscosity from 2 to 4 $\text{mm}^2\ \text{s}^{-1}$ at 20 °C are mainly considered. The most popular additives are straight-chained alcohols, acids and esters with chain lengths from 10 to 14 carbon atoms as well as oxidation inhibitors. The activation level seldom exceeds the 5 % mark.

The selection of base oils and finish formulated rolling oils is based on a can test at 300 °C.

It is essential for users who have a distillative rolling oil preparation system available that the rolling oil additives have approximately the same boiling point as the base oil, so that they can be distilled together. The higher boiling proportions remain in the distillation residue together with the abrasion and contamination.

If no distillative cleaning system is available floating filtration is necessary using kieselguhr with added bleaching earth where needed. Other cleaning methods without filtration aids have also been tested [15.91].

In principle, foil rolling oils differ from strip rolling oils only through a lower additive content, occasionally also through lower viscosity and preferably with a very low aromatic content to ensure compliance with the stipulations for use in the food sector.

Doubling, i.e. rolling two foils together on the top of each other, is the only successful way of rolling foils down to approx. 5 to 6 μm without the rolling stock tearing. To ensure that the two foils do not weld to each other, a low boiling point hydrocarbon substance without aromatic substances is applied by dripping as the foils run into the rolls. After rolling, the two foils are separated again in a separating operation. Double rolled foils can be recognized since they have one bright side (from the work roll) and one mat side (from the other foil).

As is the case with hot rolling, to avoid spotting synthetic or barely-synthetic special lubricants and hydraulic oils are also used when cold rolling on the roll stand and in the rolling operations.

A further problem in the case of cold rolling is the enrichment of the working area air with oil vapor and oil spray. The air-pure process [15.92] has proved to be suitable for effective air de-oiling.

### 15.3.7
### Rolling Other Materials

Copper and its alloys are generally rolled in a hot roll process with water, i.e. without special lubricants, although occasionally with, stable slightly fatty emulsions.

Generally stable emulsions with fat oils and synthetic esters are also used for cold rolling. Copper and copper alloys are nobler metals than iron and aluminum. This also has an effect on the tribological conditions [15.93]. Under all circumstances, staining of the bright, mostly decorative surfaces by the emulsion or its contents has to be avoided. Consequently special attention must be paid to ensure the absence of substances containing sulfur [15.94]. Moreover, lubricant residue must be volatilized under inert gas to leave no residue at the annealing temperatures (310 to 480 °C for copper and 540 to 650 °C for brass).

Titanium is hot-rolled, dry. Fatted oils are used for cold rolling.

Zinc is rolled in a semi-hot state at 200 °C, very often dry, or cold with a slight lubrication using low viscosity oils with polar substances or emulsions. By heating to between 120 to 150 °C when cold rolling, a large part of the lubricant is volatilized so that no further cleaning is necessary.

Tin and lead are generally cold rolled. Low viscous special lubricants are used for special alloys (solders and similar materials). Tungsten and molybdenum rolling has to be carried out at high temperatures under inert gas due to sensitivity to oxidization at high temperatures. Alternatively coating with glazes is also possible. Semi-hot or cold rolling is carried out with solid matter content or strongly polar oils with additives.

Nickel and cobalt alloys are dry rolled. The cold rolling process on multiroll stands is similar to the cold rolling of stainless steels as far as the use of lubricants is concerned.

## 15.4
## Solid Metal Forming Lubricants (Solid Forming, Forging and Extrusion)

*Theo Mang and Wolfgang Buss*

Under this heading, a large number of forming methods are included. The forming processes in this group are not precisely defined. In this section we will review in particular extrusion and forging. We can see here, especially in the case of cold forming in the production of small parts in large series, a continuous increase to the detriment of cutting processes. The material savings, and the resulting savings in energy if the fusion heat is included, are of a particular advantage. Apart from this there are a number of technical advantages compared with workpieces produced by cutting, especially the utilization of strain hardening and the favorable course of the fibers as a result of the material flow. Frequently there are transitions and combinations between the processes and forming technologies so that, for example, the exact classification laid down in DIN 8583 for differentiating between the lubrication techniques does not offer a suitable basis. On the other hand the temperature of the workpieces and tools is particularly significant for the selection of lubricants.

Impression die forging is an excellent method economically. For example, the parts manufactured mainly by independent forges attained a turnover of approximately US$ 4 billion in USA in the year 1997. The most important buyers are the automobile industry (48 %), aerospace (23 %) and the manufacturers of off-highway equipment (6 %) [15.95].

### 15.4.1
### Processes

Mainly, processes are classified on material flow and the type of the tool used. However, there are frequently no clear differences between forging and extrusion.

#### 15.4.1.1 Upsetting

To be understood under this is a process with compression between flat dies. Cold heading is also to be mentioned as a cold forming process for the production of connecting elements (screws and rivets).

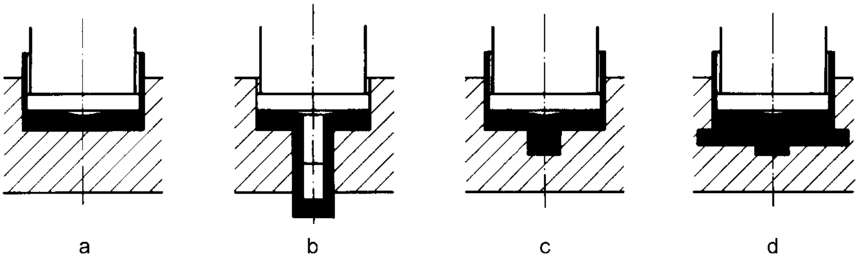


**Fig. 15.54** Some variations in the extrusion process. (a) hollow backwards (can extrusion); (b) hollow forward; (c) solid forward/ hollow backwards; (d) cross solid forward and hollow backwards.

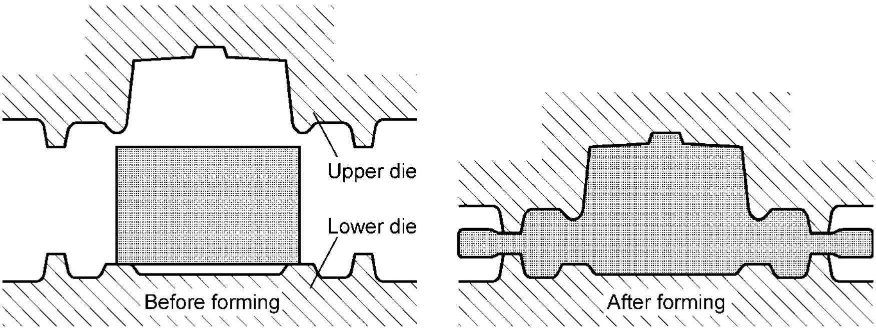


**Fig. 15.55** Impression die forging.

#### 15.4.1.2 Extrusion

During extrusion processes, the workpiece is placed in a container and compressed by means of ram movement. Figure 15.54 shows the various types of extrusion processes. Extruded products can be hollow or solid. According to the material flow, one differentiates between forward (direct), backward (reverse) and cross procedures.

#### 15.4.1.3 Impression Die Forging

This operation is shown in Fig. 15.55. Economically, it is the dominant hot forging method. This is also reflected in the significance of this group of lubricants.

Closed die forging is a special form of impression die forging. Here the filling of the die is not supported by the development of a flash. There is no possibility for excessive material to escape, and vent holes are provided for vapor generated from the lubricant to escape.

#### 15.4.1.4 Open Die Forging

Open die forging is different from impression die and closed die forging in that the metal is never completely enclosed as it is being shaped by the dies.

### 15.4.2
### Forming Temperatures

The surface temperatures of forged workpieces and tools are a decisive factor in the selection of lubricants. A difference is made between three general areas as far as both forging and extrusion are concerned:

#### 15.4.2.1 Cold

The parts are formed at ambient temperature without preheating. It goes without saying that we must also consider that considerable heat is generated at high forming speeds.

### 15.4.2.2 **Warm**

In this case the workpieces (billets) and/or the tools are heated to facilitate material forming. The billet temperature is below the recrystallization temperature of the material. Warm and cold extrusion methods can be combined with each other [15.96].

### 15.4.2.3 **Hot**

The best forming properties and lowest forces are given at temperatures above recrystallization temperature. In the case of hot steel forging the workpiece temperatures are between 1100 and 1200 °C.

## 15.4.3
## Friction and Lubrication with Cold Extrusion and Cold Forging

The best possible use of the material, good quality surface finish and dimensional accuracy, the use of strain hardening and extensive rationalization are the main features of the most important cold massive forming methods. In the foreground stand the cold extrusion presses with specific variations in methods as far as material flow and die movement are concerned.

Figure 15.54 shows a few significant variations of the extrusion method. Upsetting, embossing and reducing are methods related to the type of lubrication and are frequently combined with extruding. Numbering amongst the cold massive forming methods are also thread and profile rolling which are either follow-on process of extrusion methods, closely associated with the extrusion lubrication technology or are used in conjunction with cutting operations.

The ironing type forming method can be applied as a follow-on to either a sheet-metal deep drawing operation or be used for the cup produced by backwards extrusion.

The focal point in cold extrusion for ferrous materials lies in both non-alloyed and low alloyed steels, as well as in the case-hardened and annealed steels used in automobile production and the automobile industry which are particularly important processors of cold extrusion parts. Steels with higher carbon content are no longer extruded or only when the degree of forming is low; where stainless steels are concerned the ferrite materials can be formed better than austenitic materials [15.97].

Choosing the correct material for the extrusion tools is essential due to the extreme pressure loads; in many cases the limits to forming by extrusion are determined solely by the tensile strength of the tool. Tool steels and high speed steels, as well as carbide metals, play an important role. The preferred utilization of high speed steels as a die material has initiated developments to improve the resistance to wear; especially worth mentioning here is the possibility given for TiC-coating. New developments in the chrome steel sector (e.g. 12 % Cr, 1.2 % C, 1.4 % Mo, 2.5 % W, V), which can be further improved by the CVD method are also worth mentioning. The treatment processes such as nitration and boronization also play an essential role. Hard chromium plating has not been able to establish itself in massive forming because of the low adhesion of the chromium coating applied by

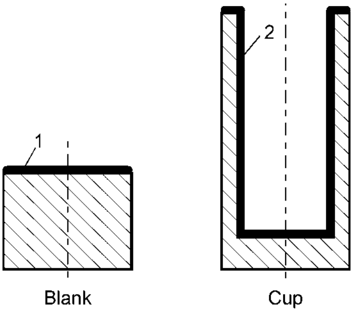


**Fig. 15.56** Lubricant stress through surface expansion with cup backwards extrusion. 1, coated surface before forming; 2, surface formed by a stamper after forming.

electrolysis. Applicable for forming tools are limit loads of 2500 N $mm^{-2}$, maximum 3000 N $mm^{-2}$ as a general rule; the limits for armored matrices with steel or carbide metal are 2000 to 2500 N $mm^{-2}$.

#### 15.4.3.1 Friction and Lubricant Testing Methods

Compared with the other forming processes covered here, the maximum surface pressures occur in cold forging and cold extrusion (up to 3500 N $mm^{-2}$). This leads to a particularly high tool load and, as a result, also to especially difficult tribological situations. To this must be added the very high surface expansion, to some extent, which has to be followed by the lubricant or the lubricant carrier. In the case of hollow backwards extrusion the surface expansion can, for example, be ten times the initial surface area (Fig. 15.56).

Table 15.16 shows the stress profile for the friction and lubrication in respect of surface pressure, relative speed and surface expansion for five processes. This also includes ironing type forming. A better comparison is possible by formulating specific values (sizes without set dimensions). The maximum surface pressure $P_{\text{max}}$ refers to the initial stress yield $k_{\text{fo}}$, the relative speed between tool and workpiece $V_{\text{R}}$, and the ratio between final and initial surface $A_1/A_0$ applies for the surface expansion. This allows conclusions about the suitability of friction and lubricant testing machines [15.98, 15.99].

**Tab. 15.16** Stress profile for friction and lubrication in respect of surface pressure, relative speed, and surface expansion for five processes [15.98].

| | *Ironing* | *Upsetting* | *Hollow forward extrusion* | *Solid forward extrusion* | *Hollow backward extrusion* |
|---|---|---|---|---|---|
| $P_{\text{max}}/k_{\text{f0}}$ | 2.1 | 5.9 | 5.5 | 6.4 | 9 |
| $V_{\text{R}}/V_{\text{WZ}}$ | 2.3 | 2.4 | 5 | 5.7 | 6.3 |
| $A_1/A_0$ | 2.2 | 4.5 | 4 | 4 | 11 |

The sliding movement between tools and workpiece, which takes place under high specific pressure and with large surface expansion, generates high friction

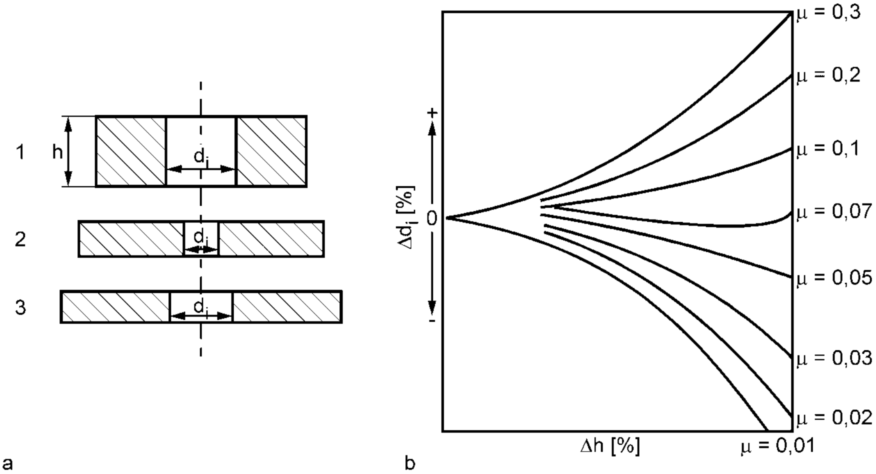


**Fig. 15.57** Ring upsetting test to determine coefficient of friction for solid metal forming. (a) geometrical changes of a ring through upsetting: 1, blank; 2, upset blank with large $\mu$; 3, upset ring with small $\mu$; (b) nomogram for the identification of the friction index through changes in the inner diameter $\Delta d$ in relation to the forming degree $\Delta h$.

losses. As a result the friction can amount to 60 % of the press force. Still, the main focus of the lubrication is not on the reduction of friction but on the material wear and surface finish of the workpiece. When changing over from soap lubricants to solid lubricants under very high stress the higher coefficient of friction is accepted to gain the high resistance to pressure and the resulting lower tool wear. Consequently, lubricant testing methods which use coefficients of friction as assessment criteria are to be applied only with the appropriate reservations.

Ring upsetting has been applied for some years to measure the friction and assess lubrication for cold extrusion. After the appropriate pretreatment of the plane fric-

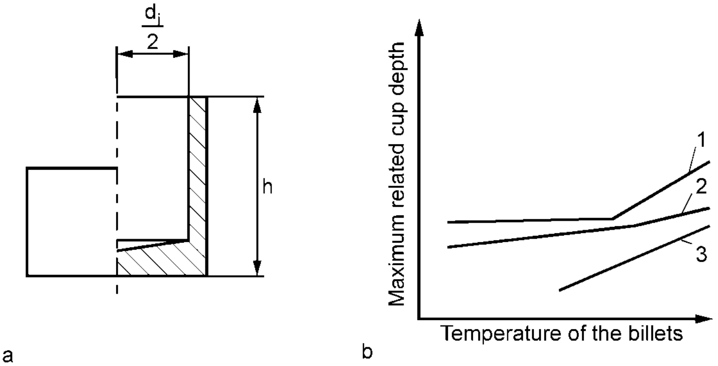


**Fig. 15.58** Testing lubricants for solid forming by cup backwards extrusion [15.102]: (a) blank and cup geometry; (b) maximum related cup height for lubricants 1, 2, and 3 are related to the temperature of the blanks.

tion surfaces a cylindrical ring is upset between two level plates. Under certain, generally negligible, restrictions a friction factor can be calculated from the change in the inner diameter of the ring alone [15.100]. Since this method can be easily applied it has also been used for warm and hot forging. Figure 15.57 shows the geometrical change to a ring in the upsetting test with large and small coefficients of friction and the determined coefficients of friction over the specimen temperature [15.101].

Considerably more suitable for lubricant assessment for cold extrusion are testing measures which are very close to practical working with higher stress. As a general rule, in the case of ring upsetting test, surface pressures below 1000 N $mm^{-2}$ occur, with form changes of $\varphi < 0.7$ and just three times the surface expansion of the ring area. This is why using the extrusion methods under extreme conditions, especially hollow backward extrusion, is seen as a test method for lubricants. In this case the maximum given cup (container) height without grooves (cold welding) is taken as assessment criteria (Fig. 15.58) [15.102].

#### 15.4.3.2 **Selection Criteria for Lubricants and Lubrication Technology**

Massive forming has countless degrees of difficulty. Playing a role, besides the material key data (yield stress $k_1$) and the change to this data with the degree of forming, are the varying degrees of forming in a forming operation, the different friction conditions (e.g. hollow backward extrusion), the demands on the workpiece surface, the admissible tool wear as well as the machine technology (multistage presses, single stage presses). The significance of a most careful analysis to determine the lubrication and surface treatment becomes clear when it is considered that the costs involved are to some extent higher than those for the entire forming itself. The results vary greatly for the application and selection of the lubricant because of the different metal forming machines. A finished part can be produced from a pretreated blank in a single stage press, or several pressing operations can be carried

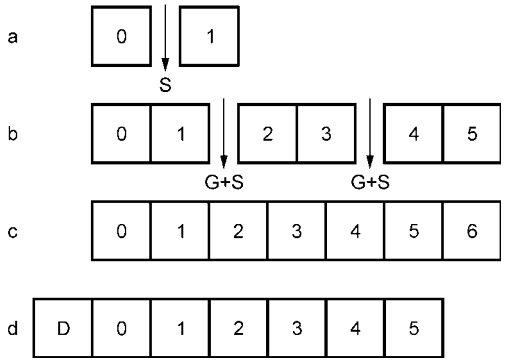


**Fig. 15.59** Examples of production sequence for cold extrusion. (a) Single-stage press for blank production (0) and pressing operation (1). (b) Multi-stage press; three press groups for shearing the blanks and with five pressing stages; option for two intermediate treatment stages with heat treatment, surface pretreatment and lubricant application for heavy forming with a large degree of forming. (c) Multi-stage press with six pressing stages without intermediate treatment of the blanks. (d) Multi-stage press with five pressing stages incorporated in front of a drawing stage for rolled wire (D)

out on single stage presses on one workpiece and, outstandingly significant in this respect, is that these parts can be also produced on multistage automatic presses. Figure 15.59 shows in diagram form the possibilities given in the production sequence of cold extrusion.

With regards to the production sequence shown in Fig. 15.59d a five-stage press is incorporated in front of a drawing stage for rolled wire. This is expedient when rolled wire with a distinctly less favorable surface than drawn wire or drawn rod material is used as the initial material. The drawing stage also allows the specific application of lubricant for the subsequent extrusion stages through a drawing box. Multistage presses of this type (Fig. 15.59c, d) have special significance for the mass production of nuts, screws, and similar parts. The extrusion stages are followed by cutting or non cutting operations (thread cutting, thread rolling).

#### 15.4.3.3 Lubricating Oils for Cold Extrusion of Steel (Extrusion Oils)

For the lubrication of cold extrusion operations with a maximum degree of difficulty, most importance is to be attached not on the oils but on the inversion operations (phosphatization, oxalization) where soap lubricants and solid lubricants are concerned. In the large scale production of nuts, screws, bolts and similar parts on multistage presses, the extrusion oils in oil circulation systems have an important lubricating and cooling function. In principle, coating the wire surface is not necessary for simple operations, especially in the case of wire parts mass produced by upsetting. Frequently the lime and dry soap film of lubricant left from the wire drafting or the remaining layer of phosphate will be adequate. As a result this can be of particular significance when determining the lubrication for wire drawing in respect of the later extrusion. Of particular significance is the lubrication with EP oils, especially when working with wire without surface pretreatment of the blank metallic shearing surfaces. In the case of tools with extremely long service life, the forming is possible with the majority of highly active EP substances. An example of this is the production of inner square socket-head screws.

Apart from lubricating properties, the extrusion oils must have high thermal stability; the oil is overheated locally on the extrusion bodies and tools and temperatures over 90 °C can occur in the circulating system. The extrusion oil also has an essential cooling function, especially in the case of high speed automatic machines. No adhesive residue must remain as a result of the temperature stress as these could cause irregularities in operation. Further demands include low evaporation, little odor nuisance and a low tendency to generate oil mist. Recommended is the use of oils which develop little oil mist as well as oils with particularly low evaporation.

Extrusion oils are preferred for flooding or spraying workpieces and tools. If the oil is subject to such high pressure in the forming operation to cause self-ignition due to the Diesel effect, this can lead to the destruction of the tool. However, this can be best avoided through tool design, and only influenced to a certain extent by oil selection.

The viscosity of extrusion oils is between 30 and 120 $mm^2 s^{-1}$ at 40 °C. The preferred viscosity range is between 35 and 65 $mm^2 s^{-1}$ at 40 °C. Selection criteria for the initial viscosity are the workpiece temperatures, size of the extruded parts, machine pumping facilities and the specific criteria for the transfer between the working stages, as well as the thickening effects as a result of enrichment by abraded phosphate particles where phosphatized material is concerned. In some cases subsequent follow-on cutting and non cutting operations will be necessary with extrusion oil and in this case a compromise has to be found with regards to both viscosity and additives.

EP additives containing sulfur and chlorine play a role in extrusion oils. Additives containing chlorine have lost their significance in Europe due to the environmental problem described earlier. Apart from the sulfurized fatty oils there are also other products in the polar additive sector such as thermally stable synthetic esters (also as base oils). In general, the difference between extrusion oils and non-water-miscible oils used for cutting operations lies in the high additive content. Sulfurized mineral oils, sulfurized fatty oils and polysulfides play a special role as sulfur carriers. Zinc dialkyldithiophosphate has favorable effects on the surface roughness of the workpieces [15.103]. Alkyl and arylphosphoric acid esters and even phosphoric acid partial esters are also used [15.104]. Because the thermal stress is considerable when using chlorinated products particular attention must be paid to their stability and the risk of corrosion on machines and extruded parts. A degreasing of the parts directly after extrusion is recommended when using strongly chlorine containing oils.

In rare cases even non oil soluble EP additives are used in suspensions; we especially want to mention here elementary sulfur and zinc sulfide. Sedimentation must be taken into account in the case of unstable suspended substances in storage and when the circulation system comes to a standstill.

In practice, extrusion oils are assessed firstly by the surface roughness of the workpieces, but more frequently by observing the service life of the tools. To be considered, especially when determining the service life, is the material spread (both on the tool and workpiece side), for example, in screw production spread has been observed between 15 000 and 60 000 parts which was not caused by oil.

For the most important areas of application, extrusion oils are classified by type and amount of additive into four classes [15.105]

- standard screws and high tension hexagonal bolts: oil with polar additives and EP additives on a phosphorus basis (also suitable for non ferrous metals)
- larger size bolts and hexagonal nuts produced from zinc phosphatized wire on multi-stage presses: oils with polar EP additives, copper active
- cylinder bolts with an inner hexagon recess or inner toothing made of wire on multistage presses (larger dimensions): oils with an active proportion of polar additives and EP additives on a sulfur basis, copper active
- high tensile rust and acid resistant steels subject to high deformation: maximum alloyed oils with a very high share of EP additives on a (chlorine) sulfur and phosphorus basis, copper active.

Thermally stable, biodegradable ester oils with excellent tribological characteristics will gain more significance in the future.

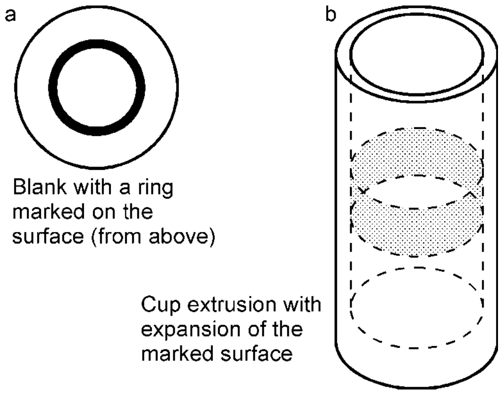


**Fig. 15.60** Determining the surface expansion for hollow backward cup extrusion.

### 15.4.3.4 Phosphate Coatings and Soap Lubricants for Cold Extrusion of Steel

It only became possible to extrude steels by the application of phosphatization by Singer (German patent 1934), if very simple upsetting and extrusion operations are ignored. As a consequence, phosphatization has remained the most important method of surface treatment for cold extrusion of steel up until today and has undergone further technical development in conjunction with other lubricating systems. Particularly favorable is the ability of the layers of zinc phosphate to go along with large expanding surfaces without breaking away when extruding. It has been proved that phosphate layers do not lose their release and lubricating properties even in the case of 20-fold surface expansion.

The surface expansion is not consistent over all the shaped surfaces but depends on the geometry of the part and especially on the geometry of the tool. In the case of backward cup extrusion – hollow backward extrusion – the structure of the high base can have a decisive influence on the surface expansion spread. If, in the case of cup extrusion, the workpiece under the die is shaped to a lesser extent, then the major proportion of the phosphate and the lubricating layer thicknesses remain so that there is little available to lubricate the wall of the cup. In this case obvious improvements are given by more extensive corner rounding on the die but a half round die is even more effective. The extent of the surface expansion can be determined by

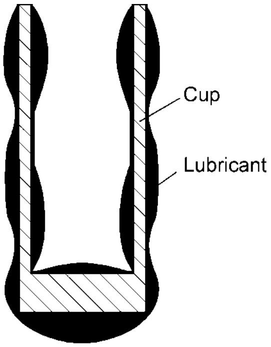


**Fig. 15.61** Lubricating layer profile on a cup after cold extrusion.

marking a ring area on the end of the blank (Fig. 15.60). Gravimetric methods can be also applied to determine the layer profile of the phosphate and the lubricating layer. Marking the zinc phosphate layer with radioactive phosphorus is another way of studying surface deformation. Figure 15.61 shows a lubricating layer profile on a cold extruded cup (container).

### Demands put on Zinc Phosphate Coatings

The high resistance to pressure, shearing and adhesion during extrusion are considered as positive. The phosphate coating must have a specific structure related to the type of lubricant used.

A fine crystalline coating approx. 5 to 7 µm is to be used for oil lubrication on multistage presses. Layers, which are too thick, lead to problems due to layers of phosphate building up on the tools. This system is preferred for parts with low piece weights such as screws, nuts, bolts or spark plug bodies produced on multistage presses.

When combining zinc phosphate and soaping, attention must be paid to the high reactivity of the zinc phosphate coating. High zinc and low iron contents are favorable for high turnover with soap. Layer thicknesses of 15 to 20 µm are applicable for both coarse and fine crystalline phosphate layers. This application is preferred for medium and large piece weights and large surface expansion. Typical examples are larger cups reduced by backwards hollow extrusion. If structure-effective solid lubricants (especially graphite and molybdenum sulfide) are used in connection with a zinc phosphate coating then the thickness of the phosphate layer is determined by the method employed to apply the solid lubricants. When applying solid lubricants from an aqueous suspension, preference is given to 10 to 20 µm thick coarse crystalline phosphate coatings. Fine crystalline layers, approx. 5 µm thick are more favorable when applying solid lubricants as dry powder lubricants by drums. Typical applications are small to medium piece weights with extreme stress and sharp edged geometry [15.105].

### Reactive Soap Lubricants

Alkali soap in connection with a zinc phosphate coating has gained particular importance. In this case, not only the absorptive and chemically absorptive binding of the lubricants to the workpiece surface is neutralized but there is also considerable chemical conversion of the soaps with the zinc phosphate. One also talks about reactive lubricants with this type of soap lubricant. The reaction of the alkali soap in the aqueous solution with the zinc phosphate coating is according to the following formula:

$$\mathrm{Zn_3(PO_4)_2 + 6CH_3(CH_2)_{\mathit{n}}COONa \rightarrow 3Zn\,[CH_3(CH_2)_{\mathit{n}}COO]_2 + 2Na_3PO_4}$$
$$\text{zinc phosphate + sodium soap} \rightarrow \text{zinc soap + sodium phosphate}$$

This conversion not only takes place with the development of the zinc soap on the surface coating of the phosphate layer but also occurs deep in the pores. Some 50 to 60 % of the phosphate coating can be converted depending on temperature, dwell time, concentration and the developing phosphate layer. The type of soap also plays a considerable role in this respect. Salts of stearic acid are more reactive than the salts of oleic and palmitic acid.

The zinc soap developed by the reaction leads to lower coefficients of friction when forming and the firm anchoring with the phosphate layer is particularly favorable for the lubricating operation. Figure 15.62 shows the basic structure of the lubricant layer in its combination of zinc phosphate and soap. A three layer structure comprising zinc phosphate, zinc soap and alkali soap over the basic metal is provided when soaping. A mixture of zinc phosphate, non converted alkali soap and phosphate layer debris is given over the leveled phosphate layer after forming. This mixture creates a so-called phosphate mirror on the surface of the workpiece as a result of the forming operation. If the formed parts should intermediately be annealed for further extrusion stages then this phosphate mirror should be removed. This is done with aqueous alkaline cleaners and a subsequent pickling operation or with special cleaners in a single stage operation.

The reactive soaps are supplied by the manufacturers in powder or flake form and mixed by the user with water to provide 2 to 10 % soap solutions, depending upon the degree of difficulty. The parts are soaped by immersion over a period of 2 to 6 min at bath temperatures from 60 to 85 °C [15.106–15.108].

As far as small parts are concerned this can be carried out after phosphatization without changing the drum. After soaping, sufficient time must be allowed for drying. Should the heat of the parts not be adequate for this purpose hot air has to be used. As a general rule, storage of the soaped parts for a few hours before forming has a favorable effect. Too heavy soaping has a detrimental influence on the edge geometry of the workpieces, because remains can be left in the tool. The average layer weight of the non-converted soap is between 1 and 5 g $ml^{-1}$. Apart from the alkali soaps, the reactive soap lubricants contain further additives to improve the lubricating properties and increase the alkalinity, as well as filling substances, such as talcum and other solid lubricants and suspension auxiliaries where necessary.

If, during the immersion process the parts remain in the soap bath too long due to the operation coming to a standstill, too much of the phosphate coating can become detached which is a disadvantage, for both the forming operation and to the

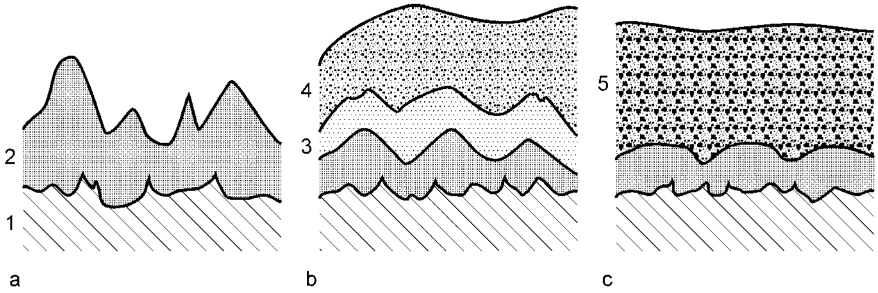


**Fig. 15.62** Schematic layer structure of reactive soaps in connection with zinc phosphate coatings for cold extrusion lubrication: (a) phosphatized blank; (b) phosphatized and soaped blank; (c) cold-extruded workpiece. 1, base metal; 2, zinc phosphate; 3, zinc soap; 4, alkali soap; 5, mixture of alkali soap, zinc soap and phosphate debris.

service life of the bath. Products have been developed for such a case in which the reaction is retarded after a specific period of time. Some of these products are soaps which contain, for example, alkali phosphates, such as trisodium phosphate and anion active phosphate compounds. The service life of the bath can be increased by desalination, for example, through centrifuging. The enrichment of non-centrifugible substances, e.g. sodium phosphate, leads to the layer thickness of the soap coating being reduced and determines when the bath has to be changed.

#### 15.4.3.5 Solid Lubricants for Cold Extrusion of Steel

Although soap lubricants in conjunction with zinc phosphate coatings are preferred for medium to larger sized parts, structure-effective solid lubricants, especially molybdenum sulfide ($MoS_2$), are used for smaller parts.

As a general rule the solid lubricants are applied to a phosphate layer as dry powder lubricants by tumbling or from aqueous suspensions by immersion or spraying. Application by immersion follows in dipping baskets or rotating screen tubs [15.105, 15.109–15.112].

In a few cases, solid lubricants can be applied as suspensions in mineral oils, synthetic oils or even solvents. In special cases, this method can solve wetting problems better than aqueous suspensions. In general, however, the aqueous dispersions offer considerable advantages as carrier fluids as a result of their environmental friendliness, especially when compared with organic solvents, and can also be better integrated in the wet treatment of workpieces.

#### Tumbling

After drying the phosphatized parts, dry powder lubricant on the basis of $MoS_2$, and in some cases even graphite and other solid lubricants, are applied to the surface in rotating drums. Average layer weights of 10 to 25 g $m^{-2}$ is recommended for $MoS_2$ lubricants.

Through the mechanical stress in the tumbling operation the phosphate layer is leveled and phosphate layer debris is produced which mixes with the excessive powder lubricant to make it unserviceable. As a result, wear resistant phosphate layers with minimal surface roughness are required which, as a general rule, are thin fine-crystalline and dense phosphate layers approx. 5 μm thick.

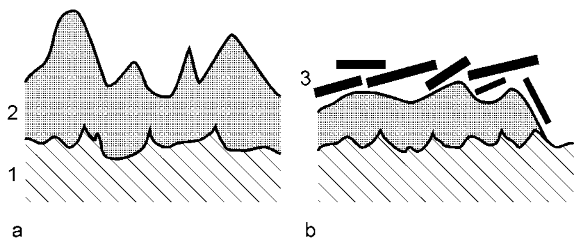


**Fig. 15.63** Tumbling powder lubricants with spraying of the phosphate layer surface and complete abrading of the phosphate layer on the edges of the workpiece: (a) blank after phosphatizing; (b) blank after tumbling of $MoS_2$ powder lubricant. 1, basic metal; 2, zinc phosphate; 3, $MoS_2$ powder lubricant applied by tumbling.

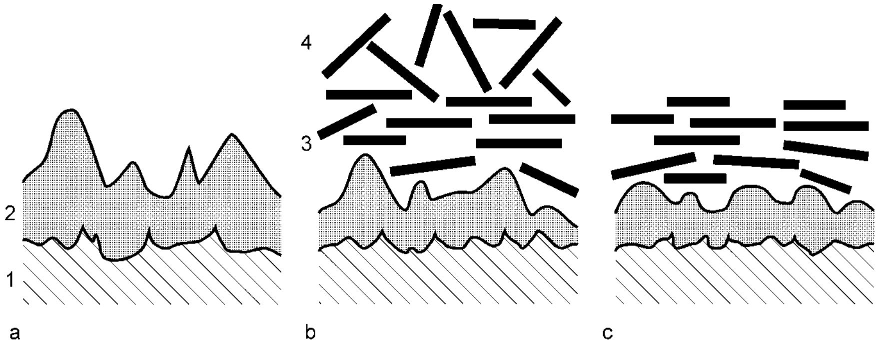


**Fig. 15.64** The application of solid lubricant on a phosphatized surface from an aqueous suspension: (a) surface after phosphatizing; (b) surface after immersion in suspension; (c) surface after removing non-oriented $MoS_2$ particles using drums after immersion and drying. 1, basic metal; 2, zinc phosphate; 3, oriented $MoS_2$ particles in organic binder; 4, non-oriented $MoS_2$ particles.

As a result of the tumbling process, the lamella solid lubricants are pressed in the direction of the preferred crystallite level parallel to the workpiece surface. One disadvantage is the frequent total destruction of the phosphate layer (Fig. 15.63). In rare cases parts treated by soaping are also tumbled with dry powder lubricants.

### Immersion in Suspensions

The wet application of a solid lubricant in an aqueous suspension can be easily integrated in the pretreatment phosphatizing process and can be carried out instead of soaping. However, while it is possible to use the same drum for soaping as was used for phosphatizing, in general, the drum is changed when immersion in an aqueous $MoS_2$ suspension takes places. After immersion, at lower drum speed and intermittent turning, the parts are dried, after which excessive, non-oriented lubricant can be removed while slowly turning the drum for a few minutes (Fig. 15.64). The coating weight is between 5 and 15 $g\ m^{-2}$ in this case.

As already mentioned, the pretreatment process for applying a lubricant carrier layer and applying the lubricant itself represents a considerable part of the total costs where cold extrusion is concerned. As a result continuous efforts are being made to rationalize this expensive process and, for example, there has been success in phosphatizing and applying $MoS_2$ layers in a single bath in one operation (molyphosphatization), [15.111]. Figure 15.65 shows such a rationalized process flow in comparison to a conventional procedure with soaping [15.113].

By optimizing the procedure, through new tool materials and coatings as well as new high capacity lubricants (special ester oils, for example) it has been possible to do without expensive phosphatization and the application of solid lubricant in a few cases (Fig. 15.65c).

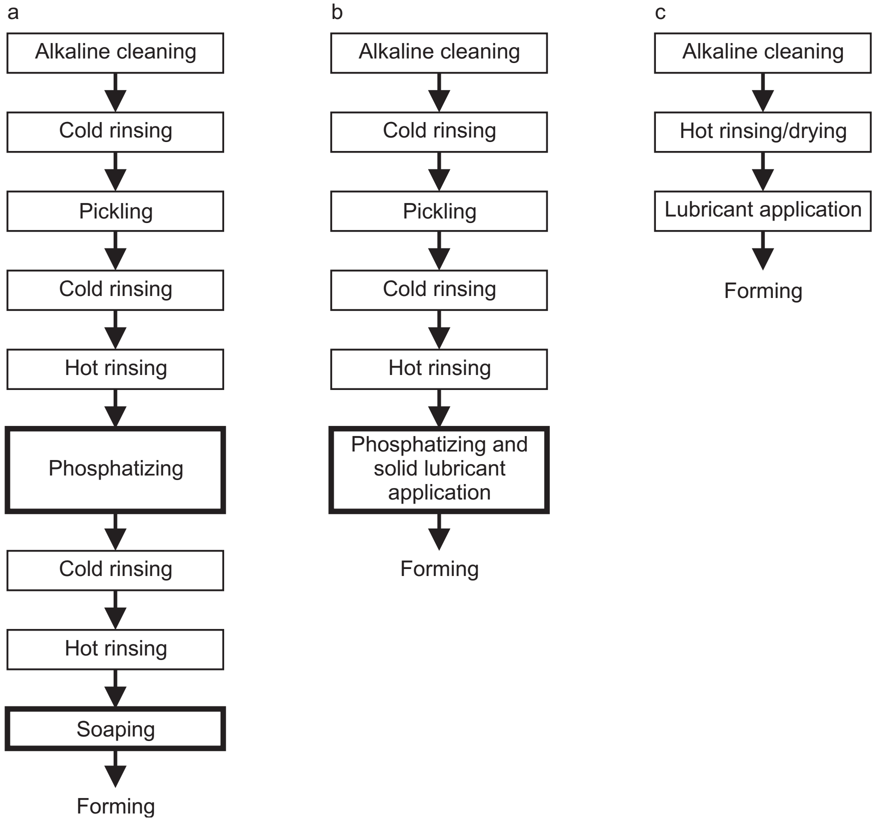


**Fig. 15.65** Process flow in three processes for the surface treatment of steel for cold extrusion: (a) conventional working flow for the combination zinc phosphate/reactive soap lubricant; (b) rationalized procedure for the combination phosphate/$MoS_2$; combining phosphatizing and solid lubricant application; (c) simplification of the procedure with 'super lubricants'.

### 15.4.4
### Warm Extrusion and Forging

The yield stress decreases with the temperature increase in the workpiece material and as a result the possibility of achieving lower forming forces and higher degrees of workability is achieved. This effect is utilized to some extent through the existing forming heat (up to 250 °C) even when cold extruding (without preheating the blanks), especially when working on high speed multistage presses. Figure 15.66a shows the change in yield stress dependent on deformation and temperature.

Figure 15.66b shows the reduction in forming force with the temperature of the workpiece when extruding a hat nut. Workpieces with higher tensile strength are also extruded by warm forming. However, the tribological problems which occur are clearly greater than given when cold extruding. Some warm extrusion processes

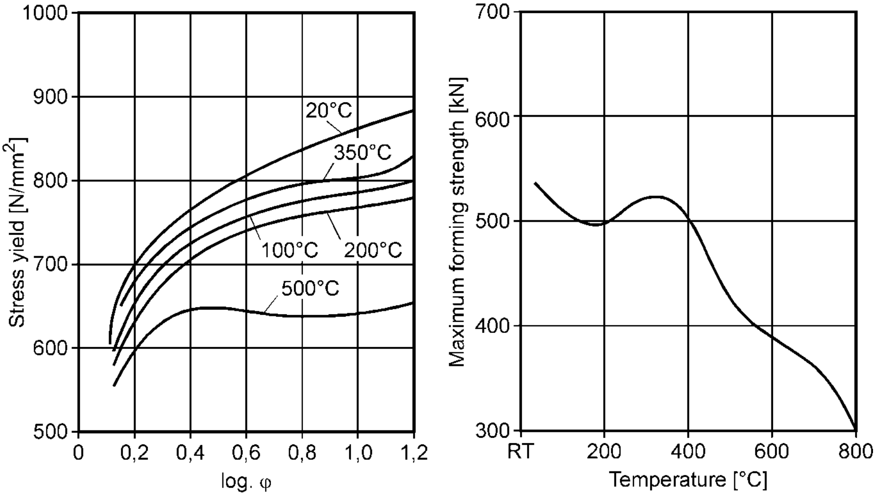


**Fig. 15.66** Warm forming: (a) Flow curves of the workpiece 16MnCr5 in relation to the temperature [15.114];
(b) Dependence of the forming force on the workpiece temperature during the lubricated extrusion of a hat nut [15.115]

lead to high tool wear and low tool service life which is only about one third of the value compared to cold extruding. This is why continuous attempts are being made to find ways of assessing the lubricant better and quicker than is possible using a test based on the tool service life. Warm forming almost covers the temperature range between cold extruding and the recrystallization temperature and in the case of steel materials this lies between approximately 300 and 800 °C. These limits for other materials such as aluminum, for example, can be very much lower.

Warm forming has the greatest significance for high alloyed steels and special materials. The method is applied when the parts cannot be cold formed or when it is possible to ensure more economical production by the warm process by less forming stages. Over and above this, the good surface quality and dimensional accuracy of the workpieces, which cannot be achieved by warm forming, is fully exploited (precision forging).

A phosphate layer is expedient at temperatures under 400 °C – as when cold extruding. Moreover, as a general rule the forming forces are so low at temperatures above approximately 400 °C that the conversion layers are no longer necessary and the main consideration for the selection of a lubricant lies more on a lower coefficient of friction and higher resistance to temperature. In many cases lubrication is necessary for the warm forming process because of the high production speed, high tool stress and the required precision of the parts and the workpieces (billet coating) and the tools (die lubrication). Billet coating in the case of some materials also has the task of protecting the surface of the material against oxidation.

When classifying lubricants according to the temperature of the preheated blanks it must be considered that the temperature load can be very different with different machines and application conditions. The tool temperature can have less significance than the workpiece temperature when the lubricant is applied onto the tool surface. Consequently, it must be considered that this can only provide a rough orientation for the breakdown by temperature classes [15.115–15.118].

The failure of lubricants due to temperature can be determined in a ring upsetting test, in accordance with Fig. 15.57, for example.

#### 15.4.4.1 Temperature Range up to 350 °C

Zinc phosphate coatings serve as carrier layers; the most important fluids are mineral oil based oils with a high flash point, polyglycols and polybutenes; they can be applied as loss lubrication and at temperatures up to about 300 °C as circulating lubrication. Solid lubricants can also be suspended in the above-mentioned oils; in this case mainly $MoS_2$ and graphite. Aqueous $MoS_2$ and graphite suspensions are also suitable. The surface temperatures of the tools should be over 150 °C to facilitate quick water evaporation. The application of the lubricant must not contribute to excessive cooling of the workpieces.

#### 15.4.4.2 Temperature Range 350 to 500 °C

Those most used in this sector are graphite dispersions in water or organic carrier media. In this case one intentionally accepts the decomposition of synthetic polymer oils if necessary. In this case thermal resistance limits are set for $MoS_2$ lubricants. Water soluble salts with melting points in the region of 300 and 400 °C have proved successful in some processes.

#### 15.4.4.3 Temperature Range 500 to 600 °C

Here again aqueous graphite dispersions are given preference and applied by spraying. Decomposition of the solid lubricants graphite and $MoS_2$ can be prevented when these are mixed with boric oxide powder ($B_2O_3$). The solid lubricants are in the melt before oxidation as a result of the boric oxide melting at 460 °C [15.119].

#### 15.4.4.4 Temperature Range > 600 °C

In this case the lubricants and the application technology are similar to those applied for the hot forging process. Factors affecting the choice of lubricant, among other things, are also tool temperature and dwell time in contact with the hot workpiece: aqueous graphite dispersions, graphite dispersions in water-soluble organic carriers and glasses, in so far as their difficult removal can be accepted. As well as graphite, zinc sulfide has also proved successful as a solid lubricant because of its particular resistance to temperature in individual cases; it is also used in operations with graphite and water soluble organic carriers such as polyglycols. The warm extrusion of steel has gained certain significance for manufacturing small mass produced parts on multistage presses in a temperature range between 500 and 700 °C. When starting with a graphite coated wire, the blank shearing surface given after the shearing stage can be coated by spraying with graphite suspensions before entering the forming stage. This calls for a highly developed spray technology synchronized with the machine kinetics.

To demonstrate the complexity of warm forging at 750 °C, Fig. 15.67 shows the schematic layout of a complete warm forging line [15.152, 15.153].

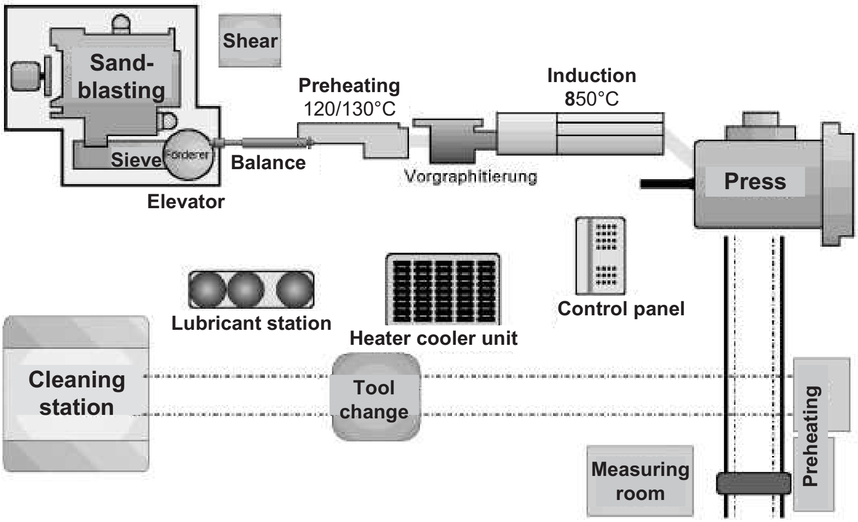


**Fig. 15.67** Warm forging: The schematic layout of a complete warm forging line [15.152, 15.153].

### 15.4.5
### Lubrication when Hot Forging

The hot workpiece material (in the case of steel approximately 1200 °C) flows into the cavities of the die during the forming operation (Fig. 15.57). This is carried out to some extent at a high relative speed which depends, firstly, on the speed of the striking tool (hammer or forging press) and, secondly, on the die. Frequently the material flow runs in the following sequence during a forming operation (Fig. 15.55):

- free upsetting without large sliding movements
- widths with preferential material flow vertical to tool movement and large sliding movements
- rise generally as last phase after the material has run into the flash gap which generates considerable resistance in width direction. The cavities of the die form are filled and material flow is generated under considerable tool stress against the direction of the tool movement. Relative movements also occur in this case with high friction losses and comparably high tool wear.

Figure 15.68a shows the different tool loads and Fig. 15.68b explains the influence of friction on the flow resistance in the flash gap. The material flow, and as a result also the die filling operation, can be influenced both by the flash ratio $b/s$ and the lubrication. This is demonstrated in Fig. 15.69 where too low friction as a result of good lubrication in the flash gap has caused too great material flow in the flash and has prevented the cavity of the die from being filled as a result of too little rise. In the case of steel one works with flash ratios from 5 to 10, in which case a large rise becomes necessary.

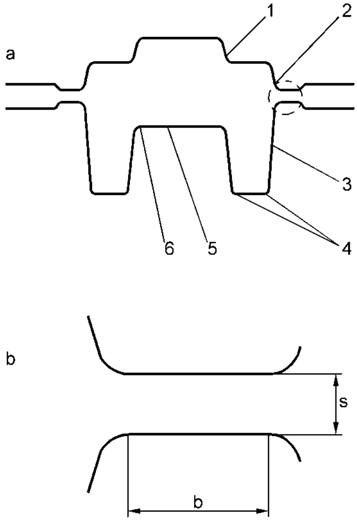


Fig. 15.68 Tool stress when impression die forging [15.120]. (a) load areas: 1, 2 and 3 are areas of high relative speed between die surfaces and workpiece material; particular risk of abrasive wear; 4, endangering of the die because of the Diesel effect in especially deep areas of the cavities, e.g. when inadequate oils or organic solid-lubricant carriers are used; 5, material fatigue as a result of overheating and thermal load; 6, plastic deformation of the die material. (b) influence of the friction in the flash gap: $P_f = 2\mu k_f b/s$, where $P_f$ is the flow resistance, $\mu$ the friction index, $k_f$ the yield strength, $b$ the flash width, $s$ the flash gap width, and $b/s$ the flash ratio.

### 15.4.5.1 Demands on Hot Forging Lubricants

The demands put on die lubricants can be summarized as follows:

- good lubricating properties which optimize the material flow, favor accurate filling of the die forms and can reduce the tool wear at those points with great relative movement and high specific pressures can reduce the forming force;
- good release properties which favor workpiece release from the die after forming and prevent sticking;
- good propellant effect, whereby, through the development of gas, generally through pyrolyses of the lubricant contents, high gas pressures are built up in the die; apart from the mechanical separating effect of the lubricant layer it is also possible in particular to avoid sticking in the deep cavities of the die form by means of this propellant effect;

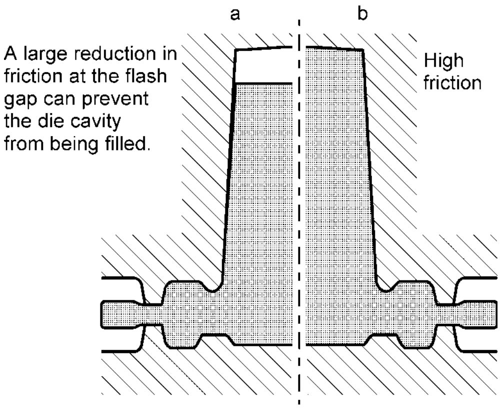


Fig. 15.69 The influence of friction on die filling [15.121].

- cooling and insulation effect: cooling is required by especially high thermal tool stress which above all can be achieved by aqueous lubricants. An insulating effect by the lubricant layer is desirable to reduce the heat transfer between workpiece and the die block during the forming operation, but is only achieved to a modest extent with the usual lubricants;
- no trouble at the place of work through evaporating or decomposing parts of lubricants (no risk of fire, no odor nuisance, no vapors harmful to health);
- no corrosion of tools and other machine parts,
- no residue (lubricant built-up) in the dies even after longer use;
- no tendency towards Diesel effect, which causes tool damage, especially in deep cavities of the die form;
- simple and economic application, even with due consideration to modern workplace conditions and the use of automatic application methods.

To fulfill these demands the lubricants must have the main properties:

- uniform wetting of the surface even at high forging surface temperatures; good wetting even in the case of geometrically difficult and very inaccessible cavities of the die form;
- high thermal stability;
- fast development of a consistently thick and closed surface film with good adaptation to the respective given die temperatures.

#### 15.4.5.2 **Lubricant Testing Methods**

Figure 15.70 provides an overview of testing methods for die lubricants. From 500 to 1000 forging operations are necessary to determine the wear behavior.

### 15.4.6
### Hot Forging of Steel

Workpiece temperatures are between ca.1100 and 1200 °C and die temperatures preferably between 150 and 300 °C (maximum up to 500 °C). A temperature, which is about the arithmetical average of the die and workpiece temperature, possibly also about 50 °C above this, is given for the main proportion of lubricant layer during the forming operation. The temperature of the lubricant layer is between 725 and 775 °C at a die temperature of 250 °C and a workpiece temperature of 1200 °C. Since lubricant reactions caused by heat and dependent on time follow according to the reaction kinetics rules, the contact time due to the process between the hot forged part and lubricant is particular significant (50 to 100 ms when working on forging presses). A build-up on the forging surface (oxide, nitrite) caused by the workpiece can play a significant role in connection with lubricant layer adhesion.

#### 15.4.6.1 **Lubricants**

Graphite preparations have gained outstanding significance. Contributing to this are the advantages of graphite with regards to its adequate resistance to temperature, its economy in use and especially its inert behavior from the medical and toxicological

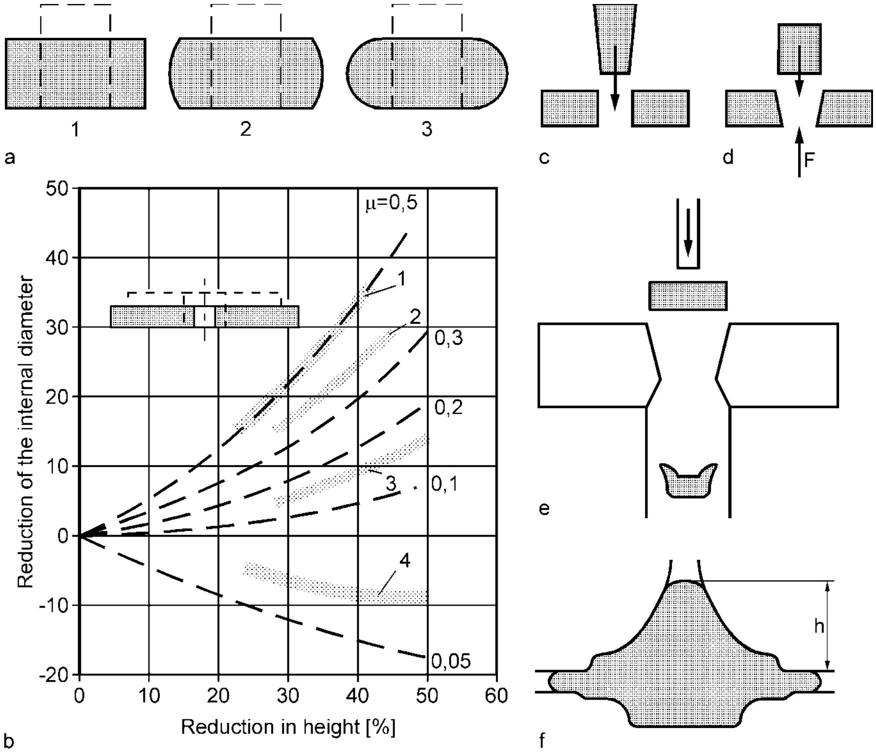


**Fig. 15.70** Testing lubricants for hot forging. (a) Upsetting test with a cylindrical test ring between two level plates: 1, low friction; 2, intermediate friction, adhesion without relative movement of the surfaces. (b) Ring upsetting test (see also Fig. 15.57): 1, without lubrication; 2, 3, and 4, various lubricants [15.109]. (c) Pressing a conical body into a cylindrical bore. (d) Pressing a cylindrical body into a conical bore; c and d show the fixing of the workpiece into the die cavity. Assessment criterion is the force $F_A$ which is required for expressing the test body out of the bore. (e) Test set-up for lubricant assessment by friction and wear measurement according to E. Doege and R. Melching [15.122, 15.123]. (f) Assessment of lubricants by die filling; measurement of the fill height, *h*.

point of view. Colloidal preparations in oil and in water as well as pasty graphite lubricants are used. Graphite preparations have replaced lubricants such as saw dust, heavy oils or powdered carbon to a very wide extent in lubrication technology.

Where graphite preparations are concerned water based suspensions are to be found at the top of the list. The wetting problems given in the case of aqueous graphite suspensions, especially at high forging temperatures, can be solved in that an organic phase with appropriate surface active substances is incorporated as an emulsion phase or even as a real solution.

The propellant effect can be improved through inorganic salts (alkali carbonate and bicarbonate) or through organic substances in aqueous solution or by dispersion. Bonding agents frequently on silicate or borate basis are used to form surface films.

**Tab. 15.17** Water-based forging lubricant [15.124].

| *Substance designation* | *Weight (%)* | *Additive function* |
|---|---|---|
| Sodium carboxymethylcellulose (CMC) | 0.77 | Thickening agent, suspension aid |
| Aqueous 30 % graphite suspension | 36.60 | Lubrication and separation |
| Sodium molybdate | 5.00 | Lubrication and separation in meltflow; corrosion protection, bonding agent |
| Sodium pentaborate | 3.18 | Wetting, film development with lubricating effect,<br>good adhesion to metal, bonding agent |
| Sodium bicarbonate | 4.83 | Propellant effect through $CO_2$ development, lubricating film development, wetting, reduction of scale development |
| Ethylene glycol | 9.02 | Propellant effect, reduction of scale development, reduction of scale adhesion, antifreeze |
| Water | 38.60 | Carrier substance, die cooling |

Table 15.17 shows one example for the composition of a water-based forging lubricant with the functions of the individual additives [15.124].

If the die temperature is clearly under 200 °C or even under 150 °C when the lubricant is applied it may well be that no film can be formed by the aqueous dispersion, depending on the available evaporation time. In such cases organic carriers can be used which either evaporate quicker or form an oily film. Care must be taken when selecting synthetic polymer oils that no toxic monomers develop during the depolymerization process during contact with the hot forging. In individual cases pasty graphite preparations are still being used as well as the oily products. In individual cases white forging lubricants are being tested in use. In such cases the graphite is replaced either by other solid lubricants (boron nitride, zinc sulfide) or even inorganic salts (silicate, phosphate). If a lubricant layer from a real aqueous solution of inorganic or organic substances can be applied, this has an advantage over many solid suspensions because no settlement can occur. Besides this advantage in application the non-coloring properties of this solution, which is free of graphite, have also promoted the use of such products. However, it must always be borne in mind that the inert graphite is completely harmless from a toxicological point of view and that white forging lubricants must not have a detrimental effect on workplace conditions as a result of toxic decomposition of substances in the product [15.125–15.129].

### 15.4.7
### Aluminum Forging

Gaining increasing significance are parts produced by massive forming from aluminum and aluminum alloys. Playing a role in this respect are low weight and the excellent mechanical properties. Because of the major adhesion problem, the technology in the usual mechanical presses with high deformation rates has still not been completely developed and there is a considerable need for development, as far as tribological parameters are concerned. The most extensively used lubricants are water-based graphite dispersions [15.130–15.133].

The use of white lubricants with no graphite content is being promoted for some aluminum-forging applications for workplace-cleanliness reasons, but in this case the tribological problems in respect of abrasion wear and galling have become worse. Oil based dispersions are also used here and there. The oil components not only take over the carrier and cooling function but make a considerable contribution to the reduction in friction and wear. A combination of the water and oil phase has been realized in water emulsions with oil containing graphite. These products are used very little. Water-based wax emulsions are used for special forging processes, for example, for isothermal forging of aluminum alloys in the aircraft industry, amongst other applications.

Considerable importance is placed upon tool coating which has already been considerably developed for extrusion tools (TiN, TiAlN, TiCN) [15.134, 15.135].

### 15.4.8
### Isothermal and Hot Die Forging

The production of parts made of titanium alloys by forging has gained particular significance as a result of developments in the aircraft industry and considerable growth rates are to be expected here in the future.

Isothermal forging is applied, amongst other processes, as a forging method in which not only the workpiece but also the tool is heated to the same high temperature. Unlike normal forging, the forming force is frequently steadily transferred by means of hydraulic presses to the workpiece over longer contact periods. A calibration operation with similar lubricant problems often follows the actual forging operation. $\alpha$-$\beta$-titanium alloys are formed over a temperature range of approx. 900 to 980 °C, $\beta$-titanium alloys between 700 and 850 °C.

Considerable demands are put on the thermal stress of the lubricants, since the lubricating film is subject to high temperature both on the workpiece and on the tool.

Apart from the demand for thermal stability the used lubricants must not cause high temperature corrosion on the surface of the tool or workpiece. The risk is particularly great as a result of the development of lubricant structures in the tool.

Frequently there is no tool lubrication and the lubricants are applied to the workpieces in the water phase or by organic carrier liquids. Glass, in which solid lubricants with effective structures or abrasive hard components such as titanium carbide (1 to 8 μm) are embedded, is an effective lubricant [15.136–15.138].

Examples of formulas for lubricants for forging $\alpha$-$\beta$ titanium alloys are (the figures are percentage by weight):

| | |
|---|---|
| 35 | Glass substance ($SiO_2B_2O_3$, CaO, FeO) |
| 3 | Titanium carbide (TiC) |
| 54 | Organic carrier liquid |
| 8 | Acrylic resin bonding agent |

or

14 Boron nitrate

86 Glass substance (67 $B_2O_3$, 33 silicate glass)

The glass forms a viscous layer on the hot surface for lubrication and protection against oxidation. Viscosities of 20 000 to 100 000 mPa s have proved successful at forging temperatures.

Isothermal forging is also used for some aluminum alloys [15.139].

### 15.4.9
### Application and Selection of Lubricant

In this case spray application is in the foreground. Modern application by spraying must be permitted by all lubricants in the four main groups: pigmented (inorganic), white (organic), mixed systems (organic/inorganic) and emulsions (organic) [15.129].

H. Seidel [15.152] summarizes the lubricant application for complex pieces as follows: Where large or complex pieces have to be formed, forging lubricants (in a ready-for-use state) are normally applied by:

- spraying
- showering
- dipping
- flooding

Simple methods of lubricant application, such as swabbing or brushing, need no explanation. The most widely used method of applying lubricants to tools is by spraying. Dipping or showering is normally preferred as the method of application where pre-treatment of the parts is required, e.g. billet coating in the warm forging process. Flooding (or hose cooling) is used on high speed forging machines where the lubricant also provides a cooling effect.

Because of the demands made on a forging lubricant, they cannot be viewed simply as a lubricant like greases or oils. The means of application is not so simple. In fact, it would be reasonable to suggest that in many instances, the method of application is as important as the materials it is applying. Figure 15.71 indicates the different types of spray application.

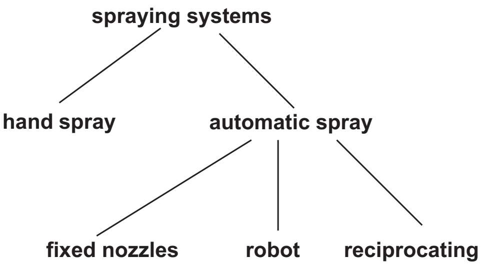


**Fig. 15.71** Spray techniques in die forging [15.152, 15.153].

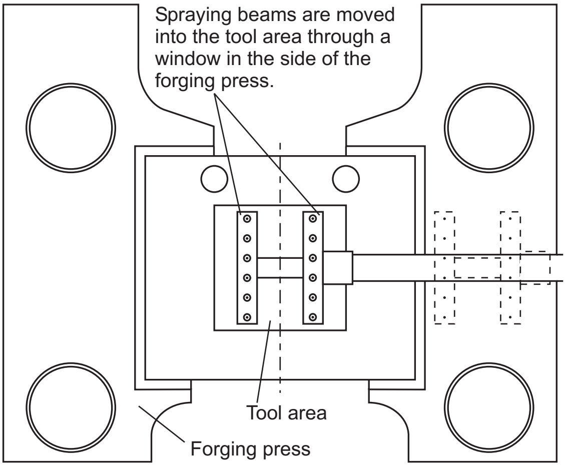


**Fig. 15.72** Automatic spraying facility for applying forging lubricants.

Modern spray equipment is extremely complex and even in the most exacting working environment, this complexity of pumps, valves and spray-heads must work reliably. With the advent of electronics, spray systems can be controlled with great accuracy. Even blocked jets and pipes (traditionally associated with dispersions containing solids) can virtually be eliminated by careful design (e.g. by-pass pipes). This means that the fluid can be constantly circulated between cycles even up into the spray nozzles. (N.B.: Particularly important in fully automated forging processes).

Figure 15.72 shows such a basic automatic spraying facility. In this case the spraying beams are traveled into the tool area through a window in the side of the forging press; both the cavities of the die form are cleaned with air and the forging lubricants sprayed by the nozzles. The thickness of the developed lubricant film can be controlled to the greatest extent by the lubricant concentration and the spraying time. It can be very helpful to optimize the nozzles in trial runs. The lubricants must be stable or be at least slightly homogenizing before dilution in delivered state. Water based lubricants must be protected against bacterial attack in recirculating systems and storage.

**Tab. 15.18** Forging lubricants recommendation for various materials [15.140].

| *Material* | *Pigment* | *Carrier* | *% of pigment* |
|---|---|---|---|
| Aluminum | Graphite | Oil solvent, water | 5–15 % |
| Aluminum, brass | Graphite | Solvent, light oil, water | 2–8 % |
| Aluminum, brass, carbon steels | Graphite or other pigment | Water, solvent, oil | 2–8 % |
| Carbon steels, high-strength alloys | Graphite | Water, oil | 2–8 %, also 2–12 % |
| Superalloys, titanium | Glass (ceramic) and graphite | Alcohol, water, other solvents | Graphite 2–8 %<br>Ceramic used as received |

Table 15.18 provides an overview of the forging lubricants for different materials. Given in this rough overview are details of the pigment, carrier and the most frequently used concentration [15.140].

The most controversial discussions in the field of forging lubricants concern graphite-free white lubricants. In this case, cleanliness at the place of work in particular is in the foreground. The development of such products has been strongly promoted in the last years.

This group of products has gained a considerable share of the market despite some tribological deficiencies compared with graphite dispersions. Observations on system costs, in which the disposal costs have been considered, shows a clear advantage in costs for the classical graphite-in-water-lubricants [15.129].

The lubricant systems for the warm and hot forging applications is shown in Fig. 15.73 (product lines of FUCHS LUBRITECH GMBH (LUBRODAL), one of the leading specialists for forging lubricants).

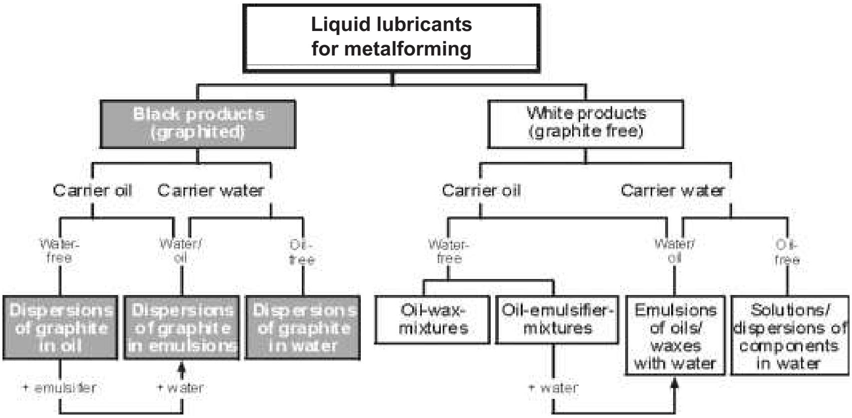


**Fig. 15.73** Liquid lubricant systems for warm and hot forging [15.152, 15.154].

# 16
# Lubricating Greases

*Wilfried Dresel and Rolf-Peter Heckler*

## 16.1
## Introduction

### 16.1.1
### Definition

Lubricating greases can be defined as solid to semi-fluid products of the dispersion of a thickening agent in a liquid lubricant [16.1]. They usually contain additional compounds to impart special properties, and usually the thickening agent is a metal soap. It is not easy to classify greases as either liquids or solids – the transitions are fluid. On the liquid side, oils containing << 5 % w/w thickening agents – in general polymers – already have structural viscosity, but not yet a yield point (Section 16.7), and therefore have been named non-fluid oils [16.2]. On the solid side suspensions of > 40 % w/w solid lubricants in oils are usually called pastes (Section 16.4). When they also contain thickening agents typical of greases, they are also called grease pastes [16.3].

It can, nevertheless, be said that greases in general contain from 65 to 95 % w/w base oils, from 5 to 35 % w/w thickeners and from 0 to 10 % w/w additives.

There is no chemical or physical need to describe synthetic greases or fully synthetic greases separately, but the terms should at least be explained. Many authors call a grease synthetic when the base oil is not a mineral oil, but a synthetic product, e.g. a carboxylic acid ester, a synthetic hydrocarbon, a polyglycol, a silicone, or a perfluoropolyether [16.4–16.6]. Sometimes the term fully synthetic grease is used when the thickener is also synthetic, e.g. the salts of amidocarboxylic acids with oligoureas.

### 16.1.2
### History

It can be speculated that grease-like lubricants were already known to the Sumerians and used by them in their wheeled vehicles from ca 3500 to 2500 BC [16.7]; it has also been stated that as early as 1400 BC the Egyptians used greases made from olive oil or tallow and lime to lubricate the axles of their chariots [16.8], although only pig fat was reported for that purpose by ancient authors such as Dioscurides and Plinius Secundus [16.9]. Probably the first grease of the industrial age was

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patented by Partridge [16.10] in 1835; it was a calcium grease, also made from olive oil or tallow. Greases based on mineral oils and thickened by soaps were probably first proposed ca 1845 by Raecz [16.11], and a sodium grease made with tallow was patented by Little [16.12] in 1849.

Two outstanding encyclopedic monographs have been written about the manufacture, and use or application of greases, the first in 1937 by Klemgard [16.13], the other in 1954 by Boner [16.14]. Both contain much general information that is still pertinent and valuable even today.

### 16.1.3
### Advantages over Lubricating Oils

In 1954 Boner [16.15] listed thirteen advantages of greases over oils in his famous book. In 1988 seven were still regarded as valid [16.16]; in 1996 Lansdown [16.17] mentioned only six advantages and looked at them rather differently (Table 16.1).

**Tab. 16.1** Advantages of lubricating greases over oils.

***1988***

1 Greases flow only under the influence of a force
2 Greases have lower friction coefficients
3 Greases cling better to surfaces
4 Greases are more water-resistant
5 The (apparent) viscosity of greases is less dependent on temperature
6 Greases operate over a wider temperature range
7 Greases act as seals against dirt and other contamination

***1996***

1 Greases have no stop–start problems
2 Greases perform better under squeeze film conditions
3 Greases solve sealing problems
4 Greases supply surplus lubricant without special design
5 Greases help to avoid contamination of clean products
6 Greases enable the use of solid additives

### 16.1.4
### Disadvantages

Compared with lubricating oils greases have only two disadvantages, they are not the right choice when heat transfer is a problem and they have a lower speed limit because of their higher effective viscosity. A third, rather theoretical disadvantage is that, because of their generally more pronounced ionic character and larger surface, they are more susceptible to oxidation than oils.

### 16.1.5
### Classification

Greases have been named, and are still being named, after the industry in which they are used, e.g. steel mill greases, after the kind of application, e.g. wheel bearing greases, after their prevalent application temperature, e.g. low-temperature greases, or after their range of application, e.g. multi-purpose greases. The meaning of the latter name has changed with the years [16.18] and the other names do not say very much about the quality of the performance of the greases in question.

The consistency of a solid to semi-fluid body is a very complex subject, but the penetration of one body into another one can be measured easily and with simple devices. Even today, therefore, greases are named according to the consistency classes defined by the US National Lubricating Grease Institute (NLGI) in 1938 and in accordance with the cone-penetration method developed in 1925 [16.19] (Table 16.2).

**Tab. 16.2** Classification of greases by NLGI numbers.

| *NLGI number* | *Appearance* | *ASTM worked penetration (1/10 mm)* | *Application* |
|---|---|---|---|
| 000 | | 445–475 | Gear greases |
| 00 | Semi-fluid | 400–430 | |
| 0 | | 355–385 | |
| 1 | Soft | 310–340 | |
| 2 | Creamy | 265–295 | Greases for bearings |
| 3 | | 220–250 | |
| 4 | | 175–205 | |
| 5 | | 130–160 | |
| 6 | Soap-like | 85–115 | Block greases |

The method is not satisfactory physically [16.20], and since the sixties attempts have been made to correlate it with, or even replace it by, rheological methods, e.g. the measurement of yield stress in a rotational viscometer [16.21, 16.22] (see Section 16.7).

Nowadays the performance of lubricating greases is described by norms such as ISO 6743-9 [16.23] or DIN 51825 [16.24] defining mainly consistency, upper and lower operating temperature, water resistance, and load-carrying capacity, and for automotive greases by ASTM D 4950 [16.25], which has been followed by the consideration of reference greases [16.26] and the introduction of the NLGI Certification Mark [16.27].

But to some extent greases are best judged simply by the chemical and physical properties of their base oils and the agents thickening them – naturally the consistency of greases increases with increasing thickener content, and with it some of their properties change. These properties at the same time best reflect the reasonable limits of practical applications.

## 16.2
## Thickeners

Thickeners not only transform liquid lubricants into consistent lubricants, they change the properties of the liquid lubricants also (see Section 16.8). When all their properties are taken into consideration none of the commercially important thickeners stands out from any other (Table 16.3) [16.28]. They are comparably competitive and suited for their tasks. The differences mainly lie in the more special demands made on them.

### 16.2.1
### Simple Soaps

Because, in general, a maximum thickening effect is achieved with carboxylic acids having 18 carbon atoms, soaps are usually prepared from vegetable-derived 12-hydroxystearic acid [16.29, 16.30] or from animal- or vegetable-derived stearic acid [16.31], or from their esters, usually their glycerides, and from the hydroxides of elements of the alkali and alkaline earth metals [16.32, 16.33]. Soaps, by gelling their base oils give greases most of their unique properties. They are not only present as crystallites and dissolved molecules, but above all in a separate phase represented by agglomerates called fibrils or fibers (see Section 16.4). All components are present, and have the properties of grease, even in the smallest lubrication gap. This means that the still popular picture of a thickener as a sponge releasing and soaking up its base oil, the real lubricant, is at least misleading (see Section 16.12).

#### 16.2.1.1 **Soap Anions**

The carbon chain length of a carboxylic acid affects the solubility and surface properties of a soap. Longer and shorter carbon chains reduce its thickening capacity. Increasing the chain length increases the solubility in the base oil, shortening the chain length reduces it. A branched alkyl chain lowers the melting point of a soap and reduces its thickening effect. Carboxylic acids containing carbon double bonds, the so-called unsaturated acids, are more soluble in mineral oils and also reduce the thickening effect and lower the dropping point. Their use is limited because of their lower oxidation stability. Hydroxyl groups increase the melting point and the thickening effect of a soap because of its increased polarity.

#### 16.2.1.2 **Soap Cations**

The soap cations also are responsible for essential properties of soap greases. The cations govern the thickener yield, the dropping point, according to DIN ISO 2176 the temperature at which a grease gets liquid under normal conditions, the water resistance, and, to some extent, the load-carrying capacity of a grease, (see Section 16.8).

In 1996 greases based on simple soaps still accounted for more than 70 % of known world production [16.34]. Lithium soaps were the most important, with ca 50 %, then the calcium, sodium and aluminum soaps. The importance of the latter has steadily decreased during recent decades.

**Tab. 16.3** Competitiveness of thickeners.

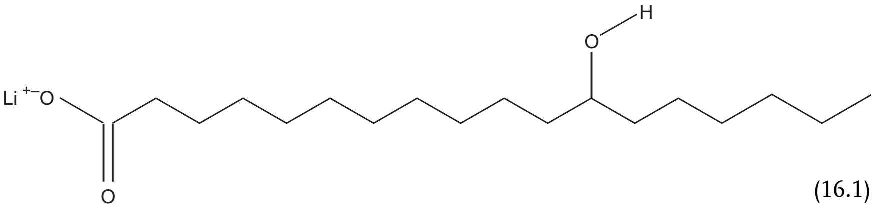
| | *I* | *II* | *III* | *IV* | *V* | *VI* | *VII* | *VIII* | *IX* | *X* | *XI* | *XII* | *Sum* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lithium 12-hydroxystearate | 2.5 | 1.0 | 2.0 | 1.5 | 2.0 | 2.0 | 2.5 | 1.5 | 2.5 | 2.0 | 1.0 | 3.0 | 2.0 |
| Calcium 12-hydroxystearate | 3.0 | 1.0 | 3.0 | 1.0 | 1.5 | 1.0 | 2.5 | 1.0 | 2.0 | 2.0 | 1.0 | 3.0 | 1.8 |
| Lithium complexes | 1.5 | 2.0 | 1.5 | 2.0 | 1.5 | 2.0 | 2.0 | 2.5 | 1.5 | 2.0 | 2.0 | 2.5 | 1.9 |
| Calcium complexes | 2.0 | 3.0 | 2.0 | 2.0 | 1.0 | 1.5 | 1.5 | 3.0 | 1.5 | 2.0 | 2.0 | 2.0 | 2.0 |
| Aluminum complexes | 2.0 | 2.0 | 2.0 | 2.5 | 1.5 | 2.0 | 2.0 | 2.5 | 2.0 | 2.0 | 2.0 | 2.5 | 2.1 |
| Inorganic thickeners | 1.5 | 1.0 | 1.0 | 3.0 | 3.0 | 1.0 | 3.0 | 1.0 | 3.0 | 3.0 | 2.5 | 3.0 | 2.2 |
| Polyureas | 1.0 | 1.5 | 1.5 | 2.5 | 2.0 | 1.5 | 2.5 | 2.0 | 3.0 | 3.0 | 1.0 | 2.0 | 2.0 |
| Terephthalamates | 1.5 | 1.5 | 1.5 | 1.0 | 2.5 | 1.5 | 2.0 | 1.0 | 2.5 | 2.0 | 1.0 | 2.0 | 1.7 |
| Calcium–sulfonate complexes | 2.0 | 3.0 | 2.0 | 2.0 | 1.0 | 2.0 | 1.5 | 3.0 | 1.0 | 1.0 | 2.0 | 1.5 | 1.8 |
| Carbamate-like thickeners | 2.0 | 1.5 | 2.0 | 2.0 | 2.0 | 2.0 | 2.5 | 1.5 | 2.0 | 2.0 | 1.0 | 2.0 | 1.9 |

I = high temperature, II = low temperature, III = aging,
IV = compatibility, V = oil loss, VI = toxicity,
VII = tackiness, VIII = flowability, IX = load,
X = shear, XI = friction, XII = wear.
1.0 = excellent, 2.0 = average, 3.0 = poor

### 16.2.1.3 **Lithium Soaps**

Lithium soap-based greases were first made by Earle [16.35] in 1942; lithium 12-hydroxystearate **16.1**-based greases by Fraser [16.36] in 1946. Nowadays they are usually prepared by reacting lithium hydroxide as a powder or dissolved in water with 12-hydroxystearic acid or its glyceride in mineral oils or synthetic oils. Whether the free acid or its glyceride is preferred depends on the relationship between cost and performance [16.37]. The production temperature is between 160 and 250 °C, depending on the base oil and the type of reactor in use. The dropping point of a mineral oil-based NLGI 2 grease is usually between 185 and 195 °C. For such a multi-purpose grease ca 6 % w/w of soap is required with a naphthenic oil, ca 9 % w/w with a paraffinic oil, and ca 12 % w/w with a polyalphaolefin, and a kinematic viscosity of ca 100 $mm^2\ s^{-1}$ at 40 °C – the thickening effect depends not only on the carbon distribution in a base oil, but also on its viscosity).

The fiber size in lithium 12-hydroxystearate greases is usually between $0.2 \times 2$ and $0.2 \times 20$ μm. Good multi-purpose properties, e.g. a high dropping point, good water resistance, good shear stability – which has been related to hydrogen-bonding of the hydroxyl groups – and good response to additives are the reasons why lithium 12-hydroxystearate-based greases have now been the most popular for more than half a century. They have found a wide range of applications from extreme-pressure (EP) greases based on oils with kinematic viscosities of ca 200 to 1000 $mm^2\ s^{-1}$ at 40 °C for heavy loads, through multi-purpose greases based on mineral oils with kinematic viscosities from 60 to 120 $mm^2\ s^{-1}$ at 40 °C for all kinds of bearings, greases made with diesters or polyalphaolefin oils and kinematic viscosities of 15 to 30 $mm^2\ s^{-1}$ for high-speeds, to those with oil-insoluble polyalkyleneglycols for gears. The lower application temperature limit for a lithium soap thickened grease, as for all other greases, depends mainly on the physical properties of its base oil, of course (see Section 16.3). The upper limit is increasingly determined by a test in the FAG FE 9 test bench, as defined by DIN 51821 [16.38] and by DIN 51825. Again depending on the base oil, it lies between 120 and 150 °C. It is readily apparent that between the dropping point and the upper application temperature limit there can be a gap of between 60 and 100 °C. Oil separation has been discussed as a criterion for both lower and upper application temperature limits [16.39]. In recent years attempts have been made to improve the structural stability of lithium soap-based greases by use of reactive polymers [16.40, 16.41].



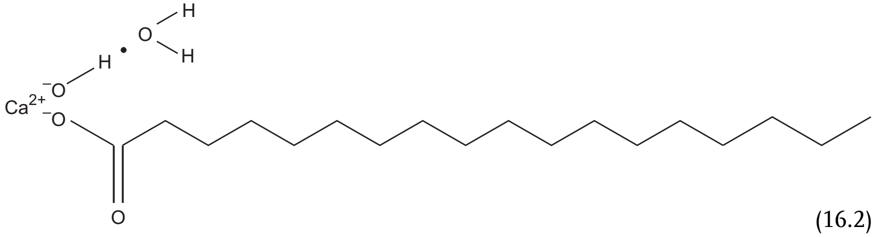
(16.1)

### 16.2.1.4 **Calcium Soaps**

Calcium soaps prepared from 12-hydroxystearic acid still are called anhydrous calcium soaps, too. Similar to the corresponding lithium soaps they indeed contain up to 0.1 % w/w water, but that water is not present as a crystallization component, as in stearic acid based soaps, although technical 12-hydroxystearates contain up to 15 % w/w stearic acid. Calcium greases of this type are produced in the same way as lithium soap-based greases, but at temperatures between 120 and 160 °C. The size of their fibers lies between those for lithium soaps and hydrated calcium soaps. The greases can be used up to 120 °C. Their dropping point is between 130 and 150 °C, depending on their base oil. They usually have a very good corrosion resistance and good oxidation stability; when prepared from suitable base oils they are possibly the best low-temperature greases.

Calcium soaps mainly based on stearic, palmitic, or oleic acid still are called hydrated calcium soaps **16.2**. For these greases the cost of raw materials, and the performance levels, are lowest. They are prepared by neutralization of a slurry of calcium hydroxide in water with fatty acids or fats in mineral oil. Fats are cleaved in the first reaction step into fatty acids and glycerol. This is usually performed in a pressure vessel. Stable greases can only be obtained in the presence of some water, usually ca 10 % w/w of the soap. The water content is usually adjusted in a second step in a stirring, or cooling vessel. The fiber size is usually ca 0.1 × 1 μm. When the water is removed, the grease structure collapses. The dropping point of this type of grease is therefore only 90 to 110 °C and the upper application temperature limit is only ca 80 °C.

The greases have very good water resistance and good adhesive properties. Because their manufacture is rather demanding in relation to the performance obtained, their importance is declining rapidly.



(16.2)

#### 16.2.1.5 **Sodium Soaps**

Compared with lithium and calcium 12-hydroxystearate greases the importance of greases based on sodium soaps is nowadays low, although in the form of semi-fluid products they still are of some interest for the lubrication of gears. Sodium greases made from fatty acids or fats have dropping points of ca 165 to 175 °C. Their upper temperature limit is ca 120 °C. Products are available with short and long fiber structures; the latter reach up to 1 × 100 μm and are responsible to some extent for the rather high load carrying capability in gears. Although the greases have extraordinary good corrosion-preventive properties if only small amounts of water are present, their main disadvantage is the solubility of the sodium soaps in larger amounts of water; this leads first to gel formation, which dramatically increases the apparent viscosity, and later to breakdown of the whole structure.

### 16.2.1.6 **Other Soaps**

Aluminum soap-based greases are usually produced with pre-manufactured aluminum soaps, usually aluminum stearate. The first greases of this type were probably introduced by Lederer [16.42] in 1933. They have dropping points of up to 120 °C, their upper temperature limit is 80 to 90 °C – above 90 °C they tend to gel. Typical of this soap is a particle size of < 0.1 × 0.1 µm, which is to some extent responsible for the low shear stability and the pronounced thixotropic behavior of the products. Aluminum greases are usually very transparent and smooth. They have good water resistance and adhesive properties, but have been widely replaced by lithium greases, partly because, to obtain consistent products, aluminum greases must not be stirred during the finishing process but poured into pans and left for several hours to cool.

Barium soap-based greases have high water resistance and shear stability, and lead soap-based greases have advantageous load-carrying capacity and wear protection properties. Both have, nevertheless, been completely been replaced, mainly for toxicological reasons.

### 16.2.1.7 **Cation Mixed Soaps $M_1X/M_2X$**

Mixtures of soap greases with different cations, mainly lithium–calcium, calcium–sodium, and sodium–aluminum are called mixed-soap greases. Their properties largely depend on the relative proportions of the two or more soaps. Lithium–calcium greases have better water resistance than pure lithium greases and often better shear stability. As long as the proportion of calcium soap does not exceed 20 % w/w their dropping points, 170 to 180 °C, are close to that of the lithium soap alone, (Fig. 16.1) and their

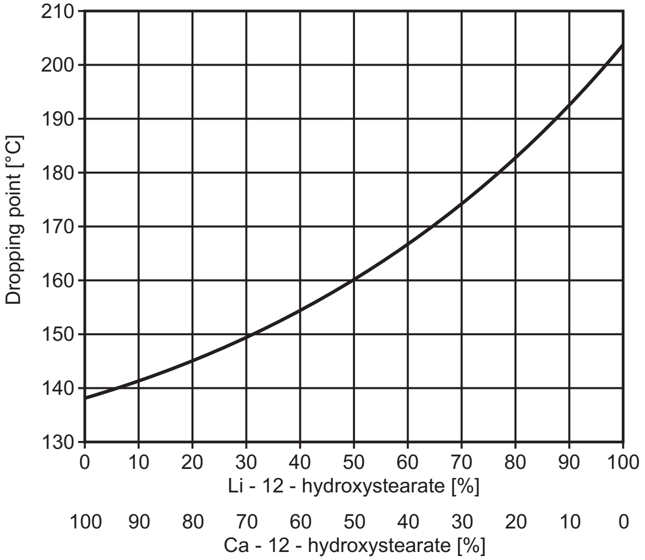


**Fig. 16.1** Dropping points of lithium and calcium 12-hydroxystearate greases (consistency NLGI 2, base oil SN 500, no additives).

wear and friction performance are better than that of pure lithium grades [16.43]. Some calcium–lithium greases perform better than pure calcium 12-hydroxystearate greases.

Lithium–calcium greases have become quite popular as specialized multi-purpose greases. Greases mainly based on sodium-aluminum stearate, already described in detail by Boner [16.44], have been used as substitutes for lithium greases, for example in the former GDR. Lithium–bismuth greases have been reported to perform better than conventional lithium greases (including those that contain bismuth additives) in terms of mechanical stability and high-temperature use [16.341]. Cation mixed soap greases are usually manufactured by a one-step process, because the stability of mixtures of finished products are not always satisfactory.

#### 16.2.1.8 Anion Mixed Soaps $MX_1/MX_2$

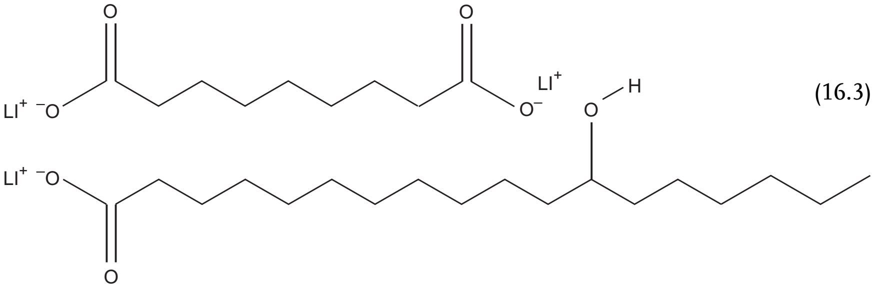
Because of the animal and vegetable origin of the acidic components most simple soap-based greases are already anion mixed soap based. Nevertheless the fine-tuning of multi-purpose greases and specialized multi-purpose greases is, especially for the comparatively pure 12-hydroxystearic acid, often achieved by substituting small amounts of the main acid by a complementary acid, e.g. behenic, naphthenic, or stearic acid.

### 16.2.2
### Complex Soaps

With complementary salts of inorganic acids, e.g. boric and phosphoric acid, or with short-chain carboxylic acids, e.g. acetic acid, or with dicarboxylic acids, e.g. azelaic [16.45] and sebacic [16.46] acids, or with more complex acids, e.g. the dimeric variety [16.47] – all derived from vegetable oils – simple soaps can form some types of complex soap. The expression ‘some types’ is used, because in a chemical–physical sense complexes formed in the way Ischuk [16.48] reported for monovalent cations such as $Li^+$, can also be understood as adducts and complexes of cations such as $Ca^{2+}$ and $Al^{3+}$ formed as reported by Polishuk [16.49], can also be understood as a different kind of mixed soap. The addition of complementary salts always leads, on the one hand, to an increase in the dropping point of from ca 50 to 100 °C and to reduced oil separation, primarily because of the increased thickener concentration, and, on the other hand, for the same reason, to reduced suitability for low-temperature application. Because of their superior properties complex soap-based greases have become very popular and nowadays account for ca 20 % of all greases sold.

#### 16.2.2.1 Lithium Complex Soaps

Apart from their higher upper-temperature limit, between 160 to 180 °C, some lithium complex soap-based greases perform similarly to the corresponding simple soap-based products, but because of the many possible complementary salts not all of their properties can be generalized. Of the many known formulations the most widespread is based on 12-hydroxystearic acid and azelaic acid **16.3**. This complex was introduced in 1974 [16.50]. The first complex was based on 12-hydroxystearic acid and acetic acid and was patented as early as 1947 [16.51]. The lithium complex soaps with the best load-carrying capacities contain boric or phosphoric acid. The fiber size of such a complex soap does not differ much from that of the simple soap and it does not change significantly with common shear, (Fig. 16.2). They used to have the highest dropping points, also, until organic complementary acids were reported to have comparable dropping points [16.52]. In addition to azelaic and boric acids, other acids have also been examined systematically (Table 16.4) [16.53]. The 12-hydroxystearic acid–azelaic acid system has been revised with regard to processing and the influence of surfactants [16.54] and with sebacic acid the same has been done mainly with regard to stoichiometry [16.55]. In 1998 a survey was published covering developments in the field of complex greases in the nineties [16.56].



(16.3)

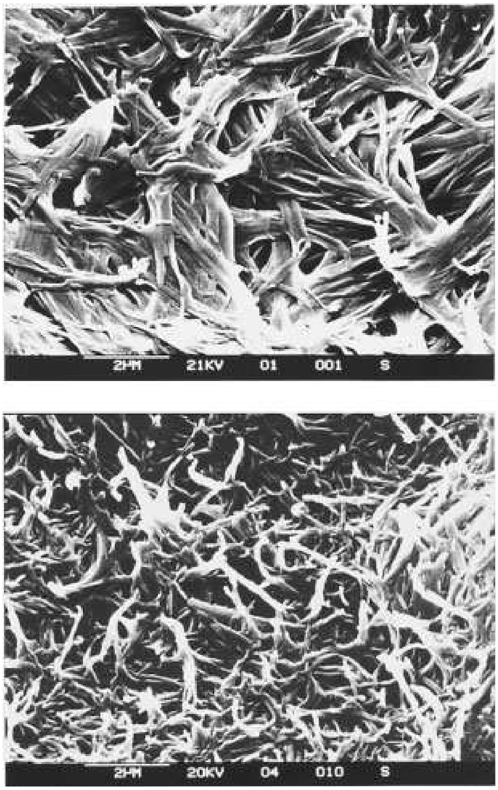


**Fig. 16.2** Lithium 12-hydroxystearate–borate complex soap fibers (under the electron microscope before and after shear at 140 000 $s^{-1}$).

**Tab. 16.4** Lithium complex soap systems.

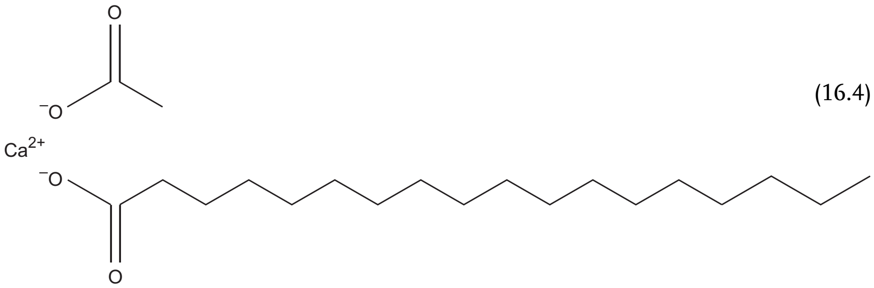
Lithium 12-hydroxystearate
+
Lithium adipate
Lithium azelate
Lithium dimerate
Lithium sebacate
Lithium terephthalate
...
Lithium borate
Lithium phosphate

The activities with lithium complex soaps are numerous, as can be seen from the patents referred to in the Chemical Abstracts Selects [16.57] because with a share of about 10 %, lithium complex greases are the most widespread complex greases.

They range from practical activity, for example optimization of specifications for fleet greases [16.342, 16.343], to more fundamental activity, for example obtaining greater insight into the formation of complexes during processing, with the aid of FTIR spectroscopy, for example [16.344], or making use of high-volume chemicals, for example dodecanedioic acid, that have previously been neglected by the lubricating grease industry [16.345], and experiments of merely speculative nature designed to provide information about the performance potential of components new to the lubrication industry, for example polyanhydrides [16.346].

### 16.2.2.2 **Calcium Complex Soaps**

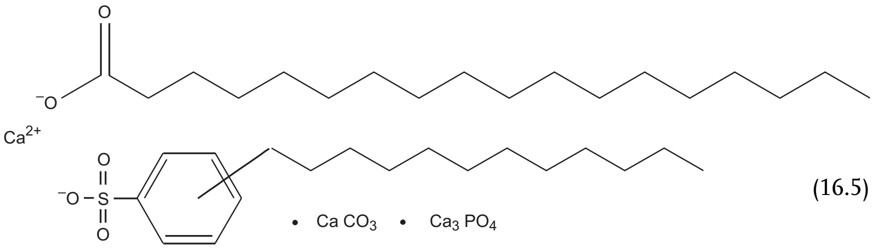
All commercial calcium complex greases contain acetic acid as a complementary acid **16.4**. The complex was first described in 1940 [16.58]. Calcium complex greases have good shear stability and water resistance, a low oil separation, and good load-carrying capacity. Their upper temperature limit is 160 °C. Because of the formation of ketones, described by common organic preparation methods, from about 120 °C pronounced hardening can occur. It has, nevertheless, been possible to retard hardening with the aid of polymeric structure-modifiers [16.59].



(16.4)

### 16.2.2.3 **Calcium Sulfonate Complex Soaps**

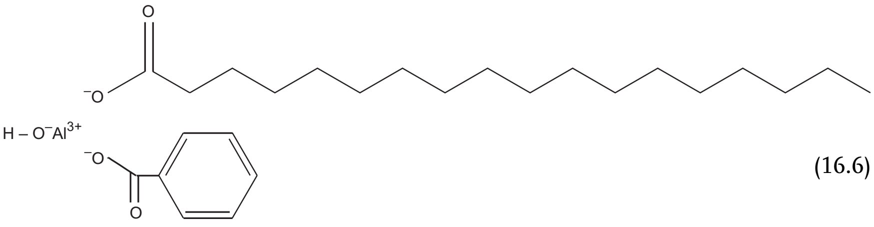
Competitive greases based on this complex were first offered in 1985 [16.60]. Initially they comprised in situ-produced overbased calcium sulfonates and the calcium salts of other sulfonates, of 12-hydroxystearic acid, and of boric acid [16.61]. The complex could be improved by replacing calcium borate by phosphate **16.5** [16.62]. Greases containing calcium acetate instead are also possible [16.63]. Polishuk [16.64] reviewed the history of calcium greases, including their culmination in the development of this new thickener system, and the first decade of their availability has also been reviewed [16.65]. The greases are extraordinarily corrosion protective and resistant to shear and have load-carrying capacities that can be matched only by greases of other soaps that contain large amounts of additives. Their dropping points start at ca 220 °C, but their upper temperature limit is ca 160 °C. Some grades can, nevertheless, survive temperatures up to 250 °C for several hours. The importance of calcium sulfonate complex greases has increased substantially during the last five years. Even food-grade varieties are now available [16.347]. The nature of the complexes, and the structure of the calcium carbonate they contain, is still a matter of discussion [16.348], and overbased carboxylates have been offered as potential substitutes for the corresponding sulfonates [16.349].



(16.5)

### 16.2.2.4 **Aluminum Complex Soaps**

Today only one of the possible aluminum complexes is still widely used. It comprises aluminum stearate and benzoate, **16.6**, and was first patented in 1952 [16.66]. Aluminum complex greases of that kind have good water resistance and good low-temperature properties. Their importance has decreased in recent years, although attempts have been made to gain better insight into the formation of the soaps [16.67], to modify the process [16.68], or to extend their range of application [16.69, 16.70], and thus to make them more attractive again. This might be possible for food grade and biodegradable greases (see Section 16.11).

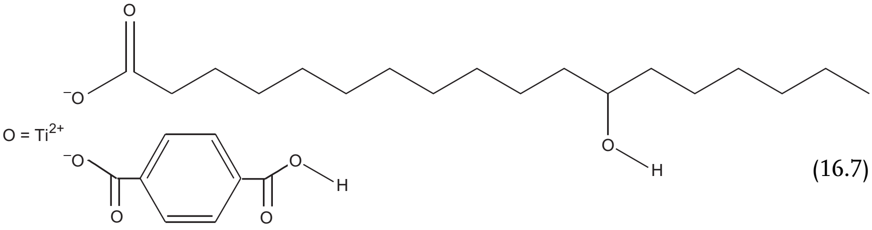


(16.6)

#### 16.2.2.5 Other Complex Soaps

Sodium complex soap-based greases have been used because of their suitability for high relative velocities, but like the simple soaps they lost importance because of their limited water resistance; barium complex soaps have been replaced almost as completely as the simple soaps.

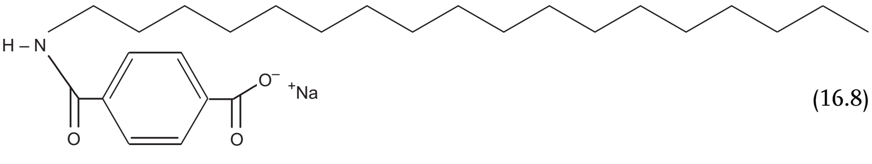
Titanium complex greases were patented in 1993 [16.71]. They are based on 12-hydroxystearic and terephthalic acid, **16.7**. Of their properties [16.72] good load-carrying capability is most worthy of mention.



(16.7)

### 16.2.3
### Other Ionic Organic Thickeners

Of many possible soap-like salts only the sodium and the calcium salts of stearylamidoterephthalic acid, **16.8**, are on a technical scale. They were patented in 1954 [16.73] and proposed for multi-purpose greases in 1957 [16.74]. Greases of this kind have dropping points of up to 300 °C and upper temperature limits of up to 180 °C. Although they have the thickening effect of simple soap greases they behave like complex greases; this makes them valuable wide-range greases. They have recently been revised and are recommended for several applications [16.350]. The thickeners are among the most expensive and are preferably used with synthetic base oils. Complex soaps comprising terephthalamate and benzoate have been described and complexes of aluminum stearate with terephthalamates have also been investigated [16.75].



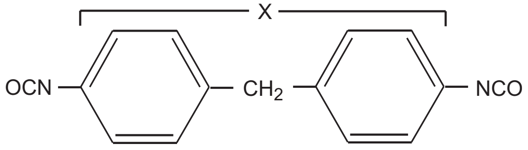
(16.8)

### 16.2.4
### Non-ionic Organic Thickeners

Of a rather large number of theoretically suitable compounds, only the oligoureas, commonly called polyureas, have gained real industrial importance.

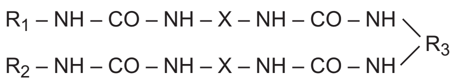
#### 16.2.4.1 **Diureas and Tetraureas**

Oligourea thickeners were introduced in 1954 [16.76]. The reaction products of one molecule of MDI (di-4,4′-isocyanatophenylmethane, **16.9**) or other diisocyanates with two molecules of monoamines are called diureas, **16.10**. Tetraureas, **16.11**, are the reaction products of two molecules diisocyanate with one molecule of diamine and two molecules of monoamine. Depending on the performance required aliphatic or aromatic amines or mixtures of both are used. With excess diisocyanate three-dimensional structures are built along biuret-like bridges, **16.12**. Oligourea-thickened systems have been reviewed in detail with regard to their properties in comparison with complex soap-based greases and the dependence of these properties on the base oil used [16.77]. The upper temperature limit of oligourea greases is not determined so much by the stability of the thickener, the decomposition of which usually starts slightly below 250 °C, as by the stability of the base oil. These greases are therefore superior to soap-based greases when the application temperatures exceed 180 °C. When an oligourea grease based on polyalkyleneglycols is overheated, breakdown is, ideally, to gaseous products only. Although tetraureas have some advantages, too, [16.78], the trend in polyureas is toward diureas [16.79]. It is not easy to decide whether aliphatic, alicyclic, or aromatic amine-based diureas perform better under standard conditions, as was learnt from studies of film thickness and response to EP additives, for example [16.351]. Oligourea complex greases containing calcium acetate were introduced in 1974 [16.80]; others containing carbonate [16.81, 16.82] and other complementary salts followed and are still favored for some applications. Oligourea complex greases have also been called polyurethane or polyurethane complex greases [16.83], but these names should be reserved for oligourea greases in which the amines are partly replaced by alcohols. In 1995 a fibrous product was presented to the public [16.84]. Although soap-based greases cannot compete with oligourea greases at higher temperatures, below 180 °C lithium complexes, for example, are at least their equal [16.85].



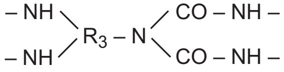
(16.9)

$$R_1-NH-CO-NH-X-NH-CO-NH-R_2 \qquad (16.10)$$



$$\left.\begin{array}{l} R_1-NH-CO-NH-X-NH-CO-NH \\ R_2-NH-CO-NH-X-NH-CO-NH \end{array}\right\rangle R_3$$

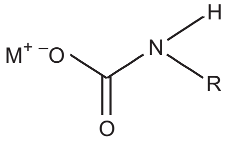
(16.11)



$$\left.\begin{array}{r} -NH \\ -NH \end{array}\right\rangle R_3-N\left\langle\begin{array}{l} CO-NH- \\ CO-NH- \end{array}\right.$$

(16.12)

Carbamate-like thickeners, **16.13**, are related to the oligoureas and simple soaps, and have properties intermediate between those of these groups. This is true for mixtures of oligourea greases with simple or complex soap greases also. In the same way as carbamate-like greases, the mixtures can be regarded as “urea soap” greases.



(16.13)

#### 16.2.4.2 Other Non-ionic Organic Thickeners

Polymerized perfluorinated hydrocarbons, usually micronized polytetrafluoroethylene (PTFE) powders, are the choice for greases that must function at temperatures above 220 °C – with an upper temperature limit of about 270 °C. For applications of this kind their liquid oligomers or preferably corresponding perfluoroalkylene ethers must be chosen as base oils [16.86–16.88]. Polymers such as polyamides or polyethylenes are mainly used as additives.

### 16.2.5
### Inorganic Thickeners

To be suitable for use in lubricating oils inorganic thickeners must be treated with between 5 and 10 % w/w reactive organic compounds. This treatment alone enables them to act as oleophilic thickeners and without it they would be like fillers, co-thickeners, and solid lubricants, that only at concentrations above ca 40 % w/w yield grease-like pastes. Apart from these hydrophobic agents additional polar activators, e.g. acetone, ethanol, or, for safety reasons, propylene carbonate [16.89], are needed to accomplish gelation. They are used at a level of 10 % w/w relative to the thickener. The thickeners themselves are stable up to temperatures of > 300 °C, but the resulting greases or gels are mainly used for applications up to 200 °C that do not require too pronounced shear stability. This is partially because of the diameter of the primary parts, only ca 0.05 μm. The tendency of inorganic thickened greases to hardening and oil separation during storage, and their sensitivity to polar additives can to some extent be overcome by addition of functionalized polymeric agents [16.90]. This is in accord with more theoretical studies with alumina [16.91].

#### 16.2.5.1 Clays

Clays, or, to be more precise, bentonite-type aluminosilicates, mainly the smectites montmorillonite and hectorite [16.92], are the most important inorganic thickeners. They are usually treated with quaternary ammonium compounds, e.g. trimethyl stearyl ammonium chloride, and the activators mentioned above.

#### 16.2.5.2 Highly Dispersed Silicic Acid

Highly dispersed silicic acid is usually prepared by burning silicon tetrachloride in an oxyhydrogen gas flame; it is made more suitable as a thickener by treating it

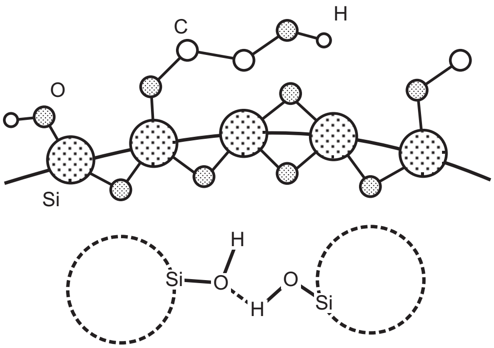


**Fig. 16.3** Highly dispersed silicic acid surface (free, epoxidized, dehydrated, and alkoxylated hydroxyl group).

with, e.g., silanes, silazanes, or siloxanes [16.93] (Fig. 16.3). One advantage of these products is the low temperature-dependence of their consistency [16.94]. With suitable base oils and activators they form white to transparent gels that are used in food or medical applications.

### 16.2.6
### Miscellaneous Thickeners

In principle inorganic and organic pigments of all kinds [16.95, 16.96] can be used as thickeners or co-thickeners or at least as fillers. The transition to lubricant additives is fluid. Only the inorganic materials soot and expanded graphite [16.97] and the organic phthalocyanines [16.98] are sometimes used on an industrial scale.

In principle it is possible to make greases with combinations of all kinds of thickener, but only some mixtures of soaps with complex soaps, or of soaps with clays and oligoureas have been reported in practice.

### 16.2.7
### Temporarily Thickened Fluids

Under defined conditions liquids and suspensions of solids in liquids have significantly increased viscosities (Table 16.5).

**Tab. 16.5** Temporarily thickened fluids.

***Magnetic Fluids***

1 Suspensions of ferrite particles in inert liquids
2 Magnetic field strength
3 Acoustic and rapidly rotating machinery

***Electrorheological Fluids***

1 Suspensions of silicates in silicone oils
2 Voltage
3 Hydraulic valves, shock absorbers, viscous clutches

***Liquid Crystals***

1 Compounds that form smectic B-phases
2 Pressure–temperature
3 Hydraulic valves, clutches

1. raw material; 2. cause of solidification; 3. application.

Some liquid crystalline systems are suitable as lubricants under the influence of changes in pressure [16.99], or temperature [16.100]. Some of the solutions that can form liquid crystals in limited concentration ranges [16.101] are comparable with consistent lubricants and some liquid crystals in concentrated point contacts are even superior [16.102].

Electrorheological or electroviscous fluids, suspensions of micronized highly polarizable and hydrophilic porous solids, originally silica in silicone oil with water as a promoter, later polyurethanes without a promoter in hydrocarbons, are characterized by an enormous increase in apparent viscosity, when subject to electric fields. The first practical applications go back to 1942 and to Winslow [16.103]. In recent years increasing use in hydraulic valves, shock absorbers, and clutches has been reported and the progress of the scientific work has been published [16.104, 16.105].

Magnetorheological fluids [16.106, 16.341], suspensions of micronized transition elements, mainly ferrites, behave similarly in magnetic fields. Both kinds of fluid have also been named ‘smart fluids’. They contain between 20 and 60 % solid particles which form more or less branched chains in the applied fields; they therefore act as Bingham plastics (see Section 16.7). Increased shear of the fluids leads first to stretching and then to breakage of the particle chains, although steady recombination of the parts of the chains enables the fluids to keep their apparent viscosity even at high shear rates [16.352]. It remains an open question whether grease emulsions, or even foams, can have a lubricating potential comparable with that of greases. A practical study of the emulsions has been reported for lithium greases. Results were promising with regard to wear in the Timken test but not in the four-ball apparatus [16.353].

## 16.3
## Base Oils

In principle all the lubricating oils already described in Chapters 4 and 5 can be used as base oils for greases, but in general only oils with kinematic viscosities from 15 to 1500 $mm^2\ s^{-1}$ at 40 °C are used. The oils with the lowest viscosities and the best low-temperature properties have the lowest operational temperatures at the highest speeds. The oils with the highest viscosities have the best performance at the lowest speeds and the highest loads, the lowest evaporation loss, the strongest adhesion and the best water or solvent resistance.

### 16.3.1
### Mineral Oils

Oils with low viscosity indexes (VIs) usually require less thickener than those with high VIs and the same kinematic viscosity. The latter can be used over a wider temperature range and are, therefore, preferred nowadays. In principle the thickening effect (yield) depends on the difference between the solubility parameters of the base oil and thickener [16.107–16.110]. Because oil separation depends on the concentration of thickener, at a given temperature oil separation is most pronounced for greases based on low VI oils, e.g. aromatic oils or alkylbenzenes. Oil separation goes through a minimum with increasing kinematic viscosity for a given kind of base oil. This can easily be understood when only the dependence of particle interactions on the size of the involved particles is considered. Some of the specific properties of greases also depend on the ratio of the temperature and pressure coefficients of the viscosity of their base oils. If all this is taken into account, the effect of even a small change in base oil composition on the performance of a grease should not be underestimated [16.111].

### 16.3.2
### Synthetic Base Oils

Many of the synthetic oils in use today became available for the first time between 1920 and 1940 as has been described by Zisman [16.112] (Chapter 5). Because of their cost, synthetic oils are used in greases only when the performance required cannot be achieved with mineral oils [16.113]. Even today greases of this kind represent less than 5 % of overall grease consumption.

#### 16.3.2.1 Synthetic Hydrocarbons

Polyalphaolefins [16.114] are the ideal base oils for wide temperature-range greases. They are usually used in the same viscosity range as their mineral oil equivalents, but whereas the upper kinematic viscosity border of the former is ca 50 $\mathrm{mm^2\ s^{-1}}$ at 100 °C, polyalphaolefins and similar products are available to ca 2000 $\mathrm{mm^2\ s^{-1}}$ at 100 °C. In contrast with mineral oils they are preferably shrinking rather then swelling polymeric sealing materials. Usually, therefore, esters with softener properties must be added. Greases made from polyalphaolefins with standard viscosities are usually meant for lifetime lubrication, those made from mixtures with mineral oils are often called semi-synthetic and are used in specialized greases to increase lifetime.

Polybutenes and comparable polymers are usually used to increase the viscosity of standard base oils; occasionally they are used as a base oil component, or even the only base oil.

### 16.3.2.2 Other Synthetic Base Oils

High molecular-weight esters made from diols or polyols and dicarboxylic acids with viscosities > 2000 $mm^2$ $s^{-1}$ at 40 °C are used as base oils in greases that must be solvent resistant.

Silicone oils can be regarded as polyethers of alkylated silicic acids. In contrast with other greases silicone greases [16.115] are better distinguished according to the properties of their base oils [16.116]. The kinematic viscosities of silicone oils for greases range from ca 75 to ca 10 000 $mm^2$ $s^{-1}$ at 40 °C. Dimethylsilicone oils are physiologically inert. When thickened with highly dispersed silicic acid they are mainly used as sealants or in applications where specific electrical and thermal conductivity is important.

Partially phenylated dimethylsilicone oils have very good low-temperature properties and are used with all types of thickener, depending on their (often military) application. Partially fluorinated dimethylsilicone oils are usually thickened with PTFE powders.

Apart from being suitable for temperatures up to 270 °C perfluorinated ethers thickened with PTFE powders are the only greases that can work in the presence of aggressive chemicals such as oxygen or chlorine. This is also their main advantage over the corresponding greases based on partially fluorinated silicone oils.

### 16.3.2.3 Immiscible Base Oil Mixtures

Greases are the only lubricants that with their thickeners have brackets at their disposal to force immiscible liquid components into one lubricant. Friction of hydrocarbon- or ester-based greases can be reduced in this way and lifetime can be prolonged with polyethers and with polyesters [16.117]. Similar results have been reported for greases made from polyalphaolefins or polyol esters and perfluorinated ethers [16.118]. Some authors refer to these as hybrid greases.

## 16.4
## Grease Structure

In 1952 the nature of soap-based greases was described and reviewed in detail by Schultze in Zerbes outstanding encyclopedic book on mineral oils and related products [16.119] and in 1954 by Boner in his famous book on greases [16.120]. About two decades later the relevant publications were reviewed again, this time by Spengler and Wunsch [16.121]. It was shown with the aid of mainly microscopic and spectrometric methods that greases have a more or less distinct lattice structure, that soaps therefore form a framework of string-like aggregates comprising micelles and crystallites that are called fibrils or fibers, and that the base oil is embedded in this framework. The oil is probably bound in three ways: firstly by molecular attraction between polar thickener and oil areas; secondly by capillary forces; and thirdly by mechanical occlusion. In soap-based greases small changes in the concentrations of the free acids and bases have a large effect on the shape and size of the aggregates. Boner did not declare his position in his second book about greases [16.122]

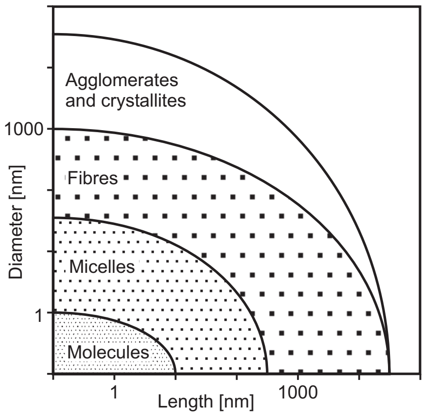


**Fig. 16.4** Dimensions of thickener components.

and even today it has not been definitively decided whether soap-based greases fit into the system of colloids [16.123] or are of a more crystalline nature. Macroscopically the thickener particles are agglomerates of crystallites and micelles and dissolved components the size of which is limited by the method of homogenization (Fig. 16.4). The nature of grease-like products formed with inorganic thickeners, pigments, or ferrites, is, however, not as difficult to describe. Here the laws of colloidal chemistry and physics can usually be applied fully [16.124]. This should also be expected for greases that are made from oligoureas, but here the structures that can be seen with the aid of electron microscopes are most similar to soap fibers [16.125].

The structures of liquid crystals are rather different [16.126, 16.127]. Study of these has revealed much of interest in recent years [16.128].

## 16.5
## Additives

Most of the additives used in liquid lubricants, already described in Chapter 6, can be used in greases also, and in the same way, although in general at substantially higher concentrations (Table 16.6). Interactions of thickeners and additives must therefore always be considered. An interesting new monograph on lubricant additives, published in 2003, has some deficiencies in this respect [16.354].

**Tab. 16.6** Usual grease additive levels (%).

| | |
|---|---|
| Antioxidants | 0.10–1.00 |
| Corrosion inhibitors | 0.50–3.00 |
| EP/AW additives | 0.50–5.00 |
| Metal deactivators | 0.05–0.10 |
| Solid lubricants (black) | 1.50–3.00 |
| Tackifiers | 0.10–1.00 |

A typical lithium 12-hydroxystearate-based multi-purpose grease contains at least 0.2 % w/w of an antioxidant or a mixture of antioxidants, between 0.5 and 1.0 % w/w of one or several corrosion inhibitors, ca 0.05 % w/w of a metal deactivator and – in its EP-version – up to 2.5 % w/w EP/AW additives – depending on the type chosen. The antioxidant level can reach 3 % (*w*/*w*) and more in high-performance greases, and high-molecular-weight products are preferred as antioxidants [16.355]. The additives partially described below are only applicable in, and only favorable for, greases.

### 16.5.1
### Structure Modifiers

Greases always contain compounds that affect the structure of the thickener itself to the same extent as they act as additives for the greases as a whole. These compounds are mainly water, glycerol, excess fatty acid, and excess alkali metal/alkaline earth hydroxides. Interactions between structure modifiers and additives, e.g. that between EP additives and glycerol [16.129] or stearic acid [16.130], can severely affect the performance of a grease. Although water, even at a level of 1 % (*w*/*w*), can be a problem in advanced-lifetime applications [16.356], a water content of 20 % (*w*/*w*) and more does not necessarily result in reduced performance in some steel-work applications. Another structure modifier of greases that can easily be overlooked is the necessarily incorporated air. Though the air content must be limited, of course – often < 3 % [16.131] – without air the transport of a grease through a long pipe would be almost impossible.

### 16.5.2
### Antirust Additives (Corrosion Inhibitors)

The most efficient corrosion inhibitor for a soap based grease is, when practicable, excess of the metal hydroxide. Sodium or calcium sulfonates, among the most powerful antirust additives, are also strong structure modifiers and can cause problems with most greases, especially greases made with inorganic thickeners. Imidazoline and sarcosine derivatives and alkenyl succinic acid esters are suitable inhibitors for soap-based greases; disodium sebacate is mainly used in gel greases. Carbodiimides have been recommended for use in ester-based greases, primarily to prevent hydrolysis [16.132].

### 16.5.3
### Extreme Pressure and Anti-Wear Additives

Of the many well known commercial types of additive [16.133] compounds containing heavy metals such as antimony or lead are, for reasons of toxicity, no longer acceptable as heavy duty- and anti-wear additives, and even the use of zinc is disputed today, because of water pollution problems. Ashless additives, e.g. dithiocarbamates, dithiophosphates, and thiadiazole derivatives are slowly taking over [16.134] –

the last also have corrosion-inhibiting properties [16.135]–and the importance of ashless alkylaminophosphorodithioates [16.136] as grease additives is also expected to increase.

Bismuth is one exception among the heavy metals. Additives containing this element have some of the properties of its toxic neighbors, but without their disadvantages [16.137, 16.138].

### 16.5.4
### Solid Lubricants

Solid lubricants are described in detail in Chapter 17. Because of their nature greases can contain these, or other additives that are insoluble in the base oil or base oil mixture, in rather high concentrations. For most applications an average particle size of the micronized solids of between 5 and 15 μm is acceptable.

Graphite and molybdenum disulfide are the oldest, the most common, and the by far best investigated (black) solid lubricants for greases. Many manufacturers use them as mixtures, because they are believed to perform better. The performance of molybdenum disulfide has been improved by the addition of zinc dithiophosphates [16.142] and by admixture of polyamide powders, urea resins [16.143], or salts of cyanuric acid derivatives [16.144]. Admixture with PTFE powders does not give comparable results [16.145]. Molybdenum dithiophosphates and dithiocarbamates, rather than the disulfide, are of growing interest for joint greases [16.146].

PTFE (polytetrafluoroethylene), although rather expensive, is still the most common (white) polymeric solid lubricant in greases. But high and ultra-high molecular-weight polyolefin powders are becoming increasingly competitive.

Inorganic (white) solid lubricants, e.g. the hydroxides and diphosphates of some of the alkaline earth metals, improve the EP/AW properties of common greases and are especially suitable when the greases are subject to oscillations [16.147]. Bismuth sulfide ($Bi_2S_3$) [16.357], hexagonal boron nitride (BN) [16.358], and tin sulfide ($SnS_2$) [16.359] have also been proposed as solid lubricant alternatives to greases.

There is a wide range of other, mostly inorganic, solids that contribute to the bulk rather than to lubrication; these are usually called fillers [16.360, 16.361].

### 16.5.5
### Friction Modifiers

In liquid lubricants carboxylic acids and their salts act as friction modifiers [16.151] and as additives for the boundary lubrication regime [16.152]. In greases that contain large amounts of these compounds, solid lubricants are sometimes called friction modifiers, instead [16.153], but sometimes polymeric additives also [16.154]. Liquid crystals have also been discussed with regard to their contribution to film strength [16.362].

Some properties of greases, for example mechanical stability, can, to some extent, be improved only by use of polymeric additives; some properties, e.g. resistance against aggressive reagents cannot be improved by use of additives.

### 16.5.6
### Nanomaterials

Carbon black is probably the oldest industrial “nanomaterial”. Its synthetic alternative, highly dispersed silicic acid – “white carbon black” – was invented by Kloepfer in 1942.

Lubricant or additive properties have been predicted for $SiO_2$ microspheres of 100–500 nm [16.148]; superfine silicic acid particles less than 40 nm in diameter have been reported to act as EP/AW additives [16.149] and the same has been found to be true for phosphate glass [16.150].

Fullerenes have been known since 1985 [16.139]. They are responsible for the start of the lubrication industry’s interest in material with particle sizes below 100 nm. Because of their size, nanomaterials have potential as additives, thickeners, (part of the) base oil of a grease, and (part of) the solids of the corresponding tribosystem.

Simple fullerenes, mainly $C_{60}$, are made from graphite and have been tested successfully as solid lubricants [16.140] [16.141]; they are still expensive, however. Layers of fullerenes between layers of graphite are thought to be able to lead to zero-friction lubrication [16.363] and with graphenes, single planar sheets of $sp^2$-bonded *carbon* atoms, that goal seems even closer [16.364].

$MoS_2$ and $WS_2$ in the shape of “inorganic fullerenes” are believed to have much potential as shock-absorbing solid lubricants, because of their ability to sustain extremely high shockwave pressures [16.365].

Organic dendrimers could be interesting nanomaterials by themselves [16.366], and this is true for metal-containing varieties also [16.367]. When fullerenes are used as a core [16.368], high expectations of their performance as lubricant components seem justified.

Among solids, fullerene filled polyimides are examples because of their wear-reduction potential [16.369]; nanotube-epoxy composites are examples of potential for passive vibration damping [16.370].

## 16.6
## Manufacture of Greases

The properties of greases, especially metal soap-based greases, depend not only on their composition but also, and to nearly the same extent, on the way in which the thickeners are prepared and dispersed.

### 16.6.1
### Metal Soap-Based Greases

#### 16.6.1.1 Batch Production with Preformed Metal Soaps

It is possible, as one stage of grease manufacture, for a metal soap prepared in an independent first step, or a commercially available soap, to be dissolved or dispersed and heated and cooled again under defined conditions in a suitable base oil. A few years ago the technology acquired a new name – "dry technology" [16.371]. But because of the higher costs of the thickener this method can be recommended only for highly sophisticated synthetic greases with precise chemistry or for functional base fluids that would react with the water or, even worse, with the steam generated during the neutralization process.

#### 16.6.1.2 **Batch Production with Metal Soaps Prepared In-situ**

In general fatty acids or their glycerides or even their methyl esters are reacted with aqueous solutions or suspensions of the described metal hydroxides in part of the base oil. Each batch of a grease is produced by following a ten-point schedule (Table 16.7).

**Tab. 16.7** Batch production of soap based greases – ten-point schedule.

1 dissolution or dispersion of the fatty acids in one third to two thirds of the base oil at temperatures up to 90 °C;
2 addition of the metal hydroxides as a solution or suspension in water;
3 heating to temperatures between 115 to 150 – or under pressure at 180 to 250 °C – depending on the type of reactor;
4 dehydration of the soap by heating to 180 to 200 °C or by pressure release;
5 crystallization during cooling to 150 to 130 °C combined with addition of more base oil;
6 addition of additives at temperatures below 80 °C;
7 (pre)filtration and homogenization with one of several possible devices;
8 adjustment of the specified consistency or flow pressure;
9 (end)filtration and deaeration; and
10 packing into containers.

Different kinds of reactor or reactor combinations are used (Fig. 16.5). Single reactors are usually open vessels heated by gas, steam, or heating oil. Mixing is achieved with single action, double action, or counter-rotating stirrers, usually designed as planetary stirrers, although turbines or dispenser disks can also be used. Scrapers are always

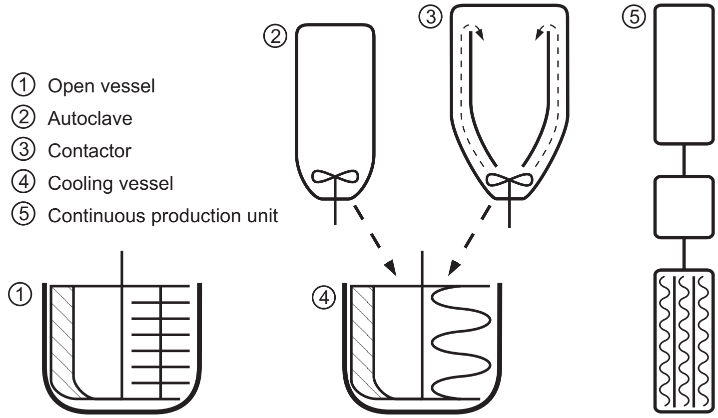


**Fig. 16.5** Reactors for the manufacture of greases.

necessary, because of the need to move crystallizing material away from the walls to prevent a drop in heat transfer and degradation of the forming product. Sometimes the vessels can be used for evaporation or used under pressure. All in all the design has not changed much over recent decades and systematic optimization has not been reported [16.155]. During the reaction stage open reactors must not be heated much above 120 °C, because of the possibility of excessive foaming. Sometimes defoamers, for example low-viscosity dimethylsilicone oils, are added in small quantities during this stage.

Double or multiple reactors, the first being only a melting vessel, a Stratco contactor, or an autoclave – the last type of reactor can also be heated inductively – have some advantages in comparison with single and open reactors. The neutralization reaction is controlled more easily and the reaction time is shorter when the dehydration process is optimized [16.156]. The second vessel is either a finishing or a diluting vessel if several different grades are to be produced from one base grease. In each instance the main intention is to put the maximum thermal and mechanical energy into the reacting material in the minimum time. Increasing reaction speed with increasing contact is the reason for the production of base greases as concentrated as possible with as much agitation as possible. In a single reactor this means that the reactor at the start should not be more than half full (unless the quality of the grease affords a different approach). One further advantage of the multiple reactor process is that the first reactor never gets contaminated with additives. A third reactor is usually used for black, colored, or white greases.

When base oils such as silicone oils used for the in-situ production of soap greases, the thickeners are sometimes formed and dispersed in the presence of solvents which are subsequently removed by extraction or evaporation [16.157]. For lithium and lithium–calcium soap greases a process variation has been described in which lithium hydroxide is desiccated in some of the base oil at a temperature above 100 °C, before addition of the corresponding fatty acids, and the maximum process temperature is reduced to below 150 °C. Energy is saved, foam is not an issue, and (steam-heated) grease kettles can be used that are not suitable for the normal hot process [16.372–16.374].

The batch production of metal soap greases can be automated. This has been achieved with the aid of value engineering [16.158] and computer control [16.159].

### 16.6.1.3 Continuous Production

Technology for the continuous manufacture of greases was first described in 1969 [16.160] and has been adapted by another manufacturer [16.161]. In principle a production unit consists of three elements only (Table 16.8). The advantages of such a unit are its compact size (a few hundred liters only), its minimal energy consumption, and the uniformity of the product; the disadvantages are the difficulty of producing different ranges of products and the capacity of such units – far bigger than the need for common greases nowadays. Continuous production of highly sophisticated greases has not been possible for many years, but work on the continuous manufacture of lithium complex greases has been reported [16.162, 16.375].

**Tab. 16.8** Continuous production of greases – the three elements of a unit.

| | |
|---|---|
| 1 | A tubular reactor – in which measured quantities of the base grease components are intensely mixed at pressures up to 2000 kPa (20 bar) and temperatures up to 210 °C |
| 2 | A vacuum chamber in which dehydration of the product takes place |
| 3 | A finishing segment – usually a static mixer – in which oil and additives are added and homogenization takes place |

The finishing equipment comprises suitable pumps, filtration units, homogenization units (mills are still common – three rollers, rotor stator mills, and other colloid mills are normally used although high-pressure injection homogenizers are preferable), deaeration units (vacuum pumps), and filling devices with geometries suitable for the types of containers to be filled – in a typical grease plant their range is usually from 400-g cartridges to containers holding 1 metric ton.

Comparison of the different processes used to make metal soap-based greases shows that no single process is optimum for every kind of grease [16.163] and investigation of four processing systems for lithium greases [16.164], including high concentrate saponification [16.165], indicates that manufacturing greases by conventional methods is not necessarily a disadvantage.

### 16.6.2
### Oligourea Greases

Oligoureas are usually prepared in a two-step process. In the first step the diisocyanate and the amine are separately dissolved or dispersed in appropriate amounts of the base oil. In the second step one of the two solutions or dispersions – usually that containing the diisocyanate – is added to the other portion [16.166]. The reaction is rather exothermic and, because of the toxicity of diisocyanates, the vessels must be sealed against the atmosphere during this step and subsequent heating. Because of the sudden and enormous increase in consistency very powerful stirring devices are necessary. To complete the reaction and for optimization of the thickener structure the greases are heated to 180 °C [16.376]. Oligourea greases made in this way are preferably homogenized with kneaders or high-pressure injection homogenizers. The manufacture of oligourea greases in a Stratco contactor has been described [16.167, 16.377] as has the manufacture of greases from preformed oligourea powders [16.168, 16.378].

### 16.6.3
### Gel Greases

The raw materials here are warmed to 60 to 80 °C only, to accelerate the activation and improve the gelation process. This is usually conducted in the same type of single reactor used for the one-step production of metal soap-based greases, but in this instance the consistency required of the product on a technical scale can best, and in many cases only, be achieved with the aid of high-pressure injection homogenizers or rotor–stator colloid mills [16.169], or, for polyester-based greases, with annular gear colloid mills [16.170].

## 16.7
## Grease Rheology

Because greases have rather complex rheological [16.171] properties they have been described as both solid and liquid or as viscoelastic plastic solids [16.172]. In the beginnings of rotational viscosimetry there was some hope of finding a correlation between yield point and cone penetration and attempts to do this continue nowadays [16.173], and the call for replacement of cone penetration is again becoming louder [16.174]. Beyond the yield point, where flow occurs under stress, greases have an apparent viscosity which depends on shear rate, temperature, and shear time, and, to some extent, even on the mechanical pretreatment. This can be explained in simple terms, as, for example, in the NLGI Lubricating Grease Guide [16.175], but when the mathematical background of plastic flow according to Bingham, pseudoplastic flow according to Ostwald, and liquid flow according to Newton is taken into account the subject can barely be treated exhaustively in a single book [16.176]. Some complications resulting from wall slip [16.177] have generated new activity [16.178]. Controlled stress rheometers seem to be the best suited devices for a better insight into grease rheology today [16.179] but will maybe be assisted by velocity imaging nuclear magnetic resonance spectrometers in the near future [16.180].

It cannot surprise that the thickener has some influence on the rheological properties of a grease [16.181]. This was found, for example, when lithium and sodium greases were compared in combined experiments with an impacting-ball apparatus [16.182]. For organoclay greases, on the other hand, different base oils do not enable any generalization [16.183, 16.184]. Of all the thickeners compared varieties of highly dispersed silicic acid seem to respond best rheologically to both low and high temperatures [16.185]. For a series of military greases a quasi linear relationship was observed between apparent viscosity and low-temperature torque [16.186]. With perfluorinated polyethers thickened with PTFE it was possible to show correlation between rheological and lubrication properties [16.187].

Improved low-temperature rheological performance has been reported for lithium greases modified with functional polymers [16.188]. Attempts to gain deeper insight into the dynamic behavior of greases include a theoretical study of rheological wear [16.191, 16.379, 16.380], a study of grease flow in pipelines [16.381], a study of the behavior of greases in oscillating applications [16.382] (a study which identified the limits of greases in comparison with grease pastes in these applications), and a study of their high-temperature structural properties by use of controlled-stress rheology [16.383], to name a few of the more practical examples.

## 16.8
## Grease Performance

The performance of a grease is, again, probably best judged in terms of the thickener. This has recently been done by Schmidt in a practical and condensed way

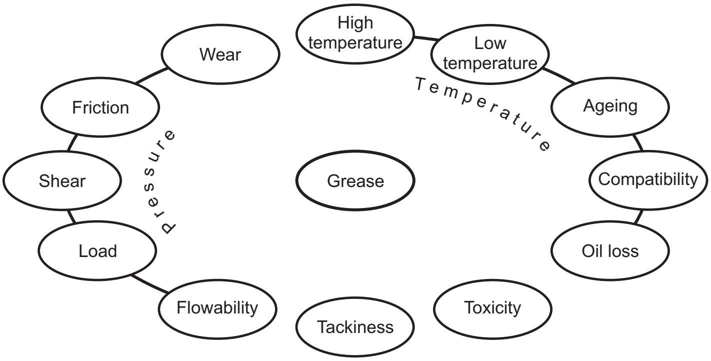


**Fig. 16.6** Twelve phenomena relevant to grease performance.

[16.193]. From a more theoretical view there are twelve main phenomena (Fig. 16.6) which must be considered. Of these twelve, all but two – tackiness and toxicity – are related either to pressure or to temperature. The action or effect of pressure or temperature result in twenty-four requirements (Table 16.9). It is out of question for a real grease to satisfy all these requirements, because there are eleven necessary performance contradictions and thirty-three more or less marked restrictions (Fig. 16.7) [16.194]. The suitability of a grease for both high- and low-temperature applications is, for example, difficult to achieve, although the lower and upper temperature limits of a grease exceed those of its base oil, by analogy with Raoult's law. This means that the thickener acts as an impurity that, on the one hand, hinders crystallization and thus lowers the pour-point of the oil, yet on the other hand, reduces its vapor pressure. The effect can be increased by use of the kinds of polymer employed as pour-point depressants and viscosity index improvers in lubricating oils. The temperature-dependence of the apparent viscosity of a grease – at constant shear rates – is less pronounced than that of its base oil.

Sometimes, usually when a unit is lubricated with a new grease, the performance of the second grease can be affected by the first. Cleaning of the unit is, therefore, always the best solution. The danger resulting from mixing two greases is nevertheless often overestimated and some of the charts describing incompatibilities in the grease literature are even contradictory. Possible incompatibility is best checked experimentally with the so-called Shell-roller test [16.195]. A change in the structure usually leads to softening of the mixture and to a decrease of the dropping point. Mention is to make of two incompatibilities only – that between lithium and sodium soap-based greases and that between inorganic thickened greases and other greases with many additives. Both occur in practical grease lubrication and are caused by the thickeners. Loss in performance otherwise is simply because of the lower efficiency of the first grease. If the greases contain very different and reactive additives this can also result in incompatibility.

Tab. 16.9 Twenty-four properties of greases.

| *Twelve phenomena* | *Twenty-four properties* |
|---|---|
| High temperature | Maximum thermal stability |
| | Minimum evaporation loss |
| | Maximum viscosity |
| Low temperature | No (regular) crystallization |
| | Minimum viscosity |
| Aging | Maximum oxidation resistance |
| | Resistance to changes in structure |
| Compatibility | No reaction with non-ferrous metals |
| | Maximum corrosion inhibition |
| | Maximum polymer compatibility |
| | Immiscibility with foreign liquids |
| | Deflection of foreign solid matter |
| Oil loss | Optimum oil loss[a)] |
| Toxicity | No toxicity |
| | Biodegradability |
| Tackiness | Optimum tackiness[a)] |
| Flowability | Optimum relaxation[a)] |
| | Maximum pumpability |
| Load | Optimum elasticity[a)] |
| | Maximum lubricating film thickness |
| | Maximum emergency running properties |
| Shear | Maximum mechanical stability, or |
| | Optimum relaxation time[a)] |
| Friction | Minimum, or optimum friction[a)] |
| Wear | Minimum wear. |

**a)** According to application.

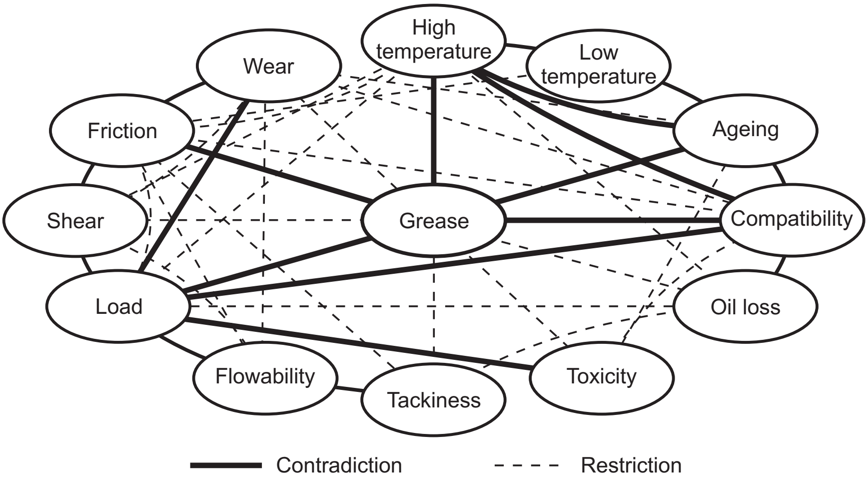


Fig. 16.7 Contradictions and restrictions inherent in the twelve phenomena.

### 16.8.1
### Test Methods

Many test methods are used today; all are meant to judge the single or combined and more or less complex properties of greases. The last summary containing detailed descriptions of ASTM and DIN methods was written by Schultze in 1962 [16.196]; another emphasizing on Russian GOST and former East German TGL methods was published in 1984 [16.197]. The most important ASTM and DIN methods are described in Tables 16.10 and 16.11. Complete collections are published regularly [16.198, 16.199], the same is true for the French AFNOR, the English IP, the Japanese JIS, and some other national collections. The development of international standards (EN and ISO) is slowly proceeding.

**Tab. 16.10** Important ASTM test methods for lubricating greases.

| | |
|---|---|
| ASTM D 0128-95 | Analysis |
| ASTM D 1092-93 | Apparent viscosity |
| ASTM D 1263-94 | Leakage tendencies of automotive wheel bearing greases |
| ASTM D 1264-96 | Water washout characteristics |
| ASTM D 1478-91 | Low-temperature torque of ball bearing greases |
| ASTM D 1742-88 | Oil separation during storage (air pressure method) |
| ASTM D 1743-94 | Corrosion preventive properties |
| ASTM D 1831-88 | Roll stability |
| ASTM D 2509-93 | Load carrying capacity, Timken method |
| ASTM D 3337-94 | Life and torque in small bearings |
| ASTM D 3527-95 | Life performance of automotive wheel bearing greases |
| ASTM D 4049-86 | Resistance to water spray |

**Tab. 16.11** Important DIN (ASTM) test methods for lubricating greases.

DIN 51350-4 (ASTM D 2596-97)
Testing in the Shell four-ball tester, determination of the welding load of consistent lubricants
DIN 51350-5 (ASTM D 2266-91)
Testing in the Shell four-ball tester, determination of the wear parameters of consistent lubricants
DIN 51801-2 (ASTM D 566-97), replaced by DIN ISO 2176
Determination of dropping point of greases
DIN 51802
Testing of rolling bearing greases with regard to their corrosion-inhibiting properties, SKF-Emcor method
DIN 51804-1 (ASTM D 217-97), replaced by DIN ISO 2137
Determination of cone penetration of greases with hollow cone and solid cone
DIN 51804-2 (ASTM D 1403-97), replaced by DIN ISO 2137
Determination of cone penetration of greases with one-quarter cone
DIN 51805
Determination of yield pressure of lubricating greases, Kesternich-method
DIN 51807-1
Test for the behaviour of greases in the presence of water, static test

DIN 51808 (ASTM D 942-90)
Determination of oxidation stability of greases, oxygen method
DIN 51810
Determination of flow behaviour of greases in the rotary viscometer
DIN 51811 (ASTM D 4048-86)
Testing of corrosive effects of greases on copper, copper strip test
DIN 51817
Determination of oil separation from greases under static conditions
DIN 51821-2
Test using the FAG roller bearing grease testing apparatus FE 9

Because of the large number of publications about the different test methods, their improvement, and their comparison, only some surveys concerned with EP [16.200], wear [16.201] and with standard tests [16.202, 16.203, 16.384] or performance tests using real components [16.204–16.208] are cited. One remark about the last group – the FAG FE 8 test, which has become an increasingly important replacement for the SKF R2F and Timken tests in Europe, has found its way to the USA where it is used for the development of greases for all kinds of heavy-duty application [16.385]. The methods themselves are described in Chapters 18 and 19.

One point in dealing with standards and test methods according to Jünemann [16.212] cannot be taken serious enough – critical analysis.

### 16.8.2
### Analytical Methods

The time of chemical separation procedures as described by ASTM D 128 has nearly gone, elemental analysis of greases is nowadays performed by spectroscopic methods, e.g. X-ray fluorescence spectrometry (XRF), inductively coupled plasma atomic emission (ICP), or atomic absorption spectrometry (AAS) [16.213], with attention being directed mostly to methods of preparation [16.214, 16.215]. Infrared spectroscopy was introduced as a means of identifying greases and their components ca 45 years ago [16.216–16.218]. Its use has steadily grown since and been extended to questions of structure, development, and manufacture also [16.219]. Nuclear magnetic resonance spectroscopy (NMR) has also been used to investigate structural questions [16.220] and electron microscopy has enabled not only the scrutiny of soap fibers [16.221] but also study of thermal changes in greases [16.222].

The use of thermogravimetry (TG) is usually limited to investigations of base oils [16.223], but the other thermoanalytical method, differential scanning calorimetry (DSC) [16.224, 16.225, 16.386, 16.387], or a combination of both, is expected to become a valuable tool in the analysis of grease and antioxidant analysis.

Chromatographic methods, e.g. gas (GC) and high-pressure liquid chromatography (HPLC), are mainly used to identify the components of liquid or liquefied grease [16.226, 16.227].

The automation of analytical methods has led to a demand for tools that not only facilitate the documentation of data but also aid their interpretation. This can be achieved with suitable laboratory information systems (LIMS) [16.228, 16.229]. The use of such a system must be accompanied by intensified concern with the efficiency of tests [16.230] and critical analysis of possible errors in the resulting data [16.231, 16.232]. Suitable databases [16.233] or internet resources [16.234] can be also used.

## 16.9
## Applications of Greases

Increased knowledge of base oils and thickener systems enables the selection and naming of greases on the basis of these chemical and physical insights. The selection of a grease is always a compromise between the demands of a customer and the circumstances the grease must face during its operational life – temperature, speed, load including centrifugal forces and vibrations, re-lubrication intervals based on a knowledge of the lubrication points, for applications which can be roller bearings, plain bearings, chassis, joints, 5th wheels, door locks, switches and seals of different design [16.235–16.237]. It has already been mentioned that the desired 24 grease properties described by 12 characteristics sometimes contradict each other.

### 16.9.1
### Rolling Bearings

The rolling bearing industry, one of the key industries for greases, supports all kinds of manufacturer not only with bearings, which can be standard or tailor-made products, but also with consulting and service concerning the design of new equipment and the maintenance of that already in existence. The lifetime of rolling bearings is connected with that of the grease used, especially under extreme conditions [16.238]. Sophisticated test equipment has been developed, mainly to ensure better lifetime predictability for the selected bearing together with the selected grease [16.239, 16.240]. The test rigs SKF R2F and later FAG FE 9 have found great acceptance in Germany [16.241, 16.242].

Clean production and low-noise greases are meeting reliability and lifetime requirements of rolling bearings. The stringent requirements of high-precision bearings, for small bearings in video and audio applications, and bearings for military use have been established for years [16.243, 16.244].

On the basis that ca 80 % of all bearings are grease-lubricated, in 1992 the American Society of Tribologists and Lubrication Engineers published a book about the life factors of rolling bearings [16.245]; the German Society for Tribology published another in 1994 [16.246]. These handbooks describe calculation of the lifetime of bearings, taking into consideration the effects of the tribological system. The latter book focuses on the so-called $a_{23}$ value, which describe the influence of a grease taking into account bearing size, speed, viscosity, and temperature. Current knowledge of the factors affecting calculation of the lifetime of these bearings has been released in the new handbook of the German Society for

Tribology, the second edition of which has been published in September 2006 [16.392]. In Ref. [16.393] it is shown that the lifetime can be prolonged substantially by changing specific influencing factors which do not reduce the lifetime but, on the contrary, increase it. Design of a grease reservoir for the bearings containing a greater volume of grease will also increase the lifetime of rolling bearings [16.394].

In general greases used in rolling bearings such as ball-, deep groove ball-, thrust ball-, spherical-, taper-, cylindrical- or needle roller bearings must have good working stability. This can be checked by the prolonged penetration test and the Shell-Roller test (ASTM D 1831) [16.247]. Conventional lithium greases in the NLGI class 2 are recommended for most types of bearing at working temperatures up to 120 °C. Greases of the NLGI class 1 are preferred for needle bearings. For bearings exposed to temperatures above 120 °C complex soap or polyurea thickened greases are preferable. For bearings that must operate under high load and/or low speed the base oil viscosity must be 200 $mm^2\ s^{-1}$ minimum at 40 °C. Greases for cold climates or for aerospace or military use have to ensure performance down to below –70 °C. Low-temperature performance can be checked with the low-temperature torque (ASTM D 1263), the low-temperature penetration (AFNOR NF T 60-171) and the flow pressure (DIN 51805) tests [16.248–16.250]. Greases of that kind need base oils with sufficiently low pour points. In many military applications long-life properties are also required; these can be fulfilled by use of synthetic base oils only [16.251]

Because plain bearings are often exposed to moisture or water, calcium soap-thickened greases are recommended. When open housings are used in a dusty atmosphere frequent re-lubrication makes it possible to wash out the contaminated grease.

The $n \times d_m$ value (speed multiplied by the average of inner and outer diameter of a bearing) as a means of selecting the right base oil viscosity for a grease can be accepted only as a rule of thumb; its dependence on base oil viscosity has not yet been established and a commonly accepted test method is not yet available [16.252].

### 16.9.1.1 **Re-lubrication Intervals**

The lubrication interval $t_f$ is based on the $F_{10}$ value of a standard grease as agreed in DIN 51 825 [16.253] under normal environmental conditions with temperatures up to 70 °C and a mean bearing load of $P/C < 0.1$. Figure 16.8 and Table 16.12 take into consideration the type of bearing and the speed. For each 15 K temperature increase the re-lubrication interval is said to be reduced by 50 %. Severe working conditions reduce the lubrication interval $t_f$ to a reduced lubrication interval $t_{fq}$.

$$t_{fq} = f_1 \times f_2 \times f_3 \times f_4 \times f_5 \times t_f \tag{16.1}$$

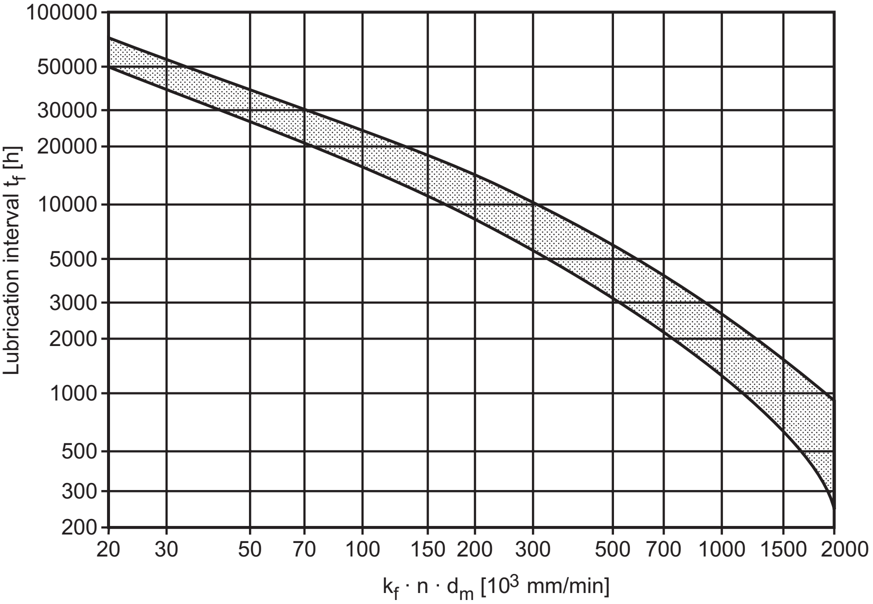


**Fig. 16.8** Re-lubrication intervals.

**Tab. 16.12** Relationship between bearing type and correction factors for re-lubrication intervals (GFT Worksheet 3).

| ***Bearing type*** | | ***$k_f$*** |
|---|---|---|
| Deep grove ball bearings | Single row | 0.9–1.1 |
| | Double row | 1.5 |
| Angular contact ball bearings | Single row | 1.6 |
| | Double row | 2 |
| Spindle bearings | $\alpha = 15°$ | 0.75 |
| | $\alpha = 25°$ | 0.9 |
| Four point bearings | | 1.6 |
| Self-aligning ball bearings | | 1.3–1.6 |
| Thrust ball bearings | | 5–6 |
| Angular contact thrust ball bearings | Double row | 1.4 |
| Cylinder roller bearings | Single row | 3–3.5[a)] |
| | Double row | 3.5 |
| | Full complement | 25 |
| Cylindrical roller thrust bearings | | 90 |
| Needle roller bearings | | 3.5 |
| Tapered roller bearings | | 4 |
| Barrel roller bearings | | 10 |
| Spherical roller bearings without lips (E-design) | | 7–9 |
| Spherical roller bearings with center lip | | 9–12 |

**a)** $k_f = 2$ for radial load or increasing thrust load and
$k_f = 3$ for constant thrust load.

The reduction factors are shown in Table 16.13.

**Tab. 16.13** Reduction factors for re-lubrication cycles. Reduction factors $f_1$ to $f_5$ for poor operating and environmental conditions (GfT Worksheet 3).

***Effect of dust and moisture on the bearing contact surfaces***

| | |
|---|---|
| Moderate | $f_1$ = 0.7–0,9 |
| Strong | $f_1$ = 0.4–0.7 |
| Very strong | $f_1$ = 0.1–0.4 |

***Effect of shock loads and vibrations***

| | |
|---|---|
| Moderate | $f_2$ = 0.7–0.9 |
| Strong | $f_2$ = 0.4–0.7 |
| Very strong | $f_2$ = 0.1–0.4 |

***Effect of high bearing temperature***

| | |
|---|---|
| Moderate (up to 75 °C) | $f_3$ = 0.7–0.9 |
| Strong (75–85 °C) | $f_3$ = 0.4–0.7 |
| Very strong (85–120 °C) | $f_3$ = 0.1–0.4 |

***Effect of high loads***

| | |
|---|---|
| $P/C$ = 0.1–0.15 | $f_4$ = 0.7–1.0 |
| $P/C$ = 0.15–0.25 | $f_4$ = 0.4–0.7 |
| $P/C$ = 0.25–0.35 | $f_4$ = 0.1–0.4 |

***Effect of air current passing through the bearing***

| | |
|---|---|
| Light current | $f_5$ = 0.5–0.7 |
| Strong current | $f_5$ = 0.1–0.5 |

In re-lubrication it is usually impossible to remove the used grease. Consequently the re-lubrication interval $t_{\text{fq}}$ has to be reduced by 30 to 50 %. General advice on the amount of grease necessary for re-lubrication is given in Table 16.14.

**Tab. 16.14** Amount of grease necessary for re-lubrication. Amounts of grease lubrication (GfT Work sheet 3).

***Re-lubrication quantity $m_1$ for weekly to yearly re-lubrication***

$m_1 = D \times B \times x$ [g]

| Re-lubrication | $x$ |
|---|---|
| Weekly | 0.002 |
| Monthly | 0.003 |
| Yearly | 0.004 |

***Re-lubrication quantity $m_2$ for extremely short re-lubrication interval***

$m_2 = (0.5\text{–}20) \times V$ [kg h$^{-1}$]

***Re-lubrication quantity $m_3$ before restarting after several year of standstill***

$m_3 = D \times B \times 0.01$ [g]

$V$ = free space in the bearing

$(\pi/4) \times B \times (D^2 - d^2) \times 10^{-9} - (G/7800)$ [m$^3$] or
$(\pi/4) \times B \times (D^2 - d^2) \times 10^{-9} - ([G' \times 0.4536]/7800)$ [m$^3$]

$d$ = bearing bore diameter [mm]
$D$ = bearing outside diameter [mm]
$B$ = bearing width [mm]
$G$ = bearing weight [kg]
$G'$ = bearing weight [lb]

### 16.9.2
### Cars, Trucks, Construction Vehicles

Most modern cars do not need re-lubrication, with the exception of door hinges, lock mechanisms, and battery poles. But among the approximately 30 hidden greases (Fig. 16.9 and Table 16.15) in a modern car [16.254, 16.255] only the constant-velocity (CV) joint greases are required in substantial quantities. Although improved conventional lithium greases containing molybdenum disulfide are still in use [16.256], lithium complex or polyurea greases are already preferred in some modern cars and this usage will increase in the future [16.257]. Most of the greases used in cars, for example the greases for CV joints, hub units, starters, alternators, seat adjustments, clutches release bearings, belt–pulley bearings, window levers and windshield wiper gears, are specified and approved by the large motor companies and developed in close cooperation with the grease manufacturers. For the same application, however, different motor companies have different grease specifications

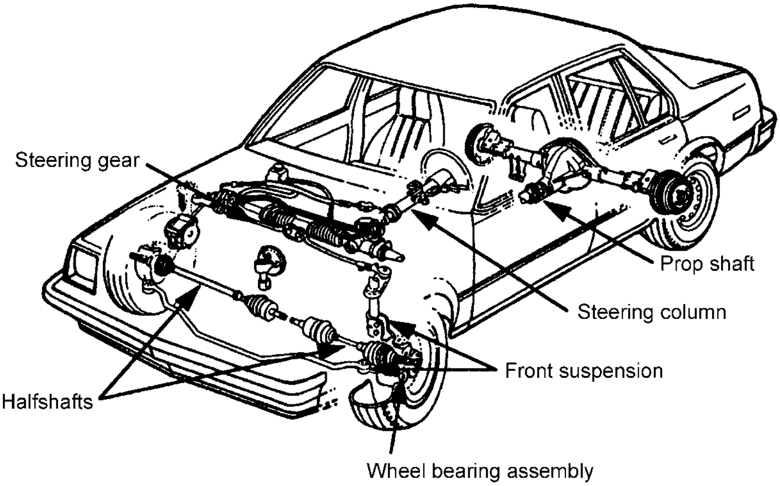


**Fig. 16.9** Greases hidden in a modern car.

Tab. 16.15 Thirty hidden greases – a partial list of grease applications.

| ***Powertrain*** | ***Brake system*** |
|---|---|
| Belt–pulley bearing | ABS follower/motor bearings |
| Clutch bearing/spline | Caliper pin |
| Cruise control module | |
| CV joint | *Electrical* |
| Fan clutch bearing | |
| Fuel pump bearing | Alternator bearing |
| Turbo/supercharger | Electrical contacts |
| | Horn contact |
| *Body hardware* | |
| | *Heating/cooling* |
| Door check arms/hinges | |
| Door locks/latches | Cable actuators |
| Electric antenna gear/clutch | Temperature sensor |
| Remote control mirror assembly | Visco fan bearing |
| Seat adjustment gears | Water pump bearing |
| Sunroof rails | |
| Window levers | *Other* |
| Windshield wiper gear | |
| | Wheel bearing |
| | U-Joint |

and approvals; for example, most European and US motor companies prefer lithium complex greases in the front wheel bearings, whereas Japanese manufacturers prefer polyurea greases.

ASTM D 4950 (Tables 16.16 and 16.17) describe the minimum requirements of current greases in automotive service–fill applications for passenger cars, trucks, and other vehicles operating under various service conditions [16.258]. Grease packs fulfilling these minimum requirements can have the NLGI certification marks as shown in Fig. 16.10, these labels are provided by the NLGI on request.

Tab. 16.16 ASTM D 4950 Specifications LA and LB.

***Chassis grease classifications – intended use of chassis (L) classified greases:***

***LA Classification***

Chassis components and universal joints under mild duty

- Frequent re-lubrication
- Non-critical applications

***LB Classification***

Chassis components and universal joints under mild to severe duty

- Prolonged re-lubrication intervals
- High loads
- Severe vibration
- Exposure to water or other contaminants

**Fig. 16.10** NLGI certification marks.

**Tab. 16.17** ASTM D 4950 Specifications GA, GB and GC.

Intended use of wheel bearing (G) classified greases:

***GA Classification***

Service typical of wheel bearings operating under mild duty

- Frequent re-lubrication
- Non-critical applications

***GB Classification***

Service typical of wheel bearings operating under mild duty/moderate duty

- Normal urban, highway and off-highway service

***GC Classification***

Service typical of wheel bearings operating under mild duty/severe duty

- High bearing temperatures
- Frequent stop and go service (buses, taxis, police)
- Severe braking service (trailer towing, heavy towing, mountain driving)

Lithium-based multipurpose greases have replaced several other greases for the re-lubrication of trucks and construction equipment. Conventional lithium soap based greases that require frequent re-lubrication are still in use for the wheel bearings of trucks and trailers. Modern trucks and trailers with prolonged oil-drain intervals require lithium complex greases with semi-synthetic or fully synthetic base oils. Lithium greases containing black solid lubricants are recommended for 5th-wheel applications, for chassis points, and for plain bearings of construction equipment. Many trucks and buses and much construction equipment uses centralized lubricating systems, designed for semi-fluid greases of NLGI class 00 or 000, for onboard re-lubrication. Other systems require greases of NLGI class 2. Lithium greases optimized for low temperature applications, good pumpability, and low oil separation are recommended.

### 16.9.3
### Steel Mills

In Europe calcium complex and sometimes polyurea greases based on mineral oil are used for lubricating continuous casting equipment. The US market prefers aluminum complex and polyurea greases and Japanese equipment manufacturers mainly recommend polyurea greases. Calcium sulfonate complex greases are achieving increasing commercial acceptance in Europe and the USA, because of their high EP values and excellent corrosion-protection properties. Some rolling bearing manufacturers equip continuous castings with double- or triple-sealed bearings that are greased for life, preferably with synthetic polyurea greases. When the sealing of the bearings is not perfect re-lubrication has a cleaning function. Contaminated grease that could result in limited lifetime is squeezed out.

Conventional EP lithium greases, calcium complex greases, calcium sulfonate complex greases, lithium complex greases, and aluminum complex greases, all based on mineral oils are used in hot rolling equipment. In India a successful trial has been conducted with a locally produced titanium complex grease [16.259]. Most customers require a minimum base oil viscosity of 200 $mm^2 s^{-1}$ at 40 °C [16.260]. The rotating shift system is typically used for hot-rolling lubrication. In general the bearings are not re-greased during operation, only during maintenance.

Many different greases, e.g. conventional EP lithium greases, lithium complex greases, calcium complex greases, and calcium sulfonate complex greases, all based on mineral oil, are used in cold rolling equipment. Modern greases use a lithium–calcium mixed soap for improved water resistance. Some calcium complex greases have helped to prolong the lifetime of bearings in the pickling section significantly.

### 16.9.4
### Mining

Open pit mines which use hydraulic excavators and dumper trucks and/or wheel bucket excavators and belt systems to ship the spoil and coal are especially large grease consumers. Lithium, lithium–calcium mixed-soap-based or lithium complex greases with base oil viscosities > 350 $mm^2 s^{-1}$ at 40 °C and an effective EP and AW additive package are used. Black solid lubricants are also recommended. Because the equipment is exposed to dust or water and mud, the sealing efficiency of the bearings determines their lifetime and must be supported by a grease.

Kilometers of wire rope are used on the excavators or drag lines and in the underground mines. During production of the wire ropes lubricants are applied to ensure corrosion protection and to minimize friction of the single wires when the rope stretches under load. The amount of lubricant in the core should be ca 25 % *w/w* of the core.

Lay-up lubricants based on wax-resin are applied during manufacture to ensure lubrication of the individual wires and stands. After production the wires must have corrosion protection; this can be achieved by painting or by applying a bitumen-based lubricant with a solvent.

During operation in some countries the wire ropes are maintained by cleaning the surface and re-lubrication. In many countries bitumen-based products are still in use, but are being replaced by bitumen-free greases or even biodegradable lubricants. It is essential to apply the lubricant on top of the sheave-wheel or shortly after bending over the winder drum when a large twisting motion or unloading opens the wires and sucks in the lubricant.

Winch greases, preferable lithium complex greases with improved water resistance, ensure prolonged winch lifetimes up to several years.

### 16.9.5
### Railroad, Railway

Depending on the design of the driving system of the locomotive a gear oil or a gear grease is required. The poor sealing properties of these kind of gears lead to leakage; this is usually minimized by use of bitumen-based products. Modern greases are thickened with lithium or sodium soap and based on mineral oils of up to 2000 $mm^2 s^{-1}$ at 40 °C. Traction motor gear greases are not described in terms of consistency; the apparent viscosity is usually checked with a Brookfield viscometer, for example with a number 3 spindle at 93 °C and at 4 rpm and giving results of 5000 to 10 000 cP [16.261].

The axle bearings are greased with conventional lithium EP greases. The development of ready-to-build-in axle boxes and the increasing speed of modern trains have led to improvement of the high-temperature performance and lifetime of greases.

Switch lubrication, wheel flange lubrication (mainly used in Europe), and rail track-side lubrication in curves, used mainly in the USA and Canada, cause environmental problems. Biodegradable greases based on esters have better wear-protection and consumption performance [16.262–16.264]. These greases must be sprayable because of the means of application.

### 16.9.6
### Gears

Fibrous sodium soap greases of NLGI-class 0, 00, or even 000 consistency are used. Although the load-carrying capacity, anti wear properties, and adhesion of these greases is especially good, lithium-based greases and greases based on synthetic hydrocarbons or polyglycols are gaining in importance

Rather sophisticated greases are used in the gears of do-it-yourself tools. Greases in modern drilling machines must fulfil several tasks – lubricate the gears, the bearings, and the piston responsible for the hammer drilling operation. Power tools for professional use have even more stringent requirements with regard to low temperature torque, high temperature performance, and lifetime.

Girth gear drives are a type of drive widely used for many large systems in the primary industries [16.265]. These open gears require an adhesive grease based on high-viscosity oils. Such greases are usually sprayed on the teeth by means of a centralized lubricating system. Precise adjustment is required, because these girth gear drives occupy a key position in manufacturing processes. Most manufacturers of adhesive greases have, therefore, developed a maintenance scheme with a tribological emphasis and offer this as a service to their clients.

### 16.9.7
### Food-Grade Applications

Greases for lubricating machines used in food processing must fulfil specific requirements relating to food legislation, human health protection, taste, and odor. These properties must be approved by internationally accepted tests and organizations [16.266].

The FDA (Food and Drug Administration, USA) lists ingredients permitted for use in food-grade grease formulations (21 CFR §178.3570); white oils are listed in 21 CFR §178.3620. The USDA (United States Department of Agriculture) has approved the finished products. USDA H-1-approved products can be used in food processing in which incidental and unavoidable contact between food and lubricant can occur. For white and synthetic oil-based products maximum contamination of 0.1 g $kg^{-1}$ food is allowed. Since USDA stopped its activities in 1999. The NSF (National Sanitation Foundation, Ann Arbor, Michigan, USA) has taken over the role of USDA by developing standards and certifying products being used in the food industry or in drinking-water systems.

### 16.9.8
### Textile Machines

Because greases could contaminate textiles, they are formulated with white oils and thickened with water-soluble sodium soaps, and are thus removable by washing.

### 16.9.9
### Application Techniques

Greases are applied by hand and brush, with gloves, grease dispensers, grease guns, and pumps from all kinds of container (e.g. tins, pails, drums, or even bulk) [16.267]. The pumps can be driven manually, by use of a power supply, or by compressed air. Most pump systems use a follower plate fitted into the container. The plate will follow the grease level. The rubber lip must follow the inner surface of the grease container, to avoid any sucking in of air.

Trucks, buses, construction and forestry equipment, continuous casting and cold-rolling equipment, paper mills, printing machines, presses, excavators for open pit mines, and many more machines used industrially have centralized lubricating systems. These systems can be designed for greases in NLGI-class 2 as parallel type for single or dual line systems usually using valves or manifolds, or as series type usually using progressive piston-type metering manifolds. For NLGI-classes 00 and 000 single-line systems with valves, of similar design to oil-centralized lubrication systems, are used. The amount of grease and the re-lubrication time is mechanically or electronically adjustable. The greases are selected for the lubrication point, but have to fulfil specific requirements, e.g. pumpability at low temperature, reduced oil separation, and no tendency to hardening in progressive plunger metering devices.

The greases must be free from air bubbles, because transport of the grease to the lubricating points will be affected.

Greases should be kept clean during storage and application, because any contamination will increase the risk of reduced lifetime. Because oil-wet surfaces catch any dust, grease containers must be stored closed [16.268].

### 16.9.10
### Special and Lifetime Applications

Fire-resistant greases as described by the British Coal Specification [16.269] are still based on phosphate esters or their mixtures with other base oils. Since Spengler and Wunsch described the applications of greases in precision instruments in 1970 [16.270] nothing essential has changed in this area. The same is true for applications of greases in nuclear power plants [16.271]. In spacecraft or high vacuum applications new findings have been reported [16.272]. Greases based on perfluoropolyalkylethers are still doing their job [16.273, 16.274], but greases based on multiply alkylated cyclopentane have also generated interest [16.275, 16.276]. These applications are mainly meant for lifetimes up to decades and the limiting factors are mainly wear [16.277] and high operating temperature [16.278].

### 16.9.11
### Applications with Polymeric Materials

There are three ways in which greases and polymeric materials can be present together: polymers can be contained in greases as thickeners, as solid lubricants, and as additives; they can act as sealants [16.279]; and they can be one or even both of the solid partners in friction couples. Although base oils are the primary consideration [16.280], when the interaction of greases with sealants is important [16.281, 16.282] – the performance of oils is described in Chapter 11 – the influence of additives, and among these especially EP additives, must always be considered [16.283]. Even migration of soap molecules into a polymer can occur [16.284]. An enormous number of polymers is known and used as components of machinery [16.285]. Although the tribological fundamentals for use of polymeric solids have been described in detail by Bartenev and Lavrentev in 1981 [16.286], by Uetz in 1985 [16.287], and by Yamaguchi in 1990 [16.288], and a brief overview over the tribology of plastics was published in 1994 [16.289], the lubrication of polymers has been described in detail only by Spengler and Wunsch [16.290].

An early test method for the action of lubricants on polymeric materials was the pendulum according to Barker [16.291]. During the automation of this process a more meaningful friction and wear tester was developed [16.292, 16.293]. It proved also to be necessary to test the tendency of a polymer to form cracks under the influence of its lubricant and tension [16.294].

The lubrication of polymers or polymers and metals as friction couples is one field in which silicone greases and, better, fluorinated silicone greases, or, even better, greases based on perfluoropolyalkylethers are of some advantage. Although silicone greases made with highly dispersed silicic acid should be used as general lubricants and as sealants [16.295], silicone greases made with the usual thickeners are sometimes preferable to other synthetic greases. With calcium stearate or 12-hydroxystearate, for example, they are permitted in food applications [16.296], although even here compatibility problems can occur, for example with POM (polyoxymethylene) and dimethylsilicone oils [16.297] and – this is primarily necessary for the base oils – creeping should be avoided with the aid of an epilamization agent, i.e. a fluorinated polymer applied to the polymer in high dilution and which forms a kind of network on the solid surface, thus dramatically reducing surface tension [16.298].

## 16.10
## Grease Market

In 2004 36.1 million tons of lubricants were used worldwide. Of these 1.2 million tons were greases [16.299]. The NLGI (the National Lubricating Grease Institute) has reported that 828,442 tons of grease were produced in 2004 [16.300] and it must be considered that only approx. 67 % of the world's grease manufacturers contributed to this figure.

Conventional lithium greases (56.9 %) are the most used. Lithium complex greases account for 15.1 % and are followed by conventional calcium greases with 8.5 % (Table 16.18).

**Tab. 16.18** Global share of thickeners (2004).

| *Thickener system* | *%* |
|---|---|
| Conventional lithium soap | 56.9 |
| Lithium complex soap | 15.1 |
| Conventional calcium soap | 7.4 |
| Aluminium complex soap | 4.8 |
| Polyurea | 4.6 |
| Calcium complex soap | 3.1 |
| Organophilic clay thickeners | 2.5 |
| Sodium soap | 1.4 |
| Anhydrous calcium soap | 1.1 |
| Other metallic soap | 0.7 |
| Other thickeners | 2.4 |

Although lithium soap based greases are the most used products worldwide, there are significant differences among local markets with regard to other grades of grease. In the US and Canadian market lithium complex greases (33.3 %) are the most used. Aluminum complex greases (9.3 %) are also above the global average. In Europe the calcium complex greases (6.2 %) are above the global average, but behind of lithium complex grades (10.4 %). In Japan the polyurea greases (21.4 %) are behind conventional lithium greases (58.2 %); both figures are above the global figures (Table 16.19). Among the calcium complex soap type the calcium sulfonate complex soaps hold 40 %, steadily increasing.

**Tab. 16.19** Local share (%) of high-temperature thickeners (2004).

| *Thickeners* | *Global* | *USA* | *Europe* | *Japan* |
|---|---|---|---|---|
| Lithium complex | 15.1 | 33.3 | 10.4 | 1.8 |
| Aluminium complex | 4.8 | 9.3 | 5.0 | 1.7 |
| Polyurea | 4.6 | 6.4 | 2.1 | 21.4 |
| Organophilic clay | 2.5 | 5.4 | 2.2 | 0.7 |
| Calcium complex | 3.1 | 4.1 | 6.2 | 0.3 |

The main consumer market is the automotive market, with ca 50 %. Depending on the specific structure of a country it is less than 50 % in highly industrialized countries and more than 50 % in less industrialized countries. The grease used are conventional lithium greases, conventional calcium greases, and often sodium greases for re-lubrication of trucks, construction, farming, and forestry equipment, and old-fashioned cars.

The industrial grease market is divided into a wide range of different and tailor-made greases. Big consumers are steel mills, mines, especially open-pit mines, railroad companies, motor companies, and manufacturers of all kind of machinery; they rely on many suppliers. Often these suppliers emphasize the performance of a wide variety of greases. Among industrial grease customers the roller bearing industry is very important; in addition to high-quality conventional lithium greases they increasingly need specific greases to fulfil their customers' requirements.

In the field of biodegradable greases in Germany products used in switches have been completely replaced by ester based biodegradable greases. Trials with biodegradable wheel-flange greases in Austria, The Netherlands, and Germany [16.301] have been successfully completed. Testing in Canada of a biodegradable grease for rail track-side lubrication proved its superior load-carrying and wear-protection performance [16.302].

## 16.11
## Ecology and the Environment

Pre-industrial greases consisted of tallow or vegetable oils and their reaction products with lime; they were therefore, centuries ago, not merely ecologically compatible lubricants, but even rapidly biodegradable. When mineral oils took over, most of the thickeners and some of the additives remained ecologically acceptable. The ecological and environmental aspects of modern base oils have been described in detail in Chapter 7. Of all the vegetable oils that could be chosen as base oils for greases, in Central Europe only rape seed oil has been introduced on a technical scale [16.303] and only for loss-lubrication applications. Initially calcium 12-hydroxystearate was chosen as the most suitable thickener [16.304]. Greases of that kind proved superior to lithium- and mineral oil-based general-purpose grades (Fig. 16.11). Clay-based greases were also tried, but did not come up to expectations [16.305]. The limitations of vegetable oil-based greases soon led to the introduction of transesterified vegetable oils [16.306] and synthetic esters. The latter have been described in detail in Chapter 5. Because of industrial demands greases based on calcium 12-hydroxystearate were usually soon replaced by lithium and lithium–calcium greases [16.307–16.309]. Polyurea greases [16.310], aluminum complex greases [16.311], and titanium complex greases [16.312] followed.

The biodegradability of greases essentially reflects the biodegradability of their base oils, although the CEC L-33 test had to be modified for them [16.313] and in Germany DIN 51828 was introduced [16.314]. In round robin tests greases based on rape seed oil and calcium 12-hydroxystearate achieved biodegradability close to 100 %. Methods enabling the use of emulsifiers led to significantly lower results [16.315].

Biodegradability is one aspect of the necessary reduction of the toxic potential of greases. The elimination of toxic components such as polycyclic hydrocarbons from base oils is almost complete. This is also true for chlorine substitution. The substitution of antimony and lead cations and nitrite anions in additives is not yet complete, because it is not easy to maintain the same performance level, but the work by the suppliers of additives remains focused on minimizing ecotoxicity [16.316–16.319]. In Germany the demand for environmentally harmless products [16.320] became an additional aspect of the performance of greases; changes in the demands for water pollution class (WGK) 0, that came into force in 1999 aggravated the situation. The most suitable antioxidants are only accepted in concentrations which are too low, hydrocarbons including polyalphaolefins, white oils, and low-viscosity polybutenes are not accepted and the most suitable soaps are also excluded. In Sweden a project, called Ren smörja i Göteborg, is gaining attention. Here the thickeners are regarded as a separate group [16.321].

Although experiences with biodegradable greases are promising [16.322], and although customers should be encouraged to try these in bearing applications that

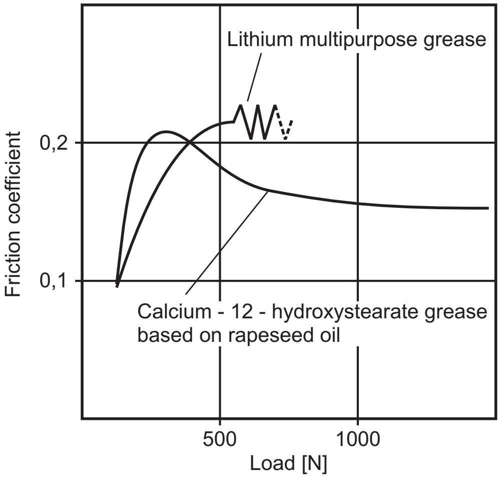


**Fig. 16.11** The friction–load behavior of rapeseed oil-based calcium 12-hydroxystearate grease.

used to be the domain of mineral oil-based greases [16.323], some problems, mainly relating to legal aspects of waste management [16.324] and the balance between ecological requirements and economical possibilities [16.325], remain unsolved. Therefore the third and possibly most important aspect of applying greases in a manner that is both environmentally responsible and economical is to minimize the amount of lubricant necessary and to maximize lifetime by using the most inert materials [16.326, 16.327]. Growing awareness of a rising "peak oil" problem is expected to contribute to acceleration of corresponding efforts [16.388].

## 16.12
## Grease Tribology

General aspects of friction and wear [16.328], and of lubricating oils and their viscosity [16.329], have been described in detail in Chapters 2 and 3. Recent tribological research on greases has dealt with the time-dependence of their behavior under stress as a fourth factor in addition to temperature, pressure, and thickener properties [16.330]. Behavior in elastohydrodynamic (EHL) contacts, where the thickness of grease films has been reported to exceed that of their base oil under comparable conditions [16.331, 16.332], but which has also been reported to decrease to some extent during operation [16.333], and the behavior under starved conditions, when the load is supposed to be carried by thickener particles deposited in the center of the contact [16.334], has also been of concern. Soap deterioration is another important subject [16.335, 16.336, 16.389]. The field of research is widening every day, from more practical problems, for example electroerosion in rolling bearings [16.390], to more theoretical problems, for example the flow of magnetorheological and electrorheological fluids (and the application of a simplified Herschel–Bulkley model) [16.391]. A combination of simple numerical and experimental methods has been used as a grease design tool [16.337]. A few published reflections, focusing on problems concerning the critical energy levels [16.338] and the chaotic behavior [16.339] of greases and the peculiarities of tribometry, the collective term for the measuring techniques and test methods used in tribology, have attracted interest [16.340].

# 17
# Solid Lubrication

*Christian Busch*

Lubricants are used between surfaces which are in tribological contact, and move relative to one another, to achieve a particular value of the coefficient of friction or to reduce the wear of rubbing surfaces, or both. Solid lubricants are required for lubrication under extreme conditions where the bearing surfaces in tribological contact must still be effectively separated. The life of lubricated machine parts depends on the functional and tribological design and optimization of lubricants as calculable functional elements. To make a systematic choice of a suitable lubricant it is absolutely necessary to understand the relationship between friction, wear, and lubrication and the interaction between the elements of the tribological system and the specific properties of each element.

Special attention must be given to the different stress factors and the structure of the tribological system (see Chapter 2).

Products containing solid lubricants are often used to solve problems, particularly in boundary and mixed frictional states when high specific loads are applied to sliding surfaces and at very low hydrodynamically effective speeds but also in critical applications where, e.g., the lubricant must perform over a wide temperature range or under extreme temperature conditions, for example in aviation and in rocket technology.

Dry lubrication with solid lubricants is also required in nuclear reactors, in high vacuum applications, in aggressive environments, and in applications where contamination by lubricating oils or greases cannot be tolerated.

## 17.1
## Classification of Solid Lubricants

A solid lubricant is often defined as any solid material which reduces friction and/or wear of contacting surfaces in relative motion. A vast range of materials and coatings could be judged to behave as solid lubricants on the basis of this definition. Various systems are used to classify the different types of solid lubricant. An arbitrary, but useful, classification is into structural lubricants, mechanical lubricants, soaps, and chemically active lubricants (Sections 17.1.1 to 17.1.4, respectively).

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The main purpose of all these substances is to build up a continuous adherent hard or soft film on the rubbing surfaces. These films can be applied by mechanical, chemical, electrochemical or physical processes, for example dipping, lapping, painting, immersion, electrolysis, electrophoresis, spraying, plating, welding, baking, cupping, sintering, or ionic plating in vacuum.

### 17.1.1
### Class 1: Structural Lubricants

The most widely used solid lubricants are graphite and molybdenum disulfide. Their satisfactory lubricating properties are assumed to result from their layered lattice structures. In addition to these two substances are other solids, for example metal halides and sulfides, which have, in the main, inherent lubricant properties, a lamellar hexagonal crystal structure, and are usually anisotropic (Table 17.1).

**Tab. 17.1** Structural lubricants.

| *Name* | *Formula* |
|---|---|
| Graphite | C |
| Graphite fluoride | $(CF_x)_n$ |
| Molybdenum disulfide | $MoS_2$ |
| Molybdenum diselenide | $MoSe_2$ |
| Tungsten disulfide | $WS_2$ |
| Tungsten diselenide | $WSe_2$ |
| Niobium disulfide | $NbS_2$ |
| Niobium diselenide | $NbSe_2$ |
| Tantalum disulfide | $TaS_2$ |
| Tantalum diselenide | $TaSe_2$ |
| Titanium disulfide | $TiS_2$ |
| Titanium diselenide | $TiSe_2$ |
| Titanium telluride | $TiTe_2$ |
| Cerium fluoride | $CeF_3$ |
| Barium hydroxide | $Ba(OH)_2$ |
| Cadmium chloride | $CdCl_2$ |
| Cobalt chloride | $CoCl_2$ |
| Zirconium chloride | $ZrCl_2$ |
| Lead chloride | $PbCl_2$ |
| Lead iodide | $PbI_2$ |
| Boron nitride | BN |
| Silver sulfate | $Ag_2SO_4$ |
| Borax | $Na_2B_4O_7$ |
| Talc | $Mg_3(OH)_2Si_2O_{10}$ |
| Mica | $KAl_2(Si_3Al)O_{10}(OH)_2$ |

Other than graphite and molybdenum disulfide these are not yet extensively used in industry.

## 17.1.2
## Class 2: Mechanical Lubricants

There are different types of substances within this class whose lubricating effect is based on different physical and mechanical properties or special conditions. A common classification divides these lubricants into self-lubricating substances, substances that need a supporting medium to create lubricating properties, and substances with indirect lubricating properties based on their hardness.

### 17.1.2.1 Self-Lubricating Substances

These can be classified as organic compounds (Table 17.2), metal films (Table 17.3), chemical surface layers, and glasses.

#### Organic Compounds

**Tab. 17.2** Self-lubricating organic substances.

| ***Linear polymers (thermoplastic types)*** |
|---|
| Polytetrafluoroethylene (PTFE) |
| Fluoroethylenepropylene (FEP) |
| Perfluoroalkoxy (PFA) |
| Polyethylene (PE) |
| Polypropylene (PP) |
| Polyurethane (PU) |
| Polyamide |
| Polyacetals |
| Polyterephthalate |
| Polysiloxanes |
| Nylon |
| ***Cross-linked polymers (thermosetting types)*** |
| Phenol–formaldehyde |
| Urea–melamine–formaldehyde |
| Epoxy resin |
| Phenolic resin |
| Unsaturated polyester resin |
| Polyimides |

The sliding characteristics of polymers depend on their chemical nature and the mating partner. The permissible load is a function of heat dissipation. Temperature changes have little influence on the friction characteristics of polymers. Addition of $MoS_2$, graphite, and metal powder improves the frictional characteristics of the polymers and increases their hardness. Graphite also increases the elasticity module of PTFE.

### Metal Films

**Tab. 17.3** Self-lubricating metal films.

| | |
|---|---|
| Lead | Pb layers |
| Tin | Sn layers |
| Silver | Ag layers |
| Indium | In layers |
| Barium | Ba layers |
| Gold | Au layers |
| Aluminum | Al layers |
| Nickel | Ni and Ni–Cr alloys |
| Copper | Cu and Cu alloys |
| Zinc | Zn and Zn alloys |

Friction can be reduced by the coating of the body material surfaces with a thin film of a soft metal, because the friction depends on the shear strength of the soft metal film. The durability depends on the film hardness, homogeneity, and adhesion. The lubricating effect of soft metal layers is limited by their melting point.

### Chemical Surface Layers (Conversion Films)

In addition to the naturally occurring oxide films present on the surface of most base materials exposed to air, other solid lubricant films can be formed by chemical or electrochemical action on the metal surface.

Chemical surface coatings such as zinc, iron, or manganese phosphates behave similarly to soft metal coatings but consist not of a metal but of metal salts. Bonderization (phosphate treatment) creates a thin, microcrystalline, strongly adhering phosphate layer on the metal surface; this reduces the coefficient of friction and the danger of seizure during the running-in period. The lubricating efficiency of the layer, which is normally 2–5 μm thick, is based on its lower shear strength in comparison with the metal.

### Glasses

The structure of glasses consists of random three-dimensional networks in which the formation of chains or sheets are possible. The constituents of glass are divided into network formers ($SiO_2$, $B_2O_3$, $Al_2O_3$, $Na_2O$, etc.) and network modifiers ($K_2O$, MgO, CaO, PbO, etc.). The strongest bonds in these glasses are the Si–O bonds with an average bond distance of 1.62 Å. There is no absolute Si–O bond distance in a glass because of the absence of symmetry and this means that glass softens and has no fixed melting point.

The lubrication properties of glass depend on the composition. The coefficient of friction at a given temperature is a function of the viscosity, the thermal conductivity, the rate of shear, the area of shear, the capacity to dissolve different amounts of oxide from the surface of the material to be lubricated, and the contact angle between the glass and the material, because this determines the capacity of the glass to wet the material.

The importance of glasses used as lubricants is specially to be seen in metal-forming operations with operating temperatures up to approx. 1500 °C.

### 17.1.2.2 Substances with Lubricating Properties that Need a Supporting Medium

These can be classified as inorganic compounds (Table 17.4) and metal powders.

#### Inorganic Compounds

**Tab. 17.4** Inorganic compounds needing a supporting medium.

| | |
|---|---|
| Metal sulfides | $ZnS$, $SnS_2$, $FeS$, ... |
| Metal fluorides | $CaF_2$, $LiF$, ... |
| Metal phosphates | $Zn_2P_2O_7$, $Ca_3(PO_4)_2$, $Fe_2P_2O_7$, ... |
| Metal hydroxides | $Ca(OH)_2$, $Mg(OH)_2$, $Zn(OH)_2$, ... |
| Metal oxides | $PbO$, $ZnO$, $FeO$, $Fe_2O_3$, ... |

Natural oxide films on metals, which are usually approx. 100 Å thick, have been investigated by Whitehead with regard to their influence on the coefficient of friction. He found that their action depends primarily on the relative mechanical properties of metal and oxide. It is generally accepted that the oxide film reduces surface damage, makes sliding smooth, and often reduces friction.

Sulfides, fluorides, phosphates and hydroxides are claimed to act as a supporting agent or a catalyst by producing friction- and wear-reducing layers. Calcium hydroxide, for example, supports the production of a layer of $Fe_3O_4$ iron oxide on the rubbing surface of steel. This oxide has better tribological properties than the more common $\alpha$-$Fe_2O_3$ ferric oxide, possibly because of its more favorable close-packed cubic lattice structure compared with the corundum-like lattice structure of ferric oxide.

The process of formation of these layers depends on the chemical composition of steel and, in particular, on its surface chemistry.

Phosphate layers can also be applied by galvanic techniques. Such procedures are mainly used to create phosphate layers as precoatings for dry-film application and as lubricant carriers in cold metal-forming processes. In addition to acting as a lubricant carrier, the phosphate coating can be plastically deformed with the steel slug and, therefore, in conjunction with the lubricant, prevents metal–metal contact and thus reduces surface friction and wear.

The three main types of phosphating solutions contain zinc, iron, and manganese phosphates, and of these the zinc phosphate is probably the most widely used.

#### Metal Powders

In contrast with structural lubricants and self-lubricating mechanical lubricants, the lubricating properties of the other mechanical lubricants are mainly based on the supporting effect of a carrier substance or a binder. The main purpose of these substances, which include Pb, Sn, Zn, Cu, Ag, and In, is to improve the adhesion and cohesion properties of the non-self-lubricating mechanical lubricants.

#### 17.1.2.3 Substances with Indirect Lubricating Properties Based on their Hardness (Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD), and Diamond-like Carbon (DLC) layers)

Vapor-deposited (VD) coatings of oxides, borides, nitrides, or silicates, for example TiN, TiNC, CrN, ZrN, and AlTiN, usually have quite high coefficients of friction but can prevent seizure even at high temperature and offer excellent wear resistance.

Another relatively new form of thin-film layer with proven good tribological properties is diamond-like amorphous carbon, referred to as DLC. Hardness values for DLC range from 500 to 13 000 HV depending on the amount of hydrogenation, from 50 % to zero. Hydrogenated DLC is shown as (a-C:H) and hydrogen-free DLC as (a-C). In addition to hard-coatings there are also soft physical vapor-deposition coatings containing $MoS_2$ or $MoS_2$–metal composites. These films, normally deposited by various sputtering technologies like IBAD (ion-beam-assisted deposition) or CFMS (closed-field-magnetron sputtering), afford excellent tribological properties in terms of wear-resistance and low coefficient of friction. They are used for cutting and forming tools, ball bearings, and sliding contacts with steel and ceramics.

### 17.1.3
### Class 3: Soaps

Soaps are the metal salts of the higher saturated and unsaturated fatty acids and of resin acids and they are sometimes understood to include salts of naphthenic acids and synthetic ‘fatty’ acids. The most effective of these are polar compounds with active groups in a long-chain molecule, presumably because the reactive group attaches itself to the surface being lubricated and resists removal. They often give the lowest coefficients of friction obtainable with solid lubricants but in general cannot be used above their melting points or at high loads.

The main function of soaps in lubrication technology is in the preparation of greases. The subject is discussed in more detail in Chapter 6. The main use of soaps as lubricants in their own right depends on their formation in situ on a metal surface, by the chemical attack of a fatty acid on the metal.

### 17.1.4
### Class 4: Chemically Active Lubricants

This category includes Extreme Pressure (EP) and Anti Wear (AW) additives and a variety of chemicals which interact with the metal surface to produce a lubricating or protecting layer (see Chapter 6).

## 17.2
## Characteristics

The properties of a solid which might determine its value as a solid lubricant can be different if we are considering the solid only, or its use as a lubricant in the dispersed phase. The following discussion of the characteristics of a solid is limited to the solid only.

### 17.2.1
### The Crystal Structures of Lamellar Solid Lubricants

#### 17.2.1.1 Graphite

The ideal structure of graphite was proposed by Bernal in 1924 as a structure of plane hexagons with uniform side lengths stacked in such a way that the vacant centers in any sheet are always overlaid by the atoms of the next sheet.

Graphite has a resonance structure which enables binding of the carbon atoms as firmly with three bonds as with four. Each carbon atom is linked to its nearest neighbors by $sp^2$ type $\sigma$ bonds. The strength of the latter is increased above the normal single covalent C–C bond by the interaction of non-localized electrons associated with the 2pz orbital of the planar carbon atoms. It is this extra bonding which gives graphite its thermal stability; the $\pi$ electrons are responsible for the magnetic and electric properties of graphite. The planes themselves are held together largely by van der Waals forces which are much weaker than the planar bonding forces and the strength of bonding to a substrate.

The low friction of graphite is not based solely on its crystal structure. It also depends on adsorbed films, particularly of water vapor, which provide surfaces with low cohesion. Thus, graphite develops its most favorable lubricating properties in the presence of moisture.

#### 17.2.1.2 Molybdenum Disulfide

The crystal of molybdenum disulfide has a hexagonal structure with trigonal symmetry. Each Mo atom is surrounded by a trigonal prism of sulfur atoms. $MoS_2$ is chemically very stable. It is resistant to most acids and is insensitive to radioactive radiation. The temperature limit in air, above which there is increasingly rapid oxidation to molybdenum trioxide, is 370 °C. Moist air reduces the onset of oxidation to 350 °C. In vacuum it decomposes at 1100 °C to molybdenum and sulfur. When sufficiently pure, it is a semiconductor and diamagnetic.

The lubricating effect of $MoS_2$ is also based on its crystal structure. The weak bonds (van der Waals forces) between the sulfur layers enable easy movement of the lamellae over each other, resulting in a low friction between the sliding parts. On the other hand, the ionic bond between Mo and S gives the layers a high strength so that they can resist the penetration of surface asperities.

### 17.2.2
### Heat Stability

Heat stability is required if thermal decomposition of the solid lubricants is to be avoided, especially in the presence of oxygen-, carbon dioxide-, or water-containing atmospheres. Very often the decomposition products are either corrosive vapors or abrasive solids.

When heated in air, graphite forms carbon dioxide above 450 °C; this escapes without leaving any grinding particles behind. $MoS_2$ is attacked by oxygen even at 20 °C. In air, appreciable oxidation to molybdenum trioxide occurs above 400 °C and effects the lubricating properties and the adhesion of $MoS_2$ to metal surfaces. When oxidation can be excluded, $MoS_2$ retains its lubricating properties up to 800 °C. Lead monoxide is an excellent lubricant up to 700 °C. Metal-free phthalocyanines have been established as very efficient lubricants up to temperatures of 800–900 °C. Glass has proved to be an efficient lubricant at extreme loads and temperatures up to 1200 °C.

Other solids, for example metal oxides, sulfides or phosphates, also afford excellent heat stability. Without a supporting liquid, however, these solids provide no lubricating properties but build up separating layers only. The lubrication effect is, therefore, limited by the heat stability of the carrying substance.

### 17.2.3
### Melting Point

The melting point of a solid lubricant can be regarded as a physical aspect of heat stability. If, as a result of rising temperature, the elastic vibrations in a crystal become so violent that two component particles become separated by a distance greater than a given critical distance then the lattice yields at this point, because the stress which has forced it apart is capable of tearing the lattice open. Under this metastable condition the crystal melts and the cohesive energy of the crystal which gives it mechanical strength is destroyed.

### 17.2.4
### Thermal Conductivity

Frictional heat, developed by rubbing of material surfaces at the high spots, is also transmitted to the solid lubricant, which must be capable of dissipating this heat as quickly as possible – or local welding of the material might occur, despite the presence of the solid. This is particularly true of plastic solid lubricants, which all have poor thermal conductivity.

### 17.2.5
### Adsorbed Films

These can occasionally be very significant, and generalizations cannot be made about their mode of action. The adsorbed film of gas or vapor might be attached to the external surface of the solid or chemically to an internal surface (intercalation). The effect of the physically or chemically adsorbed molecules on the external surface of the solid can be:

- to influence the sintering of the aggregates of the particles under the action of heat;
- to influence the ease of breakdown of particles under shear stress; and
- to influence the wear properties of the edges of lamellar solids.

The chemisorbed molecules within the particles can affect:

- the decomposition or oxidation temperature of the solid;
- the mechanical properties of the solid; and
- the conductivity (thermal and electrical) of the solid.

### 17.2.6
### Mechanical Properties

The mechanical properties of a given solid are much less important than the mechanical properties of the solid relative to the metal it is to lubricate. Possibly the most important properties relate to hardness, compressibility, and tensile strength.

### 17.2.7
### Chemical Stability

Chemical stability is closely linked with heat stability. When solid lubricants are to be used intermittently over long periods in relatively inaccessible places, tendency to corrosion can be serious. Corrosion can occur in three ways:

- interaction of the solid with the metal surface;
- action of heat and/or the atmosphere on the solid to give decomposition products, which then react with the metal surface; and
- action of heat and/or the atmosphere on material chemisorbed on to the solid lubricant producing material which has a corrosive action on the metal.

### 17.2.8
### Purity

The importance of purity should never be overlooked. Contamination can arise from three sources:

- incomplete purification of the natural ore;
- by-products from synthesis; and
- products of oxidation during size reduction.

### 17.2.9
### Particle Size

Particle size is a geometrical term which does not take into account the internal structure of a particle or, indeed, very fine details of particle-surface topography. With this note of caution, it can be assumed that the smaller a non-porous particle, the more active it is. Although such particle activity is required in some applications there are other fields, for example many metal-forming operations, for which it is desirable to have a relatively large particle size range to give the required film properties. With graphite and molybdenum disulfide, relatively coarse particle sizes are advantageous because this results in optimum resistance to oxidation.

## 17.3
## Products Containing Solid Lubricants

### 17.3.1
### Powders

To ensure that solid lubricants in the form of powder give sufficient coverage in a tribological system, these lubricants must have the appropriate properties to enable them to create a film (cohesive properties) and adhere (adhesive properties).

Three requirements result from this:

(i) The level of adhesion between the lubricant film and the surface of the material must be great enough to ensure that this lubricant film adheres to this surface when it is subjected to friction.

(ii) The internal cohesion of the film must be sufficiently large that the film does not split when subjected to friction.

(iii) The adhesion between the particles and layers in the shearing direction should be as small as possible to keep the resistance to friction low.

These main requirements can be met only by self-lubricating dry lubricants. $MoS_2$, because of its crystalline structure (layer grid) and bonding type (covalent bonding of a metal (Mo) and strong polarizing effect of a non-metal (S) with a high degree of polarization), meets these requirements as well as can be achieved; it is, as a result, the most commonly used. Other solid lubricants which are applied in powder form are organic compounds such as PTFE and graphite, although these fulfil the requirements listed above to a limited extent only.

Before the solid lubricant powder can be applied, the surface of the material must be thoroughly cleaned. Roughing the surface mechanically or with phosphates improves adhesion and therefore the lifetime. The lubricant can be effectively applied by simple rubbing with cloths, sponges, brushes, or polishing pads or polishing buffs, by applying it using suitable carriers, and coating by cathode evaporation in an ultra-high vacuum.

#### 17.3.1.1 Solid Lubricants in Carrying Media

As already mentioned, very few solid lubricants have the appropriate adhesive and cohesive properties which enable them to create an effective lubricating layer with low friction coefficients and give them a sufficient lifetime.

Most substances used as solid lubricants require a carrying medium, a bonding agent and/or pretreatment of the material surface, to help create, or improve, their adhesive and film-creating properties. The substances used as bonding agents are:

- organic binding agents, resins;
- inorganic binding agents, silicates; and
- oils, greases, water...

It is also advantageous to pretreat the surfaces by:

- degreasing;
- sandblasting, corroding, etching;
- phosphating, anodizing; and
- activating (e.g. low-pressure plasma)

### 17.3.2
### Dispersions and Suspensions

Dispersions and suspensions in carrying liquids with low volatility are mainly used in areas in which, for tribological reasons, a dry lubricating film should be created, but where effective application of a powder is not possible for technical reasons. The same types of solid lubricant are used here in the same way as for powders.

Dispersions and suspensions of solid lubricants in water are usually used to coat mass elements for cold and hot forming. The most commonly used substances here are salts, special white solid lubricants, and graphite.

Dispersions and suspensions in oils also act as aids in forming techniques, and they are also used as additions in gear- and oil-lubricating systems. The solid lubricants used here in forming techniques are the same types as those used as dispersions and suspensions in water. $MoS_2$ plays a dominant role as an additive to lubricating gears and for general use in oil lubricating systems.

### 17.3.3
### Greases and Grease Pastes

Addition of solid lubricants to greases is primarily intended to have a positive effect on their capacity to absorb pressure, and the ability to withstand wear and tear, and friction. The specific advantages of solid lubricants in comparison with oil-soluble, chemically reactive additives is that they react neutrally to many types of plastic and elastomer, perform well at high temperatures and have good safety reserves with regard to emergency running properties, which come into action when the layer of grease collapses. The use

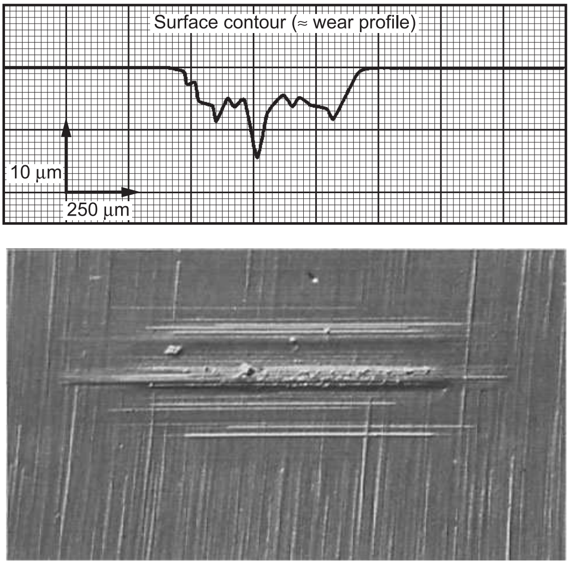

**Fig. 17.1** Lithium grease with molybdenum disulfide, showing the irregular pattern of the wear profile, the chipping of the material, and serious wear. The maximum wear depth exceeds 13 µm

of black solid lubricants such as graphite and $MoS_2$ also have a beneficial effect on running-in processes and the capacity to withstand wear and tear in boundary and mixed friction areas. Where oscillating movements or vibration is involved, white solid lubricants have the advantage. These effects are illustrated in Figs. 17.1 and 17.2.

The exceptional aging stability shown by solid lubricants is particularly important in terms of the lifetime of the lubricant, and can be used to advantage in the appropriate systems and structures. Generally speaking, solid lubricants in greases are

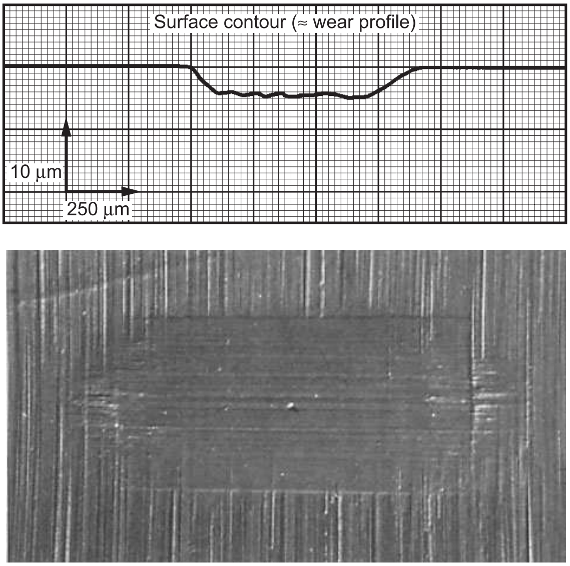

**Fig. 17.2** Lithium grease with white solid lubricants showing the smooth pattern of the wear profile. The maximum wear depth is only 5.9 µm, the wear pattern is uniform, and there is no chipping of material. Scanning electron photomicrographs (50 × magnification)

used at concentrations of 1–3 %. When this proportion rises to over 10 % they are known as grease pastes, because the solid lubricant at this concentration has a noticeable thickening effect.

High-performance lubricating greases do not usually contain one single lubricating element, but instead are made up of combinations of two or more substances, which have a synergistic effect. This applies both to the combination of graphite and $MoS_2$ and to mixtures of different inorganic solid lubricating substances, for example calcium hydroxide, zinc phosphate, or iron sulfide.

### 17.3.4
### Pastes

Pastes are solid lubricants in a carrying oil. To achieve the correct consistency, the proportion of solid lubricant must be at least 40 %. To reduce the undesirable 'bleeding' effect, modern formulations often include a small amount of soap, although tribologically this plays a minor role only.

Black pastes usually contain graphite or $MoS_2$ and combinations of both these types of solid lubricant. Because of their high load-bearing capacity, these pastes are particularly useful when movement with very slow relative speeds is involved, e.g., assembly processes, and running-in processes.

Pastes prepared from combinations of white solid lubricants are especially suitable for use where oscillating movements or vibration is involved. White pastes are particularly good at preventing the wear and tear which results from vibration and are proven to afford excellent protection against fretting corrosion.

These special white solid lubricants create thin adhesive layers on the surfaces which are subject to friction. These layers principally adhere to the surfaces because of tribochemical (e.g. zinc diphosphate) and/or physical adhesive forces (e.g. calcium hydroxide). Another special feature of these layers is that they regenerate themselves. This, and their innate high degree of resistance to wear and tear, means that longer operating periods can be achieved than when normal greases are used, without the need for additional re-lubrication.

Another area where pastes are traditionally used is for lubrication of screw connections. Special screws made of steels which are resistant to high temperatures, based on chrome/nickel alloys, are prone to seizure because an oxide layer is not created.

The pastes used in this particular application area are usually made up of combinations of solid lubricants and special metal powders. More recent developments show that the use of formulations which do not contain metal, and are based on white solid lubricants, perform well when used at high temperatures.

### 17.3.5
### Dry-Film Lubricants

Lubricating varnishes or dry-film lubricants are suspensions of solid lubricants and other additives in a solution of inorganic or organic binding agents. The most commonly used solid lubricants are $MoS_2$, graphite and PTFE. Each of these solid lubricants has characteristic properties, which determine where it can be used. The most important of the additives are pigments which protect against corrosion. The main types of binding agent used are organic resins (acrylic, phenol, epoxy, silicone, urethane, imide), cellulose, and inorganic silicates and phosphates. Hydrocarbons or water are used as solvents. Dry-film lubricants can be used in a variety of ways; these depend primarily on the number, shape, and/or particular requirements with regard to partial coating. They are applied by dipping centrifuges and drums and by various types of spraying procedure. The hardening process depends on the type of binding system and happens at ambient temperature or in an enameling stove. After they have hardened lubricating varnishes form a highly adhesive, dry film of lubricant. The proportion of solid lubricants in the layer created in this way can be as high as 70 %.

Solid lubricants with a layer structure ($MoS_2$) have a floating effect in the wet film, whereby the layers arrange themselves horizontally as the film dries and settle on top of each other in individual layers. This is how a separating layer is created between the base unit and the opposing unit of the tribological system. In an ideal situation this layer is between 10 and 15 μm thick. When placed under pressure, the texture of this layer becomes compressed and creates an extremely smooth, shiny film surface (Fig. 17.3). PTFE lubricating varnishes are usually applied in much thinner layers, e.g. 3 to 5 μm thick. Special PTFE coatings for lifetime lubrication are applied in layers up to 15–30 μm, however.

The effectiveness of the lubrication, lifetime, and reduction in wear and tear of dry-film lubricants is not constant, but is influenced by a variety of different factors (Figs 17.4–17.9). The performance of dry-film lubricants depends on:

(i) their composition, in particular the type of binding agent, the ratio of solid lubricants to the binding agent, the types of solid lubricants, and the film thickness;

(ii) the properties of the substrate to which the lubricating varnish is applied, in particular the cleanliness, roughness (porosity; Fig. 17.4), and hardness of the surface; and

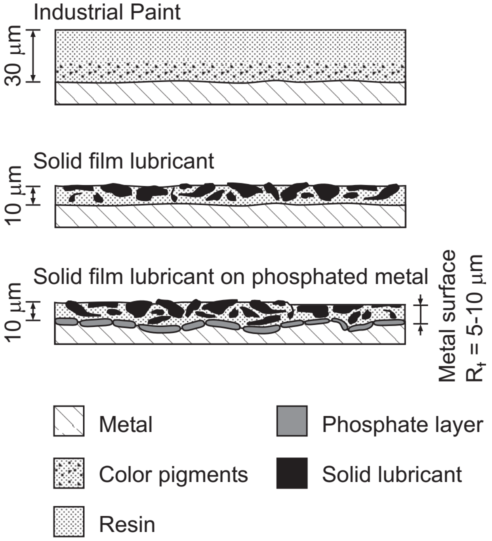

**Fig. 17.3** The composition of solid-film lubricants

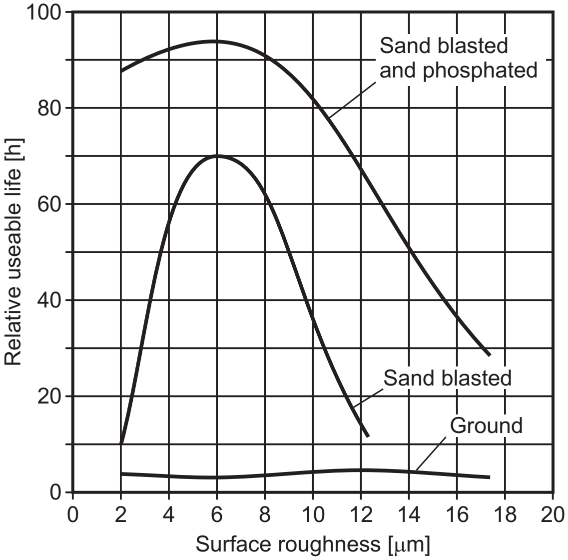

**Fig. 17.4** Relative lifetime of $MoS_2$ solid-film lubricants as a function of surface roughness

(iii) environmental (local) conditions, in particular the demands, and types of demand, placed on the lubricant (e.g. load, Figs. 17.5 and 17.8), the type and speed of movement (Figs. 17.6 and 17.7), the temperature (Fig. 17.9), atmospheric pollution (humidity, dust, radiation), atmospheric pressure, and the surrounding media (gases, liquids).

The pretreatment of the substrate surface plays a particularly important role in the successful use of lubricating varnishes – as already mentioned, the roughness of

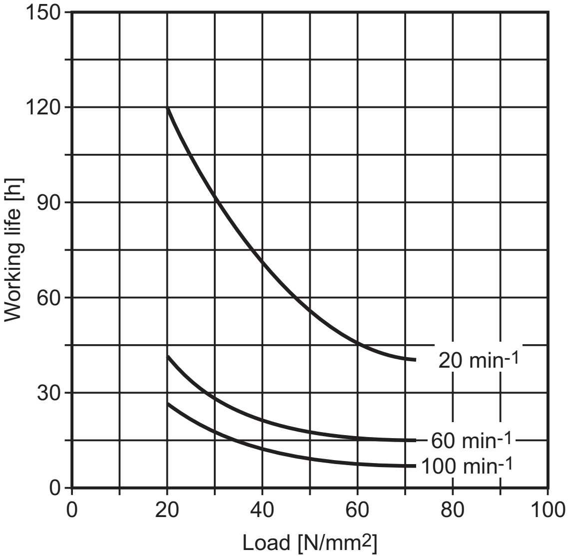

**Fig. 17.5** Dependence of lifetime on load ($MoS_2$, phenolic resin solid-film lubricant)

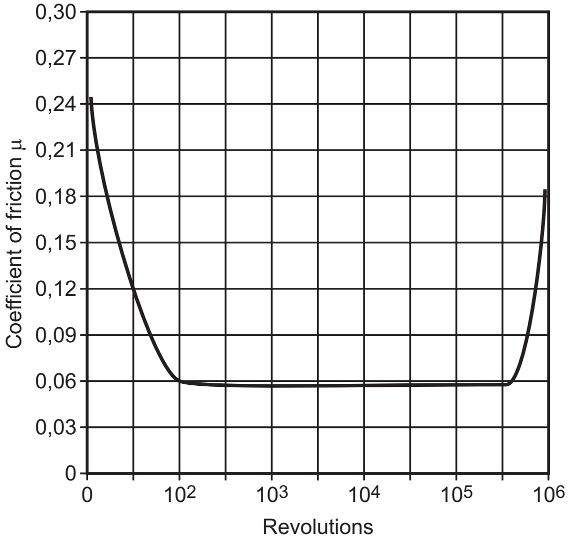

**Fig. 17.6** Working life and typical progression of the coefficient of friction of $MoS_2$ solid-film lubricants (LFW-1 testing machine)

the surface and the way in which the crystallites are arranged have a considerable impact on their lifetime. Various pretreatment procedures can be used to achieve the best possible surface. Sandblasting, phosphating, anodizing, etching, or corroding are the most suitable for metallic substrates. Plastic and elastomer surfaces can be pretreated by chemical and mechanical processes and by energetic activation procedures (e.g. plasma treatment or corona treatment).

Dry-film lubricants can be used in a multiplicity of ways.

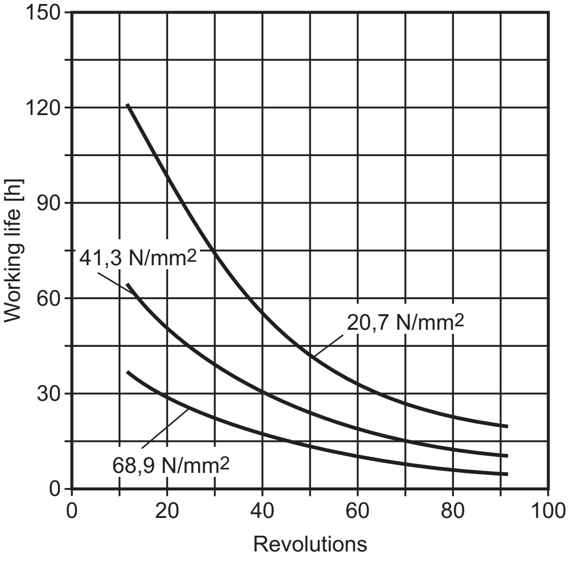

**Fig. 17.7** Dependence of lifetime on the number of revolutions ($MoS_2$ phenolic resin solid-film lubricant)

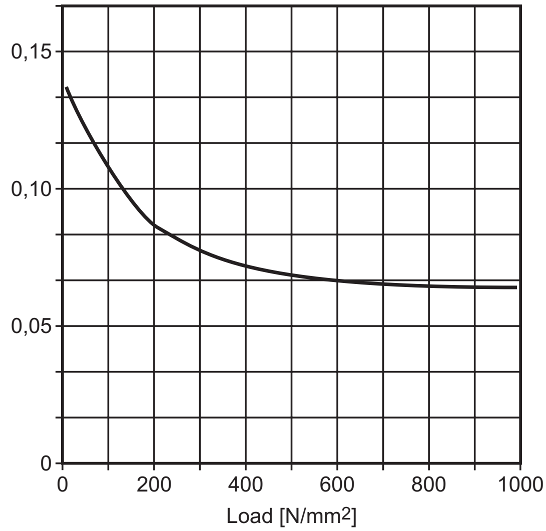

**Fig. 17.8** Dependence on load, at room temperature, of the coefficient of friction of $MoS_2$ solid-film lubricants

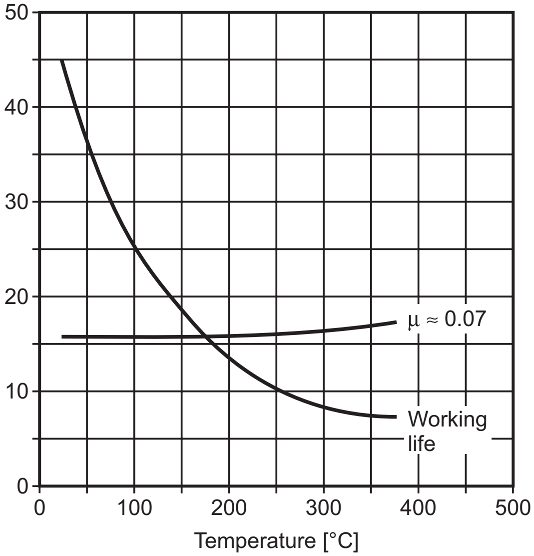

**Fig. 17.9** Influence of temperature on the working life of an $MoS_2$ solid-film lubricant (lubricating speed 8 m $min^{-1}$, load 3000 N)

## 17.4
## Industrial Uses of Products Containing Solid Lubricants

Because of the continuously increasing demands put on modern lubricating systems, almost all formulations contain a certain proportion of solid lubricants. Significant fields of application are the lubrication of machine elements and use in process auxiliaries for various production technologies such as, for example, the production of glass or metalworking.

Solid lubricants play a significant role in metalworking, especially in forming. Their main task is to generate an effective separating layer between the tool and the surface of the workpiece and thus act against the adhesion forces between the materials. Solid lubricants, in combination with their carrier medium and other additives, also reduce friction or generate a defined coefficient of friction, and keep the process temperature constant. Through their wear protection function they increase the service life of tools, protect against corrosion, ensure a stipulated surface quality and the accuracy to gage of the workpieces. The choice of a suitable solid lubricant for metalworking depends mainly on the machining operation, the process parameters, and the pair of materials involved.

In chip-forming metalworking, cutting-active additives such as fatty acids, esters, metal soaps and additives containing chlorine, phosphorous, or sulfur are used. Under the influence of pressure and temperature the surfactants form reaction products such as metal chlorides, phosphides or sulfides with the metal surface; these reduce the coefficient of friction and protect against wear.

The main field of activities of ‘classic’ solid lubricants, for example graphite, molybdenum disulfide, waxes, soaps, salts or even soft metals, lie more in the area of non-cutting forming and, especially cold and hot forging, pressure die-casting and drawing, and cold extrusion operations. Internal high-pressure forming was added as a new area of application for solid lubricants in the nineties.

Solid lubricants are available generally as powder or pigment embedded in a resin or wax film or as dispersions in oil or water. The variety of industrial uses of solid lubricants can be made more clear by studying the lubrication of different machine elements.

### 17.4.1

### Screw Lubrication

The machine element most used is the screw. Screws serve to effect non-destructive releasable connections between two or more parts. The selection of the most suitable screw lubricant depends upon the material or the surface of the screw and the thread partner, and on the conditions of use, for example temperature, humidity, and the influence of chemicals, radiation, and the constructive requirement according to a stipulated coefficient of friction or minimum pretensioning force, even after the screws have been tightened or loosened many times.

For this reason, special high-temperature pastes are used for the lubrication of screw connections at high working temperatures. These pastes comprise a basic oil which is mixed with different solid lubricants and can contain additional additives. In formulation or selection care must be taken to ensure that the components do not contain sulfur, zinc, lead, and aluminum, which can lead to damaging changes to the screw material, e.g. stress crack corrosion at high temperatures. They should also be free of health-hazardous nickel.

Screws for high-temperature applications are made of high heat-resistant generally austenitic materials with alloy components such as nickel, chromium, or molybdenum, which enable only very thin oxide layers to develop on the surface of the thread flanks.

High coefficients of friction and a pronounced tendency to adhesion are the result. By developing a pressure-resistant separating film, modern high temperature pastes prevent welding of the thread flanks and lead to defined coefficients of friction and insulate against the penetration of oxidizing gases; in this way they ensure the non-destructive release of the screw connection. Apart from these properties, which must also be fulfilled by some lubricants at lower application temperatures, there are special formulations for avoiding tribocorrosion and fit rust. Particularly effective here are greases and pastes combined with white solid lubricant prepared from metal hydroxides, phosphates, and sulfides.

For many screw lubrication applications, special dry-film lubricants are also available as an alternative to greases and pastes. The advantage of lubricating varnish is its simple application in immersion centrifuges or spray drums, both time-tested procedures for coating mass-produced parts. The handling of coated screws is clean and suitable for robot systems.

Special areas of application for lubricating varnish and slide films are metric screws, thread-cutting screws, thread-shaping screws, thread-grooving screws, all kinds of wood screws, screws made of plastic, and screws made of aluminum alloys, magnesium alloys, and titanium alloys, for example.

### 17.4.2
### Roller-Bearing Lubrication

Depending upon the application, the roller bearings are lubricated with oils, greases, pastes, or lubricating varnishes; of these grease lubrication is the most common. Many special greases, with or without solid lubricant additives, enable customization for almost every application. Important considerations include:

- temperature range,
- speed range (rotational speed, frequency),
- maximum specific surface pressure in roller contact, and
- ambient media (chemicals, humidity, etc.)

Greases or pastes containing solid lubricants must always be used when reliable separation of the roller body and bearing ring at low speeds and high loads cannot be ensured by grease alone. Over and above this, greases or pastes containing solid lubricants have good emergency running properties and afford high safety under shock and impact stress. Greases or pastes with white solid lubricants are preferable for use in roller bearings employed in oscillating operations. Lubricating varnishes are the best solution for applications in a vacuum, for example, in air and space travel, nuclear engineering, extreme temperatures, or for microsetting movements.

Special constructions also enable solid lubricants to be incorporated in the bearing material; this enables the production of maintenance-free bearings.

### 17.4.3
### Slide Bearing, Slide Guideway, and Slide Surface Lubrication

With solid lubricants, the same principles apply for slide-bearing lubrication as for roller-bearing lubrication, except that oil lubrication now plays a considerably greater role. To enable correct selection of a suitable lubricant, the construction data of the friction bearing must be compared with the lubricant key values to furnish a performance profile.

Reliable separation of the material partners must be ensured at all times to comply with the primary demand put on the lubricant for reliable load transfer at lowest wear rates. Whereas, as a general rule, the operating point of a friction bearing is in the region of hydrodynamic lubrication and therefore the viscosity of the lubricant is the most important key value, slide guideways and other slide surfaces (for example, infeed wedges) are mainly in the boundary or mixed friction range. The best solution for these operational requirements is lubrication with PTFE belts and slide liners based on epoxy resin with the incorporation of solid lubricants such as $MoS_2$, graphite, or polyurethane. Pastes or lubricating varnishes containing solid lubricants are likewise suitable and offer themselves for the running-in phase and the warm-up and cool-down phases in normal operation. In this way the boundary and mixed friction zone can be safely worked through without wear occurring, even with pure oil lubrication, and emergency running properties can be achieved.

### 17.4.4
### Chain Lubrication

The development of a hydrodynamic lubricating film is not possible because of the high surface pressure and low sliding speed between the different elements of a chain link. With the given mixed friction, it is only possible to limit wear and achieve an acceptable service life for the machine elements by using solid lubricants.

When selecting a suitable lubricant special consideration must be given not only to the different chain-drive construction types, but also to the material pairs, the operating parameters, and the ambient conditions. For solid, highly viscous lubrication care must be taken to ensure adequate creep or penetration to get to the narrow gaps, for example, between the bolt and bush. The lubricant must also have an appropriate adhesion capacity so that it is not spun off at high chain speeds or high centrifugal forces at the turning point (pinion and wheel).

### 17.4.5
### Plastic and Elastomer Lubrication

In plastic and elastomer lubrication the material compatibility plays a particularly important role. Swelling, shrinking, or embrittlement are the most serious changes which elastomers can experience when they come into contact with lubricants. Solid lubricants have limited influence on elasticity module, tensile strength, and hardness. In addition to reducing friction and protecting against wear during the assembly process, the separating effect of the lubricant is also important. Examples in elastomer lubrication are o-rings and seals in automotive applications. Most requirement profiles can be covered adequately by lubricating varnishes and films containing solid lubricants.

In plastic lubrication the special features of substances, i.e. interaction between the plastic and the lubricant, must also be taken into consideration. Particular attention must be paid to the tendency to develop stress cracking. Because of the multitude of different types of plastic available, direct compatibility tests are recommended on the plastics to be employed. As with elastomer lubrication, the influence of solid lubricants in compatibility testing is of limited significance. Oils, greases and pastes containing solid lubricants, and also lubricating varnishes and slide films, can be used successfully for lubricating plastic and steel, and plastic–plastic pairs. The running-in process must also be given enough attention in the use of plastic materials, and as a general rule, can be positively influenced by solid lubricants.

# 18
# Laboratory Methods for Testing Lubricants

*Siegfried Noll and Roman Müller*

## 18.1
## Introduction

The properties of lubricants are usually determined by use of standardized test methods. The test results obtained from these methods enable us to characterize the physical, chemical, and tribological attributes of lubricants and enable assessment of their suitability for specific applications. Industrial lubricant users define specifications which are derived from the demands of applications.

Laboratory testing is an indispensable part of the research and development of lubricants. In most industrial applications, moreover, it is necessary to monitor the actual condition of a lubricant in use, with the objective of prolonging its lifetime and sustaining the proper functionality of machines and lubricating systems.

Lubricants must be monitored carefully and, often, be maintained by treatment with appropriate chemical substances.

For chemists and engineers, laboratory test results are guidelines enabling measurement of properties most appropriate both for R&D and application requirements.

The most common laboratory test methods are listed in the following sections and an overview of the corresponding international standards is appended e.g. AFNOR [18.1], ASTM [18.2], DIN [18.3], IP [18.4], and ISO [18.5].

## 18.2
## Density

The density (DIN 51757/ASTM D 1298) of a substance is defined as the quotient of the mass and the volume. For the determination of density of lubricants different test equipment is in use such as hydrometer, pycnometer, hydrostatic balance or digital densimeter. The density in combination with the viscosity, refractive index and sulfur is a parameter for the characterization of the structure of petroleum hydrocarbons. Naphthenic oils have in general a higher density than mixed or paraffinic oils.

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## 18.3
## Viscosity

The viscosity is one of the most important properties for characterization of lubricants and their flow and transport properties. The absolute values of the viscosity depend on temperature, pressure and in some cases on the shear rate which depends on the structure of the lubricant. There is a difference between the absolute (dynamic) and kinematic viscosity. The kinematic viscosity is a measure of the relative flow of a fluid under influence of gravity. The absolute viscosity or dynamic viscosity is the product of kinematic viscosity and density of the lubricant. The SI unit is the kinematic viscosity in $m^2 s^{-1}$ whereas the SI unit for the dynamic viscosity is mPa · s (milli Pascal second).

For the determination of viscosity capillary viscometers, falling ball and rotary viscometers are used.

### 18.3.1
### Capillary Viscometers

This method with capillary viscometers (DIN 51562/ASTM D 445) is generally used for most of the automotive and industrial lubricants for determination of the kinematic viscosity. The measurement is performed with a calibrated glass capillary viscometer where the fluid passes through a fixed diameter orifice under the influence of gravity. The most frequently used viscometers are Ubbelohde for transparent liquid. For the measurement of opaque or dark oils Cannon Fenske viscometers (DIN 51366) are used.

### 18.3.2
### Rotary Viscometers

The rotary viscometers use the torque on a rotating shaft to measure a fluid's resistance to flow. The shear rates can be changed by modifying the speed of rotation, rotor dimensions and the gaps between rotor and stator. Equipment like the cold cranking simulator (CCS), mini rotary viscometer (MRV), Brookfield viscometer and high temperature high shear Ravenfield viscometer method are all rotary viscometers.

The cold cranking simulator (CCS) apparent viscosity is measured in a range between 2.000 and 20 000 mPa · s at temperatures between –10 and –35 °C (ASTM D 2602/DIN 51377). The CCS apparent viscosity correlates excellently with low temperature engine cranking.

The mini rotary viscometer (ASTM D 3829) is applied to measure the borderline pumping temperature and is related to the mechanism of pumpability of engine oils.

The Brookfield viscometer (ASTM D 2983/DIN 51398) is used for the determination of the low shear rate viscosity of automotive fluid lubricants in a temperature range between –12 °C to –55 °C.

The Ravenfield viscometer (ASTM D 4741) allows the determination of dynamic viscosity of engine oils at 150 °C using a high shear rate Tapered/plaque viscometer.

## 18.4
## Refractive Index

The refractive index (DIN 51423/ASTM D 1218) is used for the characterization of the structure of petroleum hydrocarbons like the density. Furthermore, it is mostly used as a property for fast identification of a product. The refractive index is the ratio of the velocity of light of specified wavelengths in air to its velocity in the substance under examination.

## 18.5
## Structural Analyses

There are various methods available for the characterization of non additivated mineral oils. The information provided with these different methods cannot be compared directly. Therefore, the method used must be stated.

The amount (%) of aromatic (CA) and paraffinic ($C_P$) carbon in the mineral oil can be determined by the carbon distribution method developed by Brandes [18.6]. The method determines the infrared absorbency at wavelengths of 1620 and 724 $cm^{-1}$, which correspond to the stretching frequencies of the aromatic and paraffinic hydrocarbons respectively. However, this method gives no information on isoparaffins and does not take into consideration sulfur content.

Using the method according to DIN 51378/ASTM D 2140 the kinematic viscosity at 40 °C, the density at 20 °C and the refractive index as well as the sulfur content are determined. Out of these characteristics the viscosity–density constant and the refractive intercept are calculated. By use of a nomogram or by calculation software the percentage of aromatic (CA), naphthenic (CN), and paraffinic (CP) carbon is determined, with or without sulfur correction.

For improved characterization chromatographic, gas chromatographic and NMR spectroscopy methods as well as element analyses of the narrow distillate fractions have to be carried out.

## 18.6
## Flash Point

For the determination of the flash point (DIN ISO 2592/ASTM D 92) a defined sample quantity is heated at a defined heating rate. The flash point is reported as the lowest temperature at which the application of defined test flames causes the vapors above the surface to ignite.

In accordance with DIN ISO 2592/ASTM D 92 flash points of 79 °C and above are determined by use of the Cleveland open cup.

The Pensky–Martens closed cup method (DIN 51758/ASTM D 93) is used when flash points in the range 40 to 360 °C are expected, although precision has not been determined above 250 °C.

To determine flash points below 40 °C the Abel–Pensky (DIN 51755) closed-cup method is used.

Besides its use for characterization of base fluids and lubricants, the flash point is a very important characteristic for classification of products according to the international transport regulations.

## 18.7
## Surface Phenomena

During storage or the application of lubricants there is a continuous contact with air or even with water, which might lead to formation of foam or emulsion. For evaluation of the behavior of base oils and lubricants the following methods are applied.

### 18.7.1
### Air Release

For the determination of air-release behavior (DIN 51381/ASTM D 3427) compressed air is blown through the test oil which is heated to a defined temperature. After 7 min, blowing is discontinued and the air entrainment is determined. This is the time at which the density is narrowed to 0.002 g ml$^{-1}$ of the initial density of the lubricant.

### 18.7.2
### Water Separation and Demulsibility

Water separation is crucially affected by the demulsibility characteristics of a lubricant.

For determination of demulsibility in accordance with DIN ISO 6614 / ASTM D 1401, 40-mL volumes of both lubricant and distilled water are stirred at a defined temperature and stirring rate in a graduated cylinder. The test temperature selected depends on the kinematic viscosity of the lubricant. Lubricants in the viscosity range 28 to 90 mm$^2$ s$^{-1}$ are tested at 54 °C. More viscous lubricants are determined at 82 °C. After stirring the time required for the separation of oil from the water is determined at 5-min intervals. If after 30 minutes standing the separation is not totally completed the volume of oil, water and emulsion is reported.

The demulsibility of lubricants that are exposed to water under highly dynamic conditions is determined in accordance with ASTM D 2711.

Lubricant (405 mL) and distilled water (45 mL) are mixed at 4500 rpm, for 5 min, at 82 °C, in a graduated cylinder, by means of a multi-propeller stirrer.

After settling for 5 h, the separated water ("free water") is drained off and its volume is recorded.

The remaining oil and emulsion layer is centrifuged and the amount of separated water is determined. The test result is reported as milliliters free water and milliliters water after centrifuging.

### 18.7.3
### Foaming Characteristics

The foaming characteristics (ASTM D 892) are determined by blowing for five minutes a constant rate of air into 190 mL lubricant. The volume of foam generated is measured immediately and after settling for ten minutes. The test temperature in Section I is defined at 24 °C. Section II requires a second sample which is similarly treated at 93.5 °C. Finally, Section III is run with the same sample after settling of the foam and cooling to 24 °C.

For testing of some engine oils the method is extended by Section IV which requires a test temperature of 150 °C.

## 18.8
## Cloud Point, Pour Point

The pour point (ASTM D 97) is the lowest temperature expressed as a multiple of 3 °C at which the test fluid is observed to flow when it is cooled and examined under defined conditions. The cloud point (ASTM D 2500) is the low temperature behavior where an oil starts to become cloudy when it is cooled under defined conditions.

## 18.9
## Aniline Point

The aniline point (DIN 51775/ASTM D 611) is the temperature at which equal volumes of hydrocarbon oils and aniline separate in two phases. This value gives an indication on the structure of mineral oils. The higher the temperature of separation the more paraffinic is the oil. It also gives an indication of the solvency behavior of the oil – the lower the aniline point the better the solvency for most types of additive.

## 18.10
## Water Content

There are in general two standard methods for the determination of water content. For volumes of 0.05 % and above ASTM D 95 is used. This test procedure involves azeotropic reflux distillation of the oil with xylene with water separating from the reflux distillate.

For lower water contents between 50 and 1000 ppm the Karl Fischer direct titration method (DIN 51777/Part 1/ASTM D 1744) is more accurate. The sample is titrated with a standard solution of iodine, pyridine and sulfur dioxide in methyl alcohol. The end point of the titration at which free iodine is hydrated may be registered either potentiometrically or by color indicator. Because the sulfur dioxide from the Karl Fischer titration solution can react with different alkaline components of the test sample, incorrect, i.e. apparently higher, concentrations of water might be determined. DIN 51777 Part 2 (>3 ppm water)/ASTM D 6304 (10 to 25 000 ppm water) describe the indirect Karl Fischer method. The sample is heated in a capsule of reagent-type glass to approximately 120 °C while dry nitrogen gas is blown into the fluid to transfer the evaporated water into the titration vessel. This treatment, which is performed in a completely closed system, prevents chemical side-reactions and thus reflects the actual water content.

## 18.11
## Ash Content

For unused oils the determination of the ash content (DIN EN 7/ASTM D 482) gives an indication of purity. The ash can result from water soluble metallic compounds and from solids such as dust and rust which have reached the oil during production, storage or transport. In used oils wear metals and contamination also give rise to higher ash contents.

Another method is the determination of sulfated ash from unused lubricating oils containing additives. Those additive concentrates contain a variety of metals. As this is a time consuming method it has more or less been replaced by determination of elements by atomic absorption, X-ray fluorescence or emission spectroscopy.

## 18.12
## Acidity, Alkalinity

The neutralization number (ASTM D 974) covers the determination of acidic or basic constituents in petroleum products by titration against color indicator. However, when the oil contains additives such as dispersants or detergents difficulties are accounted when observing the end point of titration and, therefore, potentiometric titration is used.

The total acid number (TAN acc. ASTM D 664) is a measure of the acidic and basic constituents in petroleum products. The acidity of unused oils and fluids is normally derived from the type and concentration of specific additive material whereas the acidity of used oil is of interest to measure the degree of oxidation of the fluid.

The total base number (TBN) characterizes the alkaline reserve in petroleum products. It is particularly used for engine oils where by acidic combustion products use up the alkaline reserve.

## 18.13
## Aging Tests

The aging of lubricants is a function of temperature or other parameters. These aging tests are carried out on base oils as well as fully formulated products to test the efficiency of additives. The different oxidation stability tests are the most common ways for testing the aging properties of lubricants. There is a big variety of standardized test methods where we have duration of test between hours and months. The majority of these tests is based on exposing the test fluids to oxygen or air at relatively high temperatures in presence of catalyst metals to increase oxidation rates and to reduce the testing period. Oxidation stability is assessed by quantitative determination of oxidation products, oxygen absorption, viscosity changes, change in acidity or formation of sludge. This chapter describes the most common test.

The TOST (turbine oxidation stability test, ASTM D 943) is used to evaluate the oxidation stability of inhibited hydraulic, turbine and R&O oils in the presence of oxygen, water, copper and iron catalyst at 95 °C. The acid number is determined periodically and the test is terminated when the TAN has reached 2.0 mg KOH $g^{-1}$. The test result is reported as the number of hours to reach an acid number of 2.0 mg KOH $g^{-1}$.

The rotary pressure vessel oxidation test (RPVOT), formerly RBOT, is used to determine the oxidation stability of hydraulic, gear, R&O, turbine, and transformer oils in the presence of water, oxygen, and copper as a metal catalyst. The rotary vessel is pressurized to 620 kPa with oxygen at ambient temperature and the vessel is rotated at an angle of 30° to the horizontal in a heating bath at 150 °C. As the temperature inside the vessel increases, the pressure increases to approximately 1300 kPa. As oxidation occurs, the volume of oxygen, and thus the pressure inside the vessel, decreases. When the pressure loss reaches 166 kPa the time in minutes is reported as the test termination criterion. The time in minutes is commonly described as the induction period.

The aging test according to Baader (DIN 51554) is an oxidation test using atmospheric air and intermittent immersion of a copper spiral at a test temperature of 95 °C. After a given time the saponification number in mg KOH $g^{-1}$ is measured.

## 18.14
## Hydrolytic Stability

Certain types of base fluids and additives have a tendency to react with water. A common method is the beverage bottle method, (coke bottle test) (ASTM D 2619) where beside the copper corrosion in presence of water the hydrolytic stability of additive systems is measured. For this test a 75 g sample of fluid plus 25 g water and a copper test specimen are sealed in a pressure type beverage bottle. The bottle rotates end to end for 48 h in an oven at 93 °C. After the test period the phases are separated and insoluble matter is weighed as well as the weight change of the copper specimen is measured; the acidity and viscosity changes of the fluid and the acidity of aqueous phase are determined. The method differentiates the relative stability of fluids in the presence of water under the conditions of this test.

## 18.15
## Corrosion Tests

The oxidation and corrosion reactions cannot be separated and quite often the metallic catalysts used are checked for corrosion, staining and deposits after the tests. For all oxidation tests the type of test specimen and the way of preparation is of importance. This chapter describes the main common methods.

The detection of copper corrosion (ASTM D 130/DIN 51759, Part 1) describes a simple procedure for investigating the aggressiveness of the fluid towards a polished copper strip at a temperature for a time selected for the fluid under test. The typical test parameters for fluids are 1 to 24 h at ambient temperature up to 150 °C. At the end of the test period the copper strip is removed, washed and compared with the ASTM copper strip corrosion standards. Ratings from 1 to 4 denote slight tarnish (1), moderate tarnish (2), dark tarnish (3) and severe corrosion, for example black tarnish (4).

The rust preventing characteristics of inhibited mineral oils (ASTM D 665/DIN 51585) in the presence of water is used for evaluating corrosion preventive properties of turbine oils and hydraulic oils. A mixture of 300 ml oil is stirred with 30 ml distilled water (Part A) or synthetic sea water (Part B) at 60 °C. A freshly polished cylindrical steel test rod is completely immersed therein and after 24 h the test rod is observed for signs of rusting and the degree of rusting is determined.

For the evaluation of rust preventive properties of metal corrosion preventives under high humidity (ASTM D 1748/DIN 51359), steel panels are prepared to a prescribed service finish and dipped in a test oil. After draining the panels they are placed in a humidity cabinet at 50 °C and the hours are recorded until rust occurs on the surface. Corrosion preventatives applied to protect metal parts against a sea water environment, e.g. in coastal regions or during ocean shipping, are usually tested in the so-called salt-spray test.

ASTM B 117/DIN 50 021 describes test procedures in which defined metal sheets protected with corrosion preventatives are exposed to a spray of 3 % sodium chloride solution at 35 °C.

The coated metal sheets are stored in a rack in a closed cabinet and at the beginning of each cumulative test hour the test solution is sprayed for 5 min within the cabinet at a defined volume per hour rate.

The test is terminated when three rust spots of 1 mm diameter in the largest dimension have appeared on the panels within an area limited to a 1 cm distance from each of the four edges of the test metal sheets.

The result is reported in hours until the described corrosion occurred.

## 18.16
## Oil Compatibility of Seals and Insulating Materials

An essential requirement for lubricants is that they should be compatible with seal materials as they are frequently in prolonged contact at elevated temperatures. In all the test methods applied, well defined test specimens are immersed in the lubricant for a defined time at defined temperatures. After the test period the volume change, the weight loss and the change of mechanical properties are evaluated.

## 18.17
## Evaporation Loss

The volatility of oils is determined by evaporation loss expressed as a percentage by mass of non additive and additive type lubricating oils (ASTM D 5800/DIN 51581, Part 1). The procedure according to Noack has been developed for motor oils but it can also be applied to other lubricants. A well defined air stream generated by a constant partial vacuum is directed over the oil which is heated for one hour to 250 °C. The evaporation loss is established by weighting. The evaporation loss can also be determined by use of a gas chromatographic procedure according DIN 51 581, Part 2. Application of this method is admissible for mineral oil fractions, especially base oils used for the manufacture of motor oils with a boiling temperature range of 160 °C, minimum, to a maximum of 635 °C.

The method enables calculation of evaporation loss from the boiling range distribution determined by simulated distillation using a nonpolar packed gas chromatography column. The gas chromatogram obtained is separated into equidistant retention-time sections and the area below each of these sections of the curve is determined by integral calculus. The areas obtained are multiplied by an evaporation loss factor which gives the evaporation loss for each interval. The sum of these partial evaporation losses will give the total evaporation loss which shall be standardized by use of a reference oil.

## 18.18
## Analysis and Testing of Lubricating Greases

### 18.18.1
### Consistency

The consistency of lubricating greases is measured by penetration ISO of a standard cone (ASTM D 217/ISO 2137). Penetration is the depth in tenths of millimeters where a standard cone assembly sinks into the grease. The unworked penetration is measured at 25 °C in a standard cup. A worked penetration is used to establish the consistency of greases within the NLGI consistency grades. The worked penetration is the penetration of a sample of lubricating grease after it has been brought to 25 °C and then subject to 60 double strokes in a standard grease worker and penetrated without delay. In addition to the standard penetrometer cone, a one-quarter and one-half cone method is described in ASTM D 1403. This method is applied when small lubricating grease samples are to be tested.

### 18.18.2
### Dropping Point

The dropping point of lubricating greases, (ASTM D 566 or ASTM D 2265 / ISO 2176) is the temperature at which a conventional soap-thickened grease passes from semi-solid to a liquid state under the conditions of the test or the temperature at which a non soap-thickened grease rapidly separates oil.

### 18.18.3
### Oil Separation

The tendency of lubricating greases to separate oil under static conditions is determined by using different international standards.

A sample of test grease is weighed in a separation cup with a gauze cone bottom. Depending on which method is used the grease is stored either unloaded or loaded with a 100-g weight for a specified number of hours.

The most common methods, with their typical test conditions, are summarized briefly below.

IP 121: test duration 42 h or 168 h at 40 °C; 240 mesh gauze cone
ASTM D 1742: test duration 24 h at 25 °C; 200 mesh gauze cone
DIN 51 817: test duration 18 h or 168 h at 40, 80, or 120 °C; 60 mesh gauze cone

### 18.18.4
### Shear Stability of Greases

#### 18.18.4.1 Prolonged Grease Working

Grease working as described in ASTM D 217/ISO 2137 can be modified in terms of the number of double strokes in the grease worker.

Prolonged grease working applying 10 000, 60.000, or 100 000 double strokes has been established to assess the shear stability of greases under mechanical stress.

#### 18.18.4.2 Roll Stability of Lubricating Greases

Grease (50 g) is transferred to a rotating steel cylinder containing a 5-kg roller. After rolling for 2 h at a rotating speed of 165 rpm and a temperature of 20 to 35 °C the grease consistency is determined in a one-quarter of one-half cone as described in ASTM D 1403 (Sect. 18.18.1).

The difference between the worked penetration before and after treatment in the grease roller is a measure of the effect of shear working on grease consistency.

This test can be modified, for example by use of higher temperatures, e.g. 80 °C, while rotating or addition of water to the grease sample, to assess the high-temperature roll stability or the water-absorption capacity, respectively.

### 18.18.5

### High-temperature Performance

The lifetime of lubricating greases at elevated temperatures can be assessed in accordance with ASTM D 3336.

A grease lubricated SAE 204 size ball bearing is driven by a constant belt tension motor assembly at 10 000 rpm at elevated temperatures. The test is usually run at 150 °C and the number of hours until grease lubrication fails is recorded. One of three conditions is used to indicate failure:

- the spindle input power increases to three times the steady state conditions at test temperature;
- the temperature of the bearing increases by more than fifteen degrees above the test temperature; or
- loading of the test bearing or slippage of the driving belt occurs.

### 18.18.6

### Wheel Bearing Leakage

#### 18.18.6.1 Leakage Tendency of Automotive Wheel Bearing Greases

In the ASTM D 1263 standard 90 g test grease is distributed in a modified front-wheel hub and spindle assembly. The hub is rotated at a speed of 660 rpm for 6 h at a spindle temperature that is increased to and then maintained at 105 °C.

Leakage of grease or oil, or both, is measured, and the condition of the bearing surface is noted.

#### 18.18.6.2 Wheel-bearing Leakage Under Accelerated Conditions

ASTM D 4290 describes the leakage tendencies of automotive wheel-bearing greases under accelerated conditions.

A specified front wheel hub–spindle–bearing assembly is filled with a total of 60 g lubricating grease and run in a 20-h cycle at 1000 rpm at a temperature of 160 °C.

The weight of grease leaking into a collector under these conditions is weighed and reported as grease leakage (%).

### 18.18.7
### Wheel-bearing Life

ASTM D 3527 contains a laboratory procedure for evaluating the high-temperature performance of wheel bearing greases under prescribed conditions.

The test grease is distributed in the bearings of a modified automobile front wheel hub–spindle–bearing assembly.

With the bearings thrust-loaded to 111 N the hub is rotated at 1000 rpm for 20 h with the spindle temperature maintained at 160 °C; the test is then turned off for 4 h in each 24-h operating cycle. The test is terminated when grease deterioration causes the drive motor torque to exceed a calculated motor cut-off value.

The accumulated on-cycle hours are reported as the result.

### 18.18.8
### Water Resistance

#### 18.18.8.1 Water Washout Characteristics

In the test described in ASTM D 1264 a specified tared ball bearing is packed with 4 g test grease, inserted in a housing, and rotated at 600 rpm. Water is then directed on the bearing housing at 5 mL $s^{-1}$ and 79 °C through a nozzle of diameter 1 mm. After 60 min the grease washed from the bearing under these conditions is determined by differential weighing and reported (%).

#### 18.18.8.2 Water Spray-off Resistance

In accordance with ASTM D 4049 a film of grease 0.8 mm thick is spread on a steel panel covering a defined area. The panel is weighed then inserted in a cabinet and the grease film is sprayed with water at 38 °C under a pressure of 275 kPa through a 3.18 mm spray nozzle. After 5 min the grease removed from a defined area is determined by reweighing. The identity of the grease and amount removed by spraying (%, to the nearest 1 %) are reported.

### 18.18.9
### Oxidation Stability of Lubricating Greases by the Oxygen Pressure-vessel Method

A method for testing the resistance of lubricating greases to oxidation under static storage conditions at elevated temperature is described in ASTM D 942. Five glass dishes each filled with 4 g grease are placed in a suitable rack in a pressure vessel equipped with a pressure gauge. The vessel is filled with oxygen of purity no less than 99.5 % and heated to 99 °C. The initial oxygen pressure is adjusted to 110 PSI and the pressure decrease caused by consumption of the oxygen is recorded at stated intervals. The extent of oxidation corresponds to the loss of pressure and will be reported as pressure drop in kPa for a specified test time or the time in hours until a defined pressure drop is obtained.

### 18.18.10
### Corrosion-preventive Characteristics

#### 18.18.10.1 Rust Test

The ASTM D 1743 test method covers determination of the corrosion-preventative properties of greases using grease-lubricated tapered roller bearings stored under wet conditions. Three new, cleaned and lubricated bearings are operated under a light thrust load for 60 s to distribute the grease in a manner comparable with that found in service. The bearings are then exposed to distilled water at 52 °C for 48 h.

The reported result is pass or fail as determined for at least two of the three bearings. The criterion for fail is the presence of a spot of corrosion 1.0 mm or larger in the longest dimension on the raceway, ignoring the number of spots. Spots that could easily be removed by rubbing slightly with a soft tissue are not regarded as corrosion of the bearing.

#### 18.18.10.2 EMCOR Test

Determination of the rust-prevention characteristics of lubricating greases under dynamic wet conditions is described in ASTM D 6138/IP 220/DIN 51 802/AFNOR T60-135 and ISO CD 6294. The bearings used are commonly the SKF 1306 K/236 725 for which precision has been evaluated. The test grease (10 g) is distributed evenly in new and cleaned bearings and the lubricated bearings are mounted in a rack that enables partial immersion in distilled water, synthetic sea water, or sodium chloride solution. At a speed of 83 rpm under no applied load the bearings are run in accordance with the schedule: two times 8-h run/16 h stand, then 8-h run/108h stand. After this procedure the bearings are dismantled and the outer ring tracks area is visually inspected for corrosion. The extent of corrosion is reported as a rating from 0 to 5, indicative of no corrosion, traces of corrosion, light, moderate heavy, and severe corrosion. The standard methods mentioned are assessment tools enabling determination of the extent of corrosion.

#### 18.18.10.3 Copper Corrosion

The tendency of lubricating greases to corrode copper under static conditions is determined by use of the ASTM D 4048 test method. A polished copper strip is stored at 100 °C in the test grease for 24 h and tarnishing of the copper is reported as described for the ASTM D 130 method (Sect. 18.15).

## 18.19
## List of Equivalent Standardized Methods for Testing Lubricants

This chapter gives an overview of corresponding standards from the national and international view. In the lists below equivalent standards are grouped together which are identical or technically equivalent respectively.

| | AFNOR | ASTM | DIN | IP | ISO | Test method |
|---|---|---|---|---|---|---|
| Aging/ oxidation stability | | | 51352 T1 | 48 | | Determination of oxidation characteristics of lubricating oil |
| | | | 51554 | | | Determination of oxidation characteristics of lubricating oil acc. to Baader |
| | | | | 280 | | Determination of oxidation stability of inhibited mineral turbine oils |
| | T60-150 | D 943 | 51587 | | 4263 | Determination of oxidation stability of inhibited mineral oils |
| | | D 2893 | 51586 | | | Determination of oxidation characteristics of extreme-pressure lubricating oils |
| | | D 4742 | | | | Oxidation stability of gasoline engine oils by thin film oxygen uptake (TFOUT) |
| | | D 2272 | | 229 | | Determination of the relative oxidation stability by rotating bomb of mineral turbine oil |
| | | D 942 | 51808 | 142 | | Determination of oxidation stability of lubricating grease – oxygen bomb method |
| Air release | T60-149 | D 3427 | 51381 | 313 | DIS 9120 | Determination of air release value of hydraulic, turbine and lubricating oils |
| Aniline point | M07-021 | D 611 | 51775 | 2 | 2977 | Determination of aniline and mixed aniline point |
| Ash | M07-045 | D 482 | EN 7 | 4 | 6245 | Determination of ash |
| | T60-143 | D 874 | 51575 | 163 | 3987 | Determination of sulfated ash of lubricating oils and additives |
| | T60-144 | D 128 | 51803 | | | Determination of ash of lubricating greases |
| Atomic spectroscopy | | D 4628 | | | | Emission spectroscopy |

| | AFNOR | ASTM | DIN | IP | ISO | Test method |
|---|---|---|---|---|---|---|
| Boiling range/ distillation | | D 2887 | | | | Boiling range distribution of petroleum fractions by gas chromatography |
| | | | 51581 T2 | | | Determination of evaporation loss – gas chromatographic method |
| | M07-002 | D 86 | 51751 | 123 | 3405 | Determination of distillation characteristics |
| Carbon distribution | | D 2140 | 51378 | | | Determination of carbon-type composition |
| | | D 3238 | | | | Calculation of carbon distribution and structural group analysis of petroleum oils by the n-d-M method |
| Carbon residue | T60-117 | D 524 | | 14 | 4262 | Ramsbottom carbon residue of petroleum products |
| | T60-116 | D 189 | 51551/T1 | 13 | 6615 | Carbon residue of petroleum products-Conradson method |
| Cloud point | T60-105 | D 2500 | | 219 | 3015 | Determination of cloud point |
| Corrosion tests | M07-015 | D 130 | 51759 T1 | 154 | 2160 | Copper corrosion of liquid mineral oil hydrocarbon |
| | T60-151 | D 665 | 51585 | 135 | 7120 | Rust prevention characteristics of steam turbine oils and hydraulic oils |
| | | D 1748 | 51359 | 366 | | Rust prevention characteristics of corrosion preventing oils in humidity cabinet |
| | | B 117 | 50 021 | | | Corrosion tests; spray tests with different sodium chloride solutions |
| | | | 50 017 | | | Climates and their application; condensed water containing climates |
| | T60-135 | | 51802 | 220 | CD 6294 | Rust prevention characteristics of lubricating greases – SKF–Emcor method |
| | | D 130 | 51811 | 154 | 2160 | Copper corrosion of lubricating greases |
| | | D 4627 | 51360/T2 | 287 | | Rust prevention characteristics of water mix metal working fluids-Chip/filter |

| | **AFNOR** | **ASTM** | **DIN** | **IP** | **ISO** | **Test method** |
|---|---|---|---|---|---|---|
| | | | 51360/1 | 125 | | Rust prevention characteristics of water mix metal working fluids – Herbert corrosion test |
| Demulsibility | T60-125 | D 1401 | ISO 6614 | | 6614 | Demulsibility characteristics of lubricating oils |
| Demulsibility | | D-2711 | | | | |
| Density | T60-101 | D 1298 | 51757 | 160 | 3675 | Determination of density, and API gravity of crude oils and petroleum products – hydrometer method |
| Dropping point/ melting point | T60-121 | D 127 | | 133 | 6244 | Determination of drop melting point of wax and petrolatum |
| | T60-102 | D 566 | | 132 | 2176 | Determination of dropping point of lubricating greases |
| Elastomer | | D 4289 | | | | Compatibility of lubricating grease with elastomers |
| | | | 53 521 | | | Testing of rubber and elastomers |
| | | | 53 504 | | 37 | Determination of tensile strength at break,tensile stress at yield, elongation at break and stress values |
| | | | 53 505 | | | Shore A and Shore D hardness test |
| Evaporation | T60-161 | D 5800 | 51581 T1 | | | Determination of evaporation loss of lubricating oil – Noack method |
| Filterability | NF E 48-690 | | | | 13357-2 | Measurement of the filterability of mineral oils |
| Flash point | T60-118 | D 92 | ISO 2592 | 36 | 2592 | Determination of open flash and fire point – Cleveland method |
| | M07-036<br>EN57 | | 51755 | | | Determination of closed flash point – Abel Pensky method |
| | M07-019 | D 93 | 51758 | 34 | 2719 | Determination of closed flash point – Pensky–Martens method |
| Flow pressure | | | 51805 | | | Determination of flow pressure of lubricating greases – Kesternich method |

| | AFNOR | ASTM | DIN | IP | ISO | Test method |
|---|---|---|---|---|---|---|
| Foam | T60-129 | D 892 | | 146 | 6247 | Determination of foaming characteristics of lubricating oils |
| | T60-185 | | | | | Aqueous machining fluid foaming tendency |
| Infrared spectrometry | | | 51451 | | | Analysis by infrared spectrometry |
| Hydrolytic stability | | D 2619 | | | | Hydrolytic stability of hydraulic fluids (beverage bottle method) |
| Neutralization number | T60-112 | D 974 | 51558/T1 | 139 | | Neutralization number – Color-indicator titration method |
| | T60-133 | D 128 | 51809/T1 | 37 | | Determination of acidity and alkalinity of lubricating grease |
| Oil separation | T60-191 | | 51817 | 121 | | Determination of separation from lubricating grease – pressure filtration method |
| | | D 1742 | | | | Oil separation from greases during storage |
| Penetration | T60-132 | D 217 | | 50 | 2137 | Determination of cone penetration of lubricating grease |
| | T60-119 | D 937 | 51580 | 179 | 3986 | Determination of cone penetration of petrolatum |
| | T60-123 | D 1321 | 51579 | 376 | 3992 | Determination of needle penetration of petroleum wax |
| pH value | | | 51369 | | | Determination of pH value of water-mixed metal working fluids |
| Polyaromatic hydrocarbons | | | | 346 | | Determination of polycyclic aromatics – dimethyl sulfoxide extraction method |
| Pour point | T60-105 | D 97 | | 15 | 3016 | Determination of pour point |
| Roll stability | | D 1831 | | | | Roll stability of greases/Shell roller |
| Saponification number | T60-110 | D 94 | 51559/1 | 136 | 6293 | Method of saponification number – titration method |

| | AFNOR | ASTM | DIN | IP | ISO | Test method |
|---|---|---|---|---|---|---|
| Shear stability | | D 3945 | 51382 | 294 | | Determination of shear stability of polymer-containing oils – diesel injector rig method |
| Sulfur | M-07-052 | D 3120 | 51400 T7 | 373 | | Determination of sulfur content – microcoulometry (oxidative) method |
| Total acid number/ Total base number | | D 2896 | | 276 | 3771 | Determination of base number perchloric acid potentiometric titration method |
| | | D 664 | | 177 | | Determination of neutralization number – potentiometric titration method |
| | T60-112 | D 974 | 51558 | 139 | 6618 | Neutralization number – color-indicator titration method |
| Viscosity | T60-100 | D 446 | 51366 | 71 | 3104 | Cannon–Fenske viscosimeter method |
| | T60-100 | D 445 | 51562 | 71 | 3104 | Kinematic viscosity of transparent and opaque liquids/Ubbelohde viscosimeter method |
| | T60-152 | D 2983 | 51398 | 267 | | Determination of low-temperature viscosity of automotive fluids – Brookfield viscosimeter method |
| | | D 2602 | 51377 | 383 | | Determination of apparent viscosity of engine oils between –30 °C and –5 °C – cold cranking simulator method |
| | | D 4741 | | 370 | | Determination of dynamic viscosity – high shear Ravenfield viscometer method |
| | T60-136 | D 2270 | ISO 2909 | 226 | 2909 | Calculating viscosity index from the kinematic viscosity at 40 and 100 °C |
| | | D 4684 | | | | Determination of yield stress and apparent viscosity of engine oils at low temperature |
| | | D 3829 | | | | Predicting the borderline pumping temperature of engine oil |
| | | | 53 015 | | | Falling ball viscosimeter according to Höppler |

| | AFNOR | ASTM | DIN | IP | ISO | Test method |
|---|---|---|---|---|---|---|
| | | D 1092 | | | | Measuring apparent viscosity of lubricating greases |
| | | | 51810 | | | Apparent viscosity of lubricating greases by rotary viscometer |
| Water | T60-154 | D 6304 | 51777/ T1/T2 | 438 | 6296 | Determination of water content of petroleum products by use of the Karl Fischer reagent |
| | T60-113 | D 95 | | 74 | 3733 | Determination of water content of petroleum products – distillation method |
| Water resistance | | D 1264 | | 215 | | Water resistance of greases under dynamic conditions/water washout test |
| | | | 51807/1 | | | Behavior of lubricating greases in the presence of water |

| **AFNOR** | **ASTM** | **DIN** | **IP** | **ISO** | **Test method** |
|---|---|---|---|---|---|
| Wear | D 1947 | 51354/1 | 334/166 |  | FZG gear test rig/load-carrying capacity |
|  | D 2266 | 51350/1 |  |  | Shell four-ball test for greases/mean wear scar diameters determination |
|  | D 2596 | 51350/4 | 239 |  | Shell four-ball test for greases EP properties |
|  | D 4172 |  |  |  | Shell four-ball test for oils/wear test |
|  | D 2783 | 51350/1 | 239 |  | Shell four-ball test for oils/weld test |
|  | D 2509 |  | 326 |  | Timken test for lubricating greases |
|  | D 2782 | 51434/T3 | 240 |  | Timken test for lubricating fluids |

# 19
# Mechanical–Dynamic Test Methods for Lubricants

*Thorsten Bartels*

Mechanical–dynamic lubricant testing has become an essential element in the development of modern lubricants. In this respect, standardized tribological, mechanical–dynamic test rigs and test methods play a decisive role in the development of lubricants and additives. Standardized test methods are the basic means of achieving the required performance of OEM specifications for lubricants. A variety of so-called house-internal methods complete the range of today's tribological, mechanical–dynamic testing.

## 19.1
## Tribological System Categories within Lubricant Tests

Today's wide range of tribological, mechanical–dynamic test machines and test methods includes small laboratory instruments and field tests under real conditions. The different types of lubricant test are classified, amongst others, by the German DIN 50322 (Fig. 19.1) [19.1]. Here, it must be remembered that the continuing, gradual reduction of the test-sequence plan from a field test to a simple friction contact leads to substantial improvement of repeatability and reproducibility of a test method. The validity of the result from a simple screening test, however, decreases proportionally. A problem of the classification according to DIN 50322 is the allocation of certain test devices and methods to the corresponding categories. The borders shown enable overlapping and combination of categories.

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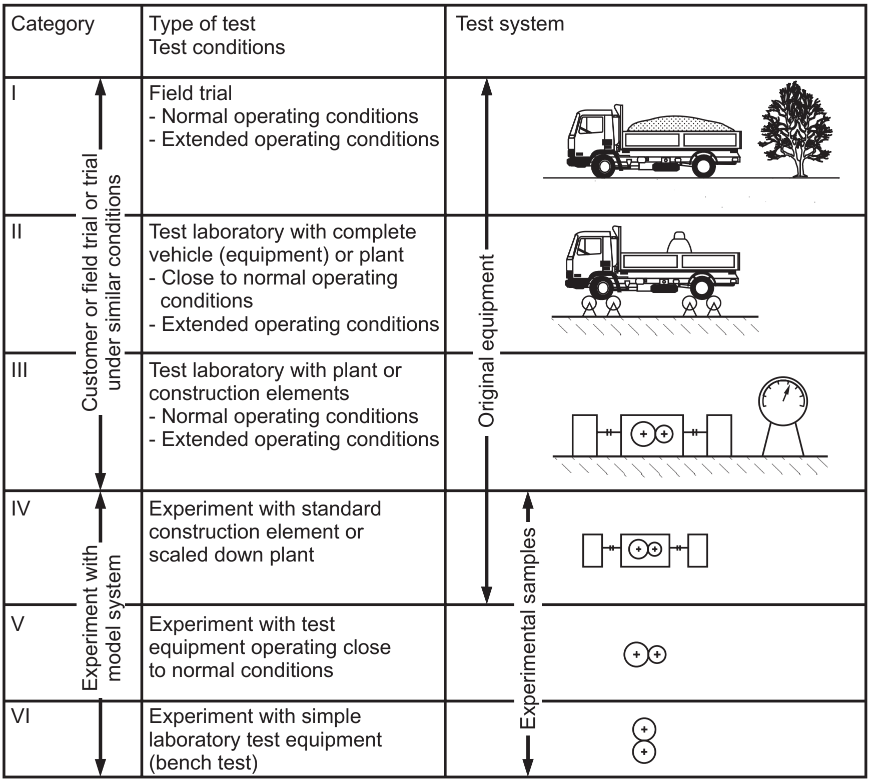


**Fig. 19.1** Tribological system categories within lubricant testing.

## 19.2
## Simple Mechanical–Dynamic Lubricant Test Machines

### 19.2.1
### Four-ball Apparatus

The four-ball apparatus is one of the oldest and best-known simple test benches for liquid and solid lubricants. The four-ball geometry is a popular test method, because test pieces are inexpensive and wear measurement is simple. This machine enables the precise determination of anti-wear properties and coefficient of friction. This simple geometry (Fig. 19.2) also enables the determination of extreme pressure characteristics of greases and oils based on welding tests that are standardized world-wide. A roller-bearing ball rotates under pressure and at constant speed on three fixed steel balls. The contact is lubricated with oil or a solid lubricant. The gradual increase of the normal force (contact pressure) enables determination of the weld load, anti-wear protection, and friction coefficients of a lubricant. During these tests the ball's surface will first produce wear marks on the fixed balls which lead to effects of different oils and additives.

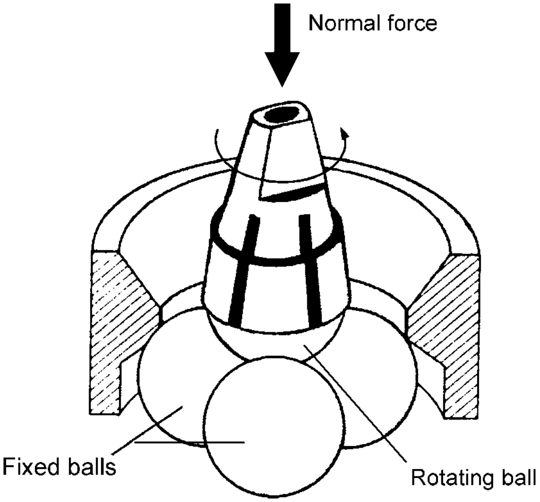


**Fig. 19.2** Schematic diagram of the test principle of the four-ball apparatus.

In recent years, several adapters have been developed for the four-ball apparatus to furnish information on the pitting load capacity and the shear stability of polymer-containing lubricants. Surface-modified steel balls (VW-PV-1444) and a variety of tapered roller bearings (VW-PV-1417, DIN 51354, part 6 or CEC L-45-T-98) are used (Fig. 19.3). In addition, further modifications of the test adapters enable the determination of friction coefficients and temperature behavior of a lubricant within the roller bearing. In accordance with VW-PV-1454 the test adapter used determines

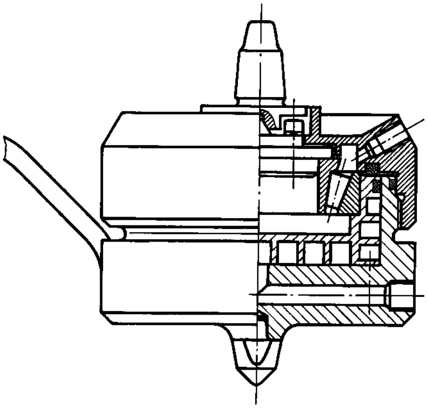


**Fig. 19.3** Test adapter for determination of the shear stability of lubricants containing polymers.

the steady-state operating temperature, temperature increase, and the friction of the test bearing in relation to the respective lubricant, using a axial thrust ball bearing. More recent results with this test adapter have shown that the steady-state oil sump temperatures measured can be transferred to transmissions and industrial gears, depending on the lubricant, after being adjusted to the realistic load and speed ratio referring to the application. As ever, today's numerous specifications for gear and hydraulic lubrication oils and for all types of grease and paste require four-ball data for most of the lubricants. Table 19.1 lists the common test standards mostly used for the four-ball apparatus.

**Tab. 19.1** Four-ball apparatus test standards.

| *Application* | *Test method* |
|---|---|
| Standard test method for wear-preventive characteristics of lubricating fluids | ASTM D 4172 |
| Wear-preventive characteristics of lubricating grease (four-ball method) | ASTM D 2266 |
| Standard test method for the determination of the friction coefficient of lubricants using the four-ball apparatus | ASTM D 5183 |
| Measurement of extreme pressure properties of lubricating fluids (four-ball method) | ASTM D 2783 |
| Measurement of extreme pressure properties of lubricating grease (four-ball method) | ASTM D 2596 |
| Standard test method for determination of load-carrying capacity and mean Hertz load | FTMS No. 791 b Method 6503.2 |
| Determination of extreme pressure and anti-wear properties of lubricants – four-ball apparatus | IP 239/85 |
| Standard test method for lubricants using the Shell four-ball apparatus | DIN 51350 |
| General working principles | DIN 51350, Part 1 |
| Weld load of liquid lubricants | DIN 51350, Part 2 |
| Wear load of liquid lubricants | DIN 51350, Part 3 |
| Weld load of solid lubricants | DIN 51350, Part 4 |
| Wear load of solid lubricants | DIN 51350, Part 5 |
| Shear stability of polymer-containing lubricants | DIN 51350, Part 6 |
| Viscosity shear stability of transmission lubricants – tapered roller bearing | CEC L-45-T-98 |
| Mechanical shear stability of engine oils | VW-PV-1450 |
| Pitting load capacity of solid lubricants | VW-PV-1417 |
| Pitting load capacity of liquid lubricants | VW-PV-1444 |
| Standard test method for temperature increase in the axial thrust ball bearing adapter (ARKL) | VW-PV-1454 |

### 19.2.2
### Reichert's Friction-wear Balance, Brugger Apparatus

The Reichert's friction-wear balance and the Brugger apparatus (according to DIN 50347) are important tools for determination of the wear characteristics of water-containing and nonwater-containing metal-working fluids, hydraulic fluids, and greases. Most manufactures in the metal-working industry specify wear-data according to Reichert or Brugger to ensure an adequate quality standard and adequate quality control for their metal-working and hydraulic fluids. Almost each development of a new formulation includes these wear tests. By means of a lever-handle system a firmly clamped cylindrical roller is pressed axial-crossed against a slip ring by an applied normal force (normal load). The slip ring rotates cross-directionally to the roller. In accordance with to Reichert approximately the lower third of the test ring is dipped into the test fluid. After a walkway of 100 m at a constant speed of the rotating slip ring, the elliptical wear mark produced on the roller's surface is measured. According to Brugger the slip ring rotates for 30 s with 5 mL fluid lubricating the friction contact. The fluid must remain on the surface for the duration of the Brugger test.

On the basis of the wear marks measured, the average specific contact pressure can be re-calculated from the known and constant normal force. The wear is a result of the wear rate. The Reichert friction-wear balance and the Brugger apparatus equalize the contact pressure by producing smaller or larger wear marks. Figure 19.4 shows a schematic diagram of the test arrangement and Fig. 19.5 gives examples of wear marks measured with the Reichert friction-wear balance. Common test standards are listed in Table 19.2.

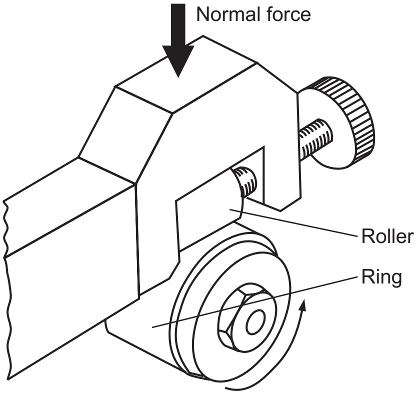


**Fig. 19.4** Schematic diagram of Reichert's friction-wear balance.

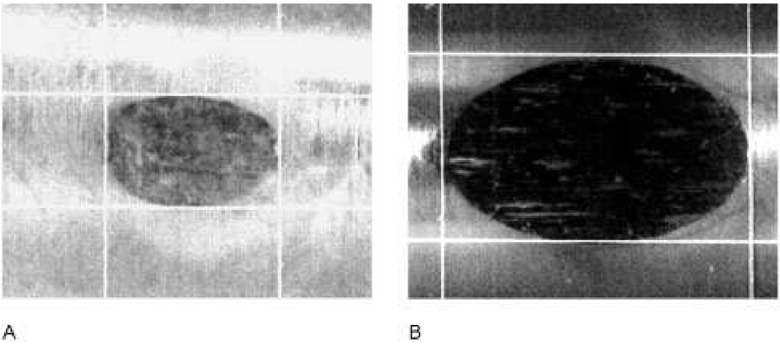


**Fig. 19.5** Examples of wear marks determined in accordance with Reichert: A. canned milk; B. cola.

**Tab. 19.2** Reichert and Brugger test standards.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| Reichert's friction-wear balance | Standard test method for determining the pressure compensation capacity using the frictional wear-balance according to Reichert | VKIS – worksheet No. 6 |
| Brugger rig | Testing under boundary lubricating conditions using the Brugger apparatus | DIN 51347 |
| | General working principles | DIN 51347, Part 1 |
| | Procedures for greases | DIN 51347, Part 2 |

### 19.2.3
### Falex Test Machines

Falex test machines are mostly standardized by ASTM. They are used for testing lubrication oils, greases and solids. World-wide these test machines are widely used to measure and evaluate the properties of lubricants.

#### 19.2.3.1 Falex Block-on-ring Test Machine

The Falex block-on-ring test machine is used as a development and quality-control instrument for simulation of sliding and oscillating wear between lubricated and dry conditions. The pressure chamber and heater cover enable testing at elevated temperature and in noncorrosive gases. Test geometry consists of a rectangular test block that is loaded on a rotating or oscillating ring. A uniform contact velocity profile is created. The block can be replaced by a ball (increase contact pressure) or conforming block (reduce pressure). Test blocks can be machined to test a wide variety of products. The test machine has a variable test load (<4000 N) and speed (<3000 rpm). A heated test chamber (150 °C; higher if special seals are used), a high pressure test chamber (<10 bar) for noncorrosive gases or regulation of humidity, and on-line measurement of friction, temperatures, and total wear are additional features. The test standards are listed in Table 19.3.

**Tab. 19.3** ASTM test standards for Falex machines.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| Falex block-on-ring | Calibration and operation of the Falex block-on-ring friction and wear-testing machine | ASTM D 2714 |
| | Wear life of solid-film lubricants in oscillating motion | ASTM D 2981 |
| | Test method for wear-preventive properties of lubricating greases using the (Falex) block-on-ring machine in oscillating motion | ASTM D 3704 |
| | Practice for ranking resistance of materials to sliding wear (using block-on-ring wear test) | ASTM G 77 |
| Falex pin and vee block | Endurance (wear) life and load carrying capacity of solid film lubricants | ASTM D 2625 |
| | Method for measuring wear properties of fluid lubricants (Falex method) | ASTM D 2670 |
| | Measurement of extreme pressure properties of fluid lubricants (Falex pin and vee block methods) | ASTM D 3233 |
| | Evaluating thin-film lubricants in a drain and dry mode using a pin and vee block machine | ASTM D 5620 |
| Falex high-performance multispecimen test machine | Test method for wear rate and coefficient of friction of materials in self lubricated rubbing contact using a thrust washer testing machine | ASTM D 3702 |
| | Test method for wear preventive characteristics of lubricating fluids (four-ball method) | ASTM D 4172 |
| | Coefficient of friction using a four-ball wear test machine | ASTM D 5183 |
| | Wear-preventive characteristics of lubricating grease (four-ball method) | ASTM D 2266 |
| | Wear testing with a pin-on-disk apparatus | ASTM G 99 |
| Falex tapping torque | Test method for comparing metal-removal fluids using the tapping torque test machine | ASTM D 5619 |

#### 19.2.3.2 The Falex Pin and Vee Block Test Machine

The Falex pin and vee block test machine is the oldest industrially standardized friction and wear-test machine. A rotating journal pin is pressed between two blocks with V-notches. At constant rotational speed the normal force is increased stepwise. The scuffing load is determined at the point at which the pin at a softer neck breaks at the designated breaking point. If a lubricant withstands the applied force, the wear-loss of the softer pin is measured. This test is conducted at ambient temperature. Figure 19.6 shows a schematic diagram of the test arrangement and the test standards are listed in Table 19.3. This test runs at fixed speeds enabling loads up to 3000 lb (13 350 N) for different test materials while controlling on-line wear, friction, load, and temperatures via the monitoring system.

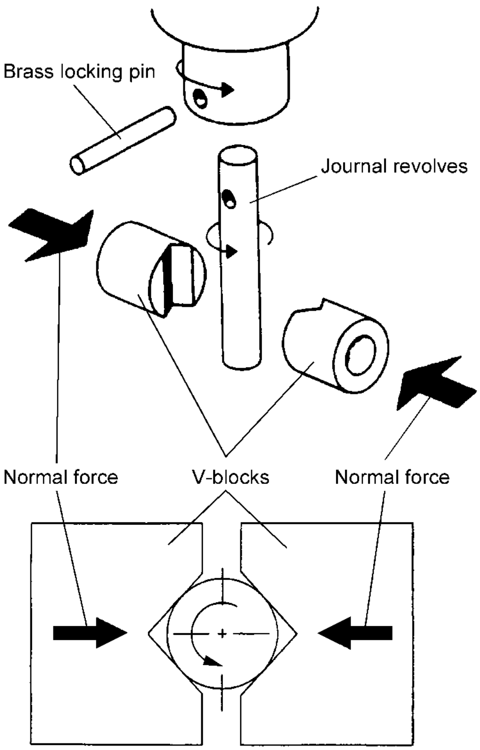


**Fig. 19.6** Schematic diagram of the Falex pin and vee block test machine.

#### 19.2.3.3 Falex High-performance Multispecimen Test Machine

The Falex high-performance multispecimen test machine is extremely flexible test equipment for industrial simulations. The Multispecimen machine can be used for almost any tribological investigation that involves sliding or rolling contacts. Over 60 standard adapters are available for performing a variety of simulation tests including sliding wear, abrasion, erosion, forming, impact, and rolling/sliding. This test machine enables any combinations of high load (<10 000 N), high speed (<10 000 rpm), and high temperature testing (up to 800 °C). The PC-controlled monitoring system enables programming of acceleration (1000 rpm $s^{-1}$) and loading profiles (1000 N $s^{-1}$). Automatic simulation of dynamic conditions and start–stop cycles, variable flexibility enabling simulation of the effects of low-component stiffness, and on-line measurement of friction, speed, load, and temperature are current standards. The test standards used worldwide are listed in Table 19.3.

#### 19.2.3.4 Falex Tapping Torque Test Machine

The Falex tapping torque test machine was developed to enable repeatable and precise characterization of cutting lubricants. A Procunier tapping head performs tapping operations and a load cell monitors torque produced on a nut blank. The efficiency of cutting fluids can be determined in accordance with ASTM D 5619 (Table 19.3). Tapping simulation by tapping operation in standardized nut blanks (aluminum, steel, and stainless steel), drilling, reaming and forming simulations are possible with this test machine. A roll-forming adapter characterizes rolling fluids. It has a dual speed range (10–450/20–900 rpm) and a data-acquisition system enabling torque trace and torque averages to be stored. Average torque values are plotted and compared with a reference to determine the efficiency of the fluid.

### 19.2.4
### Timken Test Machine

The Timken test machine is licensed by the company Timken, a steel-producer and bearing manufacturer. It is essentially used to determine the anti-scoring protection afforded by greases and oils, in accordance with ASTM D 2509 and D 2782. It can also be used to determine wear resistance or adhesion strength of coatings supporting variable speed and pneumatic loading. Figure 19.7 shows a schematic diagram of the test arrangement. In the steel industry the Timken rig is still a very important tool. Gear oils and extreme-pressure greases for roller bearings used in these industries must meet Timken specifications. The lubricant test is conducted in a friction arrangement comprising a cuboid-arranged block and a rotating test cup (Fig. 19.7). The test is run with a lubrication oil circulation system, which can be adjusted variably, and with a grease feeder with a feed volume of 45 g min$^{-1}$. The normal force (load stage), which is increased gradually, is brought into friction contact via the test block. Weighing of the test block and the test cup enables the determination of the wear-loss. The duration of the test at each load stage is 10 min at a speed of 800 rpm.

Scuffing between the test cup and the test block depends on the applied normal force and will, first, lead to score marks on the surfaces of the test pieces. Scuffing is connected with a sudden drop in rotational speed and/or with an increased noise level. If scuffing occurs during a load stage, a nonscuffing test run in the previous load stage must be proven and documented as the determined so-called "good-load-stage"

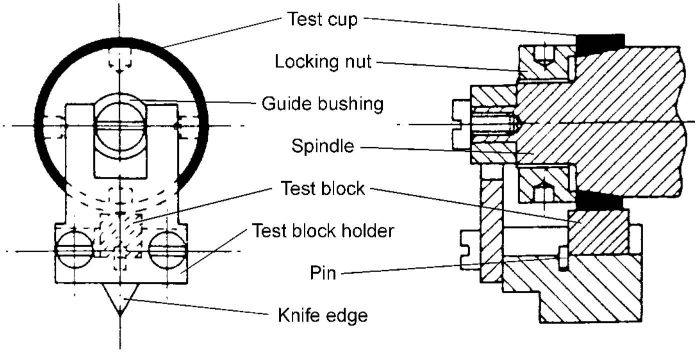


**Fig. 19.7** Schematic diagram of the Timken test machine.

together with the total wear of the test block and test cup (test ring). Greases are tested at ambient temperature, lubrication oils at 40 °C. The test standards are listed in Table 19.4. Timken specifies an EP lubricant for bearing applications as one with a good-load of 45 lb (200 N). To meet the requirement of anti-wear, the steel industry usually defines a maximum of 5 to 6 mg at a good-load-stage of 40 lb (178 N). Higher good-loads will very often lead to greater wear.

**Tab. 19.4** Test standards for Timken machines.

| *Application* | *Test method* |
|---|---|
| Measurement of load-carrying capacity of lubricating grease (Timken) | ASTM D 2509<br>IP 326/83 |
| Measurement of extreme-pressure properties of lubricating fluids (Timken) | ASTM D 2782<br>IP 240/92 |
| Standard test method in the mixed-lubrication regime using the Timken test machine | E-DIN 51434 |
| General working principles | E-DIN 51434, Part 1 |
| Procedures for lubricating oils | E-DIN 51434, Part 2 |
| Procedures for lubricating greases | E-DIN 51434, Part 3 |
| Standard test method for liquid and plastic lubricants using the Timken test machine | VDEh<br>SEB 181302 |

### 19.2.5
### High-frequency Reciprocating Test Machines

#### 19.2.5.1 High-frequency Reciprocating Rig (HFRR)

The HFRR is a microprocessor-controlled reciprocating friction and wear test system which enables rapid, repeatable assessment of the performance of fuels and lubricants. It is particularly suitable for wear-testing relatively poor lubricants, for example diesel fuels, and for boundary friction measurements of engine oils, greases, and other compounds. The HFRR test for diesel fuel lubricity gained CEC "A" (approval) status in September 1996 after an extensive round-robin program. Other standards based on the HFRR system are listed in Table 19.5.

**Tab. 19.5** Test standards for high-frequency reciprocating test machines.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| HFRR | Measurement of diesel fuel lubricity | CEC F-06-A-96 |
| | Standard test method for evaluating the lubricity of diesel fuels by use of the high-frequency reciprocating rig (HFRR) | ASTM D 6079 |
| | Assessment of lubricity by use of the high-frequency reciprocating rig (HFRR) Part 1 test method | BS ISO 12156-1<br>IP 450/2000 |
| | Automotive fuels – diesel – requirements and test methods | EN 590 |
| | Gas oil – testing method for lubricity | JPI-5S-50-98 |
| SRV | Standard method for measuring the friction and wear properties of extreme-pressure (EP) lubricating oils by use of the SRV test machine | ASTM D 6425 |
| | Standard test method for determining the extreme-pressure properties of lubricating greases by use of a high-frequency, linear-oscillation (SRV) test machine | ASTM D 5706 |
| | Standard test method for measuring the friction and wear properties of lubricating grease using a high-frequency, linear-oscillation (SRV) test machine | ASTM D 5707 |
| | Tribological test method using a high-frequency, linear-oscillation test machine (SRV); general working principles | DIN 51834<br>DIN 51834, Part 1 |
| | Determination of measured friction and wear quantities for lubricating oils | DIN 51834, Part 2 |
| | Determination of the tribological behavior of materials in reaction with lubricants | DIN 51834, Part 3 |
| | Definition of data formats for test results | DIN 51834, Part 5 |
| | Tribological test method for solids using a high-frequency, linear-oscillation test machine (SRV) | DIN 51834, Part 6 |
| | Textile machinery and accessories – needle and sinker lubricating oils for weft knitting independent needle machines – part 2 | DIN 62136, Part 2 |
| | Minimum requirements synthetic oil based | |

### 19.2.5.2 High-frequency, Linear-oscillation Test Machine (SRV)

The high-frequency, linear-oscillation test machine (SRV) is designed to simulate very small displacements under well known conditions of load, speed, and environmental control. It is used to investigate typical fretting phenomena occurring in automotive components, aircraft, and vibrating machines. The effects of humidity and operating conditions on the surface degradation of coatings and materials can be tested on such a machine.

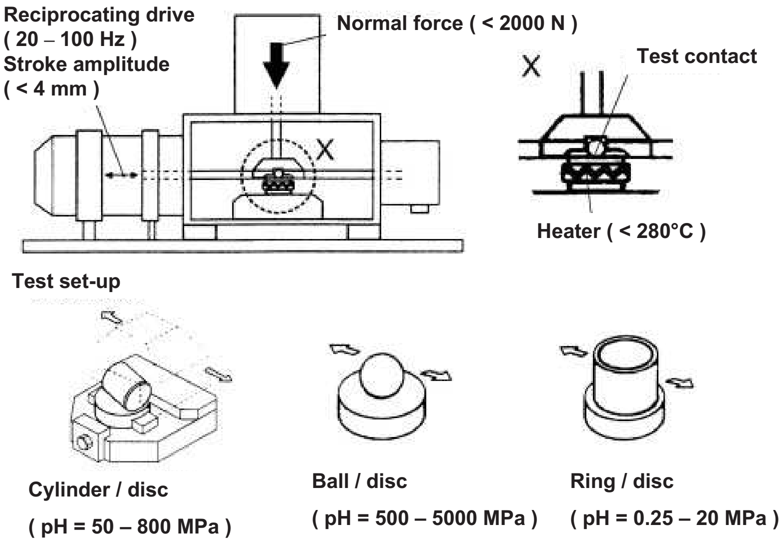


**Fig. 19.8** High-frequency, linear-oscillation test machine (SRV).

Test methods cover procedures for determining the extreme-pressure properties of lubricating fluid and greases in high-frequency, linear-oscillation motion. Further test methods cover procedures for determining the coefficient of friction of a lubricating oil or grease and its ability to protect against wear when subjected to high-frequency, linear-oscillation motion at a test load of 200 N, frequency of 50 Hz, stroke amplitude of 1.0 mm, duration of 2 h, and temperature within the range of the test machine, specifically, ambient to 280 °C. Other test loads (10 to 1200 N for model SRV-I, 10 to 1400 N for model SRV-II, and 10 to 2000 N for model SRV-III), frequencies (5 to 500 Hz) and stroke amplitudes (0.1 to 4.0 mm) can be varied if specified. The precision of this test method depends on the conditions stated and on test temperature (50 or 80 °C). Average wear scar dimensions on ball and coefficient of friction are determined and reported. Further test modes are linear reciprocation testing scuffing under variable humidity using electrical contact measurements by normal load variations or surface fatigue evaluation. Figure 19.8 shows a schematic diagram of the test arrangement and important test standards are listed in Table 19.5. A big advantage of using the SRV machine is the possibility of simply and quickly performing systematic property studies, which require only small quantities of lubricant. Measurement of properties different from those of the test standards will, however, require sufficient experience with this test machine and with interpretation of the test results.

### 19.2.5.3 **Mini Traction Machine (MTM)**

The MTM is a computer-controlled, precision traction-measurement instrument which enables fully automated traction mapping of lubricants and other fluids. The machine simulates the lubrication regime found in nonconforming components such as cams, valve trains, gears, and rolling element bearings. The test contact is formed between a polished three-quarter-inch ball and a 46-mm diameter disk, each independently driven to produce a sliding/rolling contact. To perform a test a small sample of fluid is placed in the test reservoir and the system steps through a series of loads, speeds, slide/roll ratios and temperatures following any one of several standard test programs or a custom program defined by the operator. Typically, a complete series of traction and Stribeck curves at five different temperatures up to 150 °C can be generated, guaranteeing repeatable test results. The MTM system provides a rapid way of evaluating the performance of new formulations of traction fluids at the development stage. The instrument has been designed so that high contact pressures, temperatures, and speeds can be achieved by means of a safe, compact, bench mountable system. It is an important development tool for lubricants, although no standardized test methods are available.

### 19.2.6
### Low-velocity Friction Apparatus (LVFA), Tribometer

The General Motors low-velocity friction apparatus (LVFA) was designed in the nineteen-sixties and has proved to enable very repeatable evaluation of friction–velocity characteristics at low sliding velocities [19.2, 19.3]. The LVFA uses a small-scale annular part manufactured from the original friction material from wet clutch, wet brake, or torque converter application, running against a steel counterpart. The LVFA uses a flywheel coast-down to evaluate a full range of sliding speeds. The normal load is applied by deadweights through a lever at the bottom of the apparatus. The variable-speed friction tester (VSFT) [19.4, 19.5] and the μ-v-Tester [19.6, 19.7] are modified versions of the LVFA. The tribometer test used in Europe is also very similar to the LVFA [19.8, 19.9].

### 19.2.7
### Diesel Injector Apparatus

The diesel injector apparatus enables evaluation of the shear stability of polymer-containing fluids. The test methods measure the viscosity loss (%) of polymer-containing fluids when evaluated by a diesel injector apparatus procedure that uses European diesel injector test equipment. The viscosity loss reflects polymer degradation by shear at the nozzle. This test apparatus itself is defined by a CEC L-14-A-93 procedure. The ASTM test methods differ from CEC-L-14-A-93 in the period of time required for calibration. For a specified number of cycles, a sample (170 mL) of lubricant is subjected to a shear stability test in the apparatus. Before and after the shear stress the kinematic viscosity is determined at 40 °C for hydraulic fluids and at 100 °C for crankcase oils. The shear stability is defined by the relative viscosity drop. According to DIN 51382, the number of cycles is 30 for crankcase oils and 250 cycles for hydraulic fluids. According to ASTM, viscosity loss is evaluated after both 30 and 90 cycles of shearing. The ASTM D 2603 sonic shear test (Table 19.17) has been used for similar evaluation of shear stability. Limitations are as indicated in the significance statement. No detailed attempt has been undertaken to correlate the test results of the diesel injector apparatus with those of the sonic shear test method. The ASTM D 5275 test method also shears oils in a diesel injector apparatus but may give different results, as itemized in Table 19.6.

**Tab. 19.6** Test standards for the diesel-injector apparatus.

| *Application* | *Test method* |
|---|---|
| Evaluation of the mechanical shear stability of lubricating oils containing polymers (fuel injection pump) | CEC L-14-A-93 |
| Test method for fuel-injector shear stability test (FISST) for polymer-containing fluids | ASTM D 5275 |
| Standard test method for shear stability of polymer-containing fluids using a European diesel injector apparatus | ASTM D 6278 |
| Standard test method for shear stability of polymer-containing fluids using a European diesel injector apparatus at 30 and 90 cycles | ASTM D 7109 |
| Determination of the shear stability of polymer-containing oils – diesel injector rig method | IP 294/83 |
| Determination of the shear stability of polymer-containing oils | DIN 51382 |

## 19.3
## Performance Tests for Gear Oil Applications

The Erdco universal test rig and the IAE gear machine are older test rigs specified for performance tests of gear oils. The Ryder gear-test rig is used to obtain a scuffing or load-capacity rating for aviation oils. The load capacity rating is derived from scuffing criteria only. Scuffing is one of several surface-deterioration mechanisms affecting the life and durability of aircraft bearings and gear hardware. As a result of its use for many years as a qualification test, the Ryder gear-test method has acquired a large database. The US Navy has supported efforts to provide Ryder-like load capacity data for gas turbine and gearbox oils. These efforts also expand the scope of oil characterization beyond the perspective of a pass/fail or ranking of oils, with scuffing performance as the only criterion.

To provide continuity between Ryder gear load capacity data and current or future oil-characterization methods, this test method ranks oils with regard to a scuffing failure event. The new gear-oil test methods characterize oils with regard to traction (friction, gear efficiency) behavior. The introduction of high thermal stability (HTS) oils and, particularly, corrosion-inhibited (CI) oils, has emphasized the need for greater testing sensitivity for oils with lower than average lubricating performance. Low lubricating performance, as measured by the Ryder test, is apparent as a superficial form of scuffing ("micro-scuffing"). Other gear failures that must be covered with the new test methods are fatigue life ("pitting") and superficial pitting ("micropitting"). For these reasons the FZG Gear-test rig has been further improved by much development work and many research projects led to improvement. Much of this research was performed by the FVA and by the Institute for Machine Elements and Gear Research Center (FZG, Munich, Germany). The FZG gear-test rig is currently widespread and the most widely accepted of application-related gear-oil tests. Gear-oil tests are specified by the Coordinating Research Council (CRC) and by the Coordinating European Council (CEC), which are nonprofit organizations that direct, by committee action, engineering and environmental studies on the interaction between automotive equipment and petroleum products. Sustaining members of CRC are the American Petroleum Institute (API), the Society of Automotive Engineers (SAE) and automobile manufacturers (General Motors, Ford, DaimlerChrysler, Honda, Toyota, and Volkswagen). The CEC is an industry-based organization developing new test procedures for performance testing of automotive engine oil, fuels, and transmission fluids. The CEC represents the automotive fuels, lubricants, additives, and allied industries in the development of performance tests, usually via the European industries ACEA, ATIEL, ATC, and CONCAWE.

### 19.3.1
### FZG Gear-test Rig

The FZG gear-test rig is specified in DIN 51354 part 1 [19.10]. Figure 19.9 shows a schematic diagram of the FZG gear-test rig. The FZG rig is a test machine with a mechanical power circuit. The drive gearbox and the test gearbox (slave gearbox) are connected by two torque shafts, by friction. On one shaft is a clutch to apply the load. The temperature in both gearboxes can be set and controlled. The test rig is driven by an electric motor with a variable speed, usually 1500 rpm. The speed can be adjusted from 7.5 to 3000 rpm in two directions of rotation. The load can either be applied either by use of a weight system or with the tension ratchet.

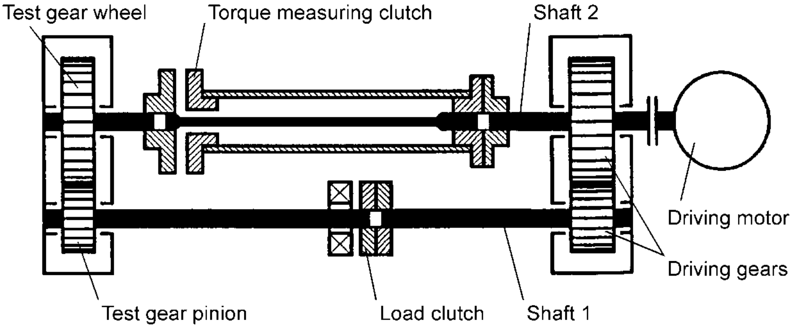


**Fig. 19.9** Schematic diagram of the FZG gear-test rig.

#### 19.3.1.1 FZG EP Tests – Scuffing

With the lubricant to be tested specific gear teeth (A-type) run at a constant speed with a pitch line velocity of 8.3 m $s^{-1}$ (1500 rpm) and with an established initial lubricant temperature of 90 °C. The test gears of type A used in this test have been adjusted to a shape which is particularly scuffing-sensitive. The FZG test method

A/8.3/90 for lubrication oils has been standardized world-wide by ISO, ASTM, IP, CEC and DIN (Table 19.7). The stress on the tooth flanks may be increased gradually. After completion of the test run, or after each load stage if testing in gradual steps (gravimetric method), changes on the tooth flank surfaces (flank damage, scoring) are recorded by description, photographs, tooth surface roughness measurements, or contrast print. Changes in weight of the test wheels may also be determined. These test results can be transferred to other gears and even play a role in the design and calculation of gears according to DIN 3990 [19.11]. Further procedures, for example A/16.6/90 and A/16.6/140, are also used as variations which have not yet been standardized.

**Tab. 19.7** Test standards for gear and axle oil.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| Erdco universal test rig | Standard test method for load-carrying capacity of petroleum oil and synthetic fluid gear lubricants | ASTM D 1947 (FTMS No. 791 B method 6512)[a] |
| Ryder gear-test rig | Load-carrying capacity of lubricating oils Ryder gear machine | FTMS No. 791 C method 6508.2[a] |
| IAE gear machine | Determination of the load-carrying capacity of lubricants – IAE gear machine method | IP 166/77[a] |
| FZG EP gear-test rig | Standard test method for lubricants using the FZG gear-test rig | DIN 51354 |
| | General working principles | DIN 51354, part 1 |
| | Standard test method A/8.3/90 for lubricating oils | DIN 51354, part 2 |
| | Standard test method A/2.8/50 for greases | DIN technical report 74 |
| | Load-carrying capacity test for transmission lubricants | CEC L-07-A-95 |
| | Determination of load-carrying capacity of lubricants – FZG gear machine method | IP 334/93 |
| | FZG test method A/8.3/90 for relative scuffing load-carrying capacity of oils | ISO 14635-1:2000 |
| | Standard test method for evaluating the scuffing load capacity of oils (FZG visual method) | ASTM D 5182 |
| FZG high-EP gear-test rig | Standard test method for determining the load-carrying capacity of high-EP oils using the FZG gear-test rig | FVA information sheet no. 243/5 rev. |
| | FZG scuffing load-carrying capacity test for high-EP oils | CEC L-84-A-02 |
| | FZG step load test A10/16.6R/120 for relative scuffing load-carrying capacity of high-EP oils | ISO 14635-2:2004 |

**Tab. 19.7** Continued.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| FZG L-42 hypoid gear-test rig | Load-carrying capacity of hypoid gear lubricants (test described in Ref. [19.12]) | FZG-L-42 |
| FZG wear gear-test rig | Standard test method for evaluating the wear characteristics of tractor hydraulic fluids | ASTM D 4998 |
| | Method to assess the wear characteristics of lubricants – FZG test method C/0.05/90:120/10 | DGMK report no. 377-1 |
| FZG micro-pitting gear-test rig | Standard test method for determining the lubricant's effect on the formation of micro-pitting – test method GF-C/8.3/90 | FVA information sheet no. 54/I-IV |
| | DGMK-FZG micro-pitting short test GFKT-C/8.3/90 | DGMK-FVA project no. 259/4 |
| FZG pitting gear-test rig | Standard test method for determining the lubricant's effect on the life cycle of pitting in load stage test: PT-C/9/90, PT-C/10/90 | FVA information sheet no. 2/IV |
| | Load-collective test: PT-C/LLS/90, PT-C/HLS/90 | No. 371 |
| | development of a practice-relevant pitting test | |
| FZG gear efficiency gear-test rig | Standard test method for determining the lubricant's effect on friction coefficients using the FZG gear-test rig | FVA information sheet no. 345/1 |
| | Comparison of gear efficiency test methods between VW-PV-1456 and FVA info sheet 345 | FVA worksheet no. 345/3 |
| | Standard test method for testing the effect on efficiency of gear lubrication oils – VW gear set | VW-PV-1456 |
| | Ecotrans method – assessment of the ability of lubricants to reduce friction losses in transmission | GFC T 014 T 85 |
| FZG gear-test rig with oil loop for the synthetic oil ageing procedure | Simulation of fill-for-life lubrication using the FZG gear-test rig oil ageing procedure (test described in Refs [19.13] and [19.14]) | FVA worksheet no. 357 |
| Axle gear | L-20 high-torque test | CRC L-20[a)] |
| DANA model 30 | L-33-1 moisture corrosion test | CRC L-33 |
| Dodge (3/4 ton) | L-37 high-torque test | CRC L-37<br>ASTM D 6121 |
| | L-42 high-speed axle test | CRC L-42 |
| L-60 model two steel spur gear | L-60-1 thermal and oxidative stability test | CRC L-60<br>ASTM D 5704 |
| Vanguard phase I | Performance of hypoid oils – high torque | IP 232/69 (2001)[a)] |
| High speed track | Hypoid oils in axles – Mira high-speed shock | IP 234/69 (2001)[a)] |

a) Obsolete but still in use

### 19.3.1.2 FZG High-EP Tests – Scuffing Load Capacity

Oils with a large additive content, so-called high EP gear oils, for example hyoid gear oils, withstand the standard FZG test procedure for lubricating oils. FVA information sheet no. 243/5 rev. and CEC test method L-84-A-02 describe mechanical test procedures for lubricating oils, primarily for use in highly-loaded gear systems with and without hypoid offsets. Three procedures have been defined in detail by the FVA [19.15]:

- the load stage test – procedure A10/16.6R/90,
- the shock test – procedure S-A10/16.6R/90, and
- the shock test at increased oil temperature – procedure S-A10/16.6R/110

All tests are run under enhanced operating conditions. The rig is driven by an electric motor running at double speed of 2880 rpm driving the gear wheel shaft. The direction of the drive is the reverse of that of the standard test method A/8.3/90 giving a corresponding pitch line velocity of 16.6 m $s^{-1}$ with a driving wheel (16.6R). Further aggravating test conditions are use of A-type test gears with a pinion face width of 10 mm (A10) and a required starting temperature of 90 or 110 °C in the oil sump (/90 or /110) and visual assessment stipulated for these procedures.

The test S-A10/16.6R/110 (shock test) can be used as a screening test for the CRC L-42 test [19.12]. To evaluate the oil's load-carrying capacity for API GL 5 it is necessary to compare the results for the candidate oil with the results from ASTM reference oil no. 114-1. The ASTM reference oils can clearly be separated by approximately 1.5 load stages. The pass reference oil no. 114-1 does not fail (LS > 10); fail reference oil no. 112-2 fails 100 % at load stage 9. Candidate oils meet or exceed the scuffing requirement of API GL 5 when the scuffing load stage is higher than that of ASTM reference oil no. 114-1.

### 19.3.1.3 FZG Pitting Tests

Pitting is a form of fatigue failure which occurs on sliding rolling contact surfaces. Lubricants consisting of base oil and additives affect the pitting load capacity of gears. The method of calculation of pitting load capacity according to DIN 3990 [19.11] considers, with the flank roughness and the tangential velocity, only the kinematic viscosity of the lubricant as a lubrication-relevant affecting condition. The actual performance of the lubricant with regard to the pitting load capacity can be experimentally determined by the "pitting test" method [19.16]. For automotive applications load-spectrum testing is possible at low (PT C/LLS/90) and high (PT C/HLS/90) loads specified by Ref. [19.17]. The "practice relevant pitting test" has proved to be an appropriate method for testing pitting load capacity with regard to practical relevance, test duration, and reproducibility of the results [19.18]. Depending on the application profile the test method is defined as a single-stage test (for lubricant developers) and as an extended application test (for lubricant users). It is recommended that helical gears are mounted in the slave gearbox. Lubrication is implemented as temperature-controlled splash lubrication. Every test run is performed with fresh oil with the oil level at the middle of the shafts. Gears of type C-PTX are specified in Ref. [19.19]. These gears are manufactured by ZF Friedrichshafen in a large production batch within the specified tolerances. The results of the test procedures require at least three or more tests under identical conditions to determine a 50 % failure probability of fatigue life using Weibull statistics [19.20]. Two procedures have been defined in detail by the FVA.

- pitting test PTX C/10/90 – single stage test, load stage 10 at an oil sump temperature of 90 °C
- pitting test PTX C/SNC/90 – application test as an extension of the single stage test PTX C/10/90 with additional test runs of the lubricant at load stage 9 or load stage 11 depending on the result of the previous stage test; the oil sump temperature is 90 °C.

For special tests at different temperatures the test designations for both procedures are modified appropriately.

#### 19.3.1.4 **FZG Micro-Pitting Tests**

Use of high-performance gearbox lubricants with EP additives dramatically reduces scuffing wear in gears but some of these additives increase the likelihood of micro-pitting. Micro-pitting or "grey staining" is increasingly being observed by industry in surface hardened (case carburized) gears. Micro-pitting cracks usually grow on planes inclined to the surface plane at angles typically between 30 and 70°, depending on the position of the micro-pitting on the gear tooth, details of the lubrication, and gear design. After some growth (5–10 mm for micro-pitting, much larger for macro-pitting) the crack plane is found to be parallel to the surface. In the past there was a tendency to regard it as a secondary wear problem and much more attention was focused on more macroscopic pitting which occurs as a result of contact fatigue. The introduction of modern clean steels in engineering applications and the use of more highly formulated lubricants to prevent scuffing changed this view, however.

The FVA–FZG micro-pitting test is well established as the standard test method GF-C/9(10)/8.3/90 (FVA information sheet no. 54/I–IV) for evaluating the micro-pitting characteristics of lubricants used in gear drives [19.21]. The FZG micro-pitting test consists of two parts – a load-stage test then an endurance test. In the load-stage test the ability of the gear–lubricant tribological system to resist micro-pitting is determined under specified operating conditions (lubricant temperature, circumferential speed) in the form of a failure load stage. The endurance test provides information about the progress of the damage after a large number of load cycles. This test is conducted at relatively high loads (load stages 5 to 10). In this test gears (C-GF-type) with special surface roughness ($Ra \geq \pm 0.5\ \mu m$) are used [19.22]. This greater surface roughness facilitates the formation of micro-pitting on the test gears [19.23]. This test method provides precise results but at relatively high cost and is quite time-consuming (up to 520 h).

This FVA–FZG micro-pitting test has been supplemented by a standardized short-test method that enables classification of candidate lubricants in a manner analogous to the FVA–FZG micro-pitting test [19.24]. Within the scope of the research project (FVA no. 259/4) the DGMK-FZG micro-pitting short test

GFKT-C/8.3/90 was developed and tested with lubricants whose classification in the standard FVA–FZG micro-pitting test is well known. The new DGMK-FZG micro-pitting short test categorizes candidate lubricants into classes of micro-pitting load capacity analogous to the FVA–FZG micro-pitting test. These classes correlate well with the classes of the FVA–FZG micro-pitting test. The correlation between the average maximum profile deviation after running the DGMK-FZG micro-pitting short test and the failure-load stage of the FVA–FZG micro-pitting test is good. The DGMK-FZG micro-pitting short test is thus regarded as a standardized short-test method suitable for differentiation of the micro-pitting load capacity of different candidate lubricants.

### 19.3.1.5 FZG Wear Tests

For maximum energy savings low-viscosity lubricants are frequently used. Greater transmitted power leads to higher temperatures and thus thinner lubricating films. These tendencies increase the probability of failure in gear contacts at low speeds, because of wear. New test methods on modified FZG gear-test rigs have been developed to evaluate the load-carrying capacity of gear lubricants. For low-speed conditions a wear test using C-type gears at low pitch line velocity of 0.05 m $s^{-1}$ (7.5 rpm) and two different temperatures, 90 and 120 °C, has been developed and applied to many different lubricants to assess response to different additives at high loading of load stage 12 (C/0.05/90:120/12; DGMK project no. 377-1; Table 19.8 and Ref. [19.25]). Under such conditions the lubricant's wear-protective additives are of particular importance.

**Tab. 19.8** FZG/DGMK project no. 377-1 wear load capacity test.

| ***Test procedure of the FZG/DGMK wear load capacity test*** | | | |
|---|---|---|---|
| ***Weight loss*** | ***Test conditions*** | ***Duration*** | ***Load cycles on the test wheel*** |
| 1st test stage | C/0.05/90/12 | 2 × 24 h | 2 × 12 500 |
| 2nd test stage | C/0.05/120/12 | 2 × 24 h | 2 × 12 500 |
| 3rd test stage | C/0.05/90/12<br>C/0.57/90/12<br>C/0.05/120/12 | 1 × 48 h | 1 × 25 000<br>1 × 250 000<br>1 × 25 000 |
| ***Test procedure of the shortened FZG/DGMK wear load capacity test*** | | | |
| ***Weight loss*** | ***Test conditions*** | ***Duration*** | ***Load cycles on the test wheel*** |
| 1st test stage | C/0.05/90/12 | 2 × 20 h | 2 × 10 400 |
| 2nd test stage | C/0.57/90/12 | 1 × 20 h | 120 000 |
| 3rd test stage | C/0.57/120/12 | 1 × 20 h | 120 000 |

The test method according to ASTM D 4998 also facilitates identification of the wear-protective capacity of lubricants, by use of the FZG gear-test rig. Test method A/0.57/120/10 is also known as the Chevron test. The pitch line velocity is 0.57 m $s^{-1}$ (150 rpm) using A-type gears instead of C-type. These wear-intensive operational conditions have been developed to examine the long-term wear-protection capacity of universal gear-lubrication oils for tractors (UTTO). By modification of the ASTM test method mentioned above, this method has been extended to 60 h duration as a more precise test of vehicle, industrial, and hydraulic oils. As a result of gravimetric determination of the weight loss of the test gear set after 20, 40, and 60h, the running-in and long-term wear of lubrication oils can be assessed satisfactorily.

### 19.3.1.6 **FZG Gear-efficiency Tests**

The major targets of transmission design today are higher efficiency, higher torque capacity, and reduced size. Increasingly smaller transmissions with higher torque lead to higher operating temperatures. The friction in the transmission is responsible for temperature increase and efficiency losses, and reducing friction is the main means of improving efficiency and keeping the operating temperature low. The main features of transmission design to increase efficiency are reduction of friction of the bearings and the rotary shaft seals, reduction of sliding of the gear flanks, and reduction of splashing and pumping of the lubricant. Losses in a gear system are classified as load-dependent and speed-dependent losses, for example churning or sealing losses. To identify and compare the effects of lubricants on gear efficiency test method VW-PV-1454 (Table 19.1) [19.26], using a modified four-ball apparatus adapter, and test method VW-PV-1456 [19.27–19.29], using a modified FZG gear-test rig, have proven successful for lubricating oils and additives.

All FZG test methods measure and compare the torque loss which the driving motor imports into the mechanical power circuit to sustain the rotational movement during the operating conditions. Another torque meter must be installed behind the input shaft of the driving motor. The same gear-set types are used in the slave gearbox and the test gearbox for these tests, and both gear boxes are filled with the same oil at an identical level. By use of this method the total losses in the no-load and load modes of operation are measured to determine their dependence on gear type, oil and additive formulation, viscosity grade, oil filling level, rotational speed, and load. Both test gears have, furthermore, been equipped with an adjustable heating system which maintain equal oil sump temperature in each gearbox [19.30, 19.31].

The advantage of the above-mentioned procedure, in comparison with the older so-called Ecotrans test method (GFC-T-014-T-85) [19.32], is more precise measurement of torque loss when using a torque meter. In the Ecotrans test method the friction torque in the mechanical power circuit is calculated by measurement of the electrical input power of the driving motor. The rotational speed and load-dependent power input of the often different systems of electrical power input lead to significant inaccuracies, however. An excellent comparison of the common FZG gear-efficiency methods currently in use is available elsewhere [19.31].

#### 19.3.1.7 **FZG Synthetic Oil Ageing Tests**

Oil ageing of lubricants can be simulated in a modified FZG back-to-back gear-test rig [19.33]. A schematic diagram of the oil ageing test arrangement is given in Fig. 19.10. For applications with long oil-drain intervals the oil ageing test was developed using C-type gears under different load, speed, and temperature conditions. Lubricants are exposed to conditions of load and speed of bearings and gears encountered in standard transmissions, and in a spray lubrication device, to elevated oil temperatures of 110 to 130 °C for enhanced thermal degradation over the runtime. The oil-ageing properties and their effect on performance under these high-temperature conditions and in other component tests after ageing are correlated with those of reference oils. The different oil application-related ageing test methods are described elsewhere [19.34]. After the shortened duration of oil ageing in the FZG rig (Fig. 19.11) the oil is tested to determine the behavior of critical components. Different component test results for commercial brands are discussed elsewhere [19.13, 19.14, 19.35]. Further correlations of automatic transmission fluids synthetically aged with FZG test methods to evaluate wet clutch friction stability compared with results from field trials are also given elsewhere [19.36]. By use of these test results oil-specific and application-specific lifetime limits, i.e. "logarithmic oil temperature/ageing time curves" can be derived, enabling determination of the effect of temperature on lifetime for each of the critical component.

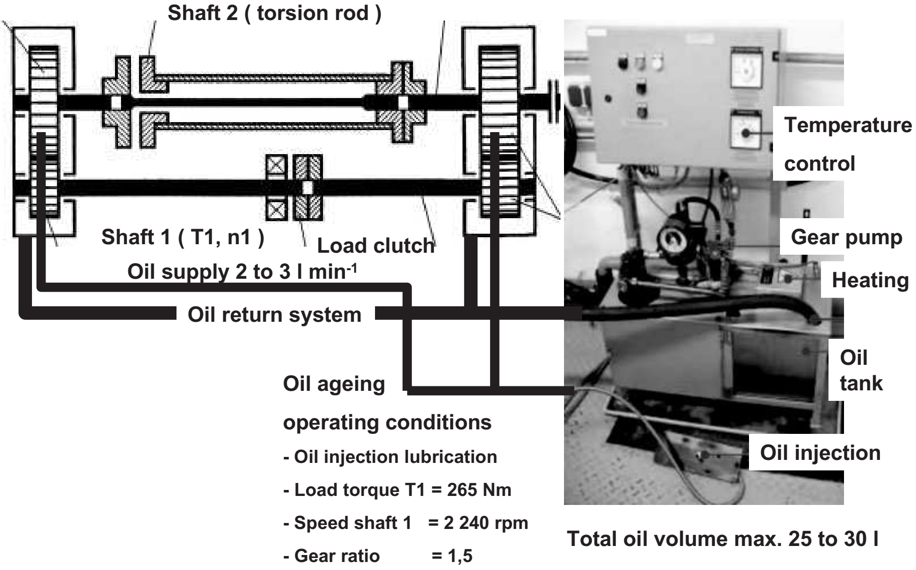
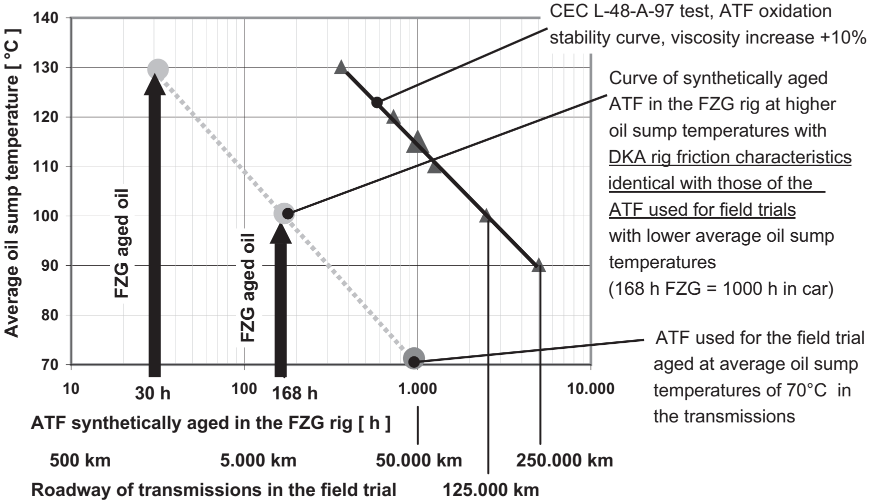


**Fig. 19.10** FZG back-to-back gear-test rig with modified oil ageing set-up.



CEC L-48-A-97 test, ATF oxidation stability curve, viscosity increase +10%

Curve of synthetically aged ATF in the FZG rig at higher oil sump temperatures with DKA rig friction characteristics identical with those of the ATF used for field trials with lower average oil sump temperatures (168 h FZG = 1000 h in car)

ATF used for the field trial aged at average oil sump temperatures of 70°C in the transmissions

**Fig. 19.11** Shortened duration of oil ageing in the modified FZG rig.

When using the FZG oil-ageing procedure the analytical figures must be taken as an indication of oil modifications that can result in different possible effects on lubricant performance and the capacity in the different components. These effects are strongly dependent on the oil type and the additives. For a specific investigation of the effects of extended oil drain intervals the oil-specific and application-specific lifetime-limiting criterion must be known, e.g. from damage or from long term investigation of the application. Aged samples from the application run under known temperature and duration conditions are also necessary for calibrating the ageing conditions in the test rig described elsewhere [19.36].

## 19.4
## Performance Tests for Roller Bearing Applications

### 19.4.1
### FAG Roller Bearing Test Apparatus FE8

The FAG Roller Bearing Test Apparatus FE8 is one of the most versatile test rigs for lubricants. The machine itself is specified in DIN 51819 part 1 [19.37, 19.38]. The method specified serves to test lubricants such as lubricating oils part 3 and greases of NLGI classes 1 to 4 (in accordance with DIN 51818 part 2 [19.39]) to assess the effect which these lubricants have under service conditions on the frictional behavior and wear of angular contact groove ball bearings, tapered roller bearings and cylindrical thrust roller bearings (cylindrical axial roller bearings). A schematic diagram of the test head and drive unit of the FE8 is shown in Fig. 19.12. Two test bearings are mounted in the FE8 test head, subjected to a given axial bearing load (thrust load equivalent to 10, 20, 50, 80, or 100 kN), operated at a given speed (variable from 7.5 to 3000 rpm), and kept, by means of a heating and/or cooling system, at any required operating temperature between ambient temperature and 200 °C. The operating temperature must be a multiple of 10 °C. Newer FE8 machines are equipped with an additional cooling device that enables use of test bearing temperatures of –20 °C. If testing oils or greases with external heating, the heating system is switched on after a short running-in time when the test is started. Grease testing is continued until a loading time of 500 h has elapsed or until lubrication of the bearings becomes inadequate and the test bearing friction torque assumes the switch-off torque for at least 10 s, when the apparatus will switch off automatically. For operations beyond a loading time of 500 h, the loss in mass of the rolling elements and of the bearing cages (wear) is used to assess the anti-wear characteristics of the grease. If testing lubricating oils, an external oil circulation system is added. A defined oil flow rate (150 to 250 mL min$^{-1}$, total oil volume 5 L) is specified for a bearing oil inlet temperature of 80 to 120 °C. The duration of the oil-test procedure is 80 h using the thrust cylindrical roller bearings (FE8 wear test).

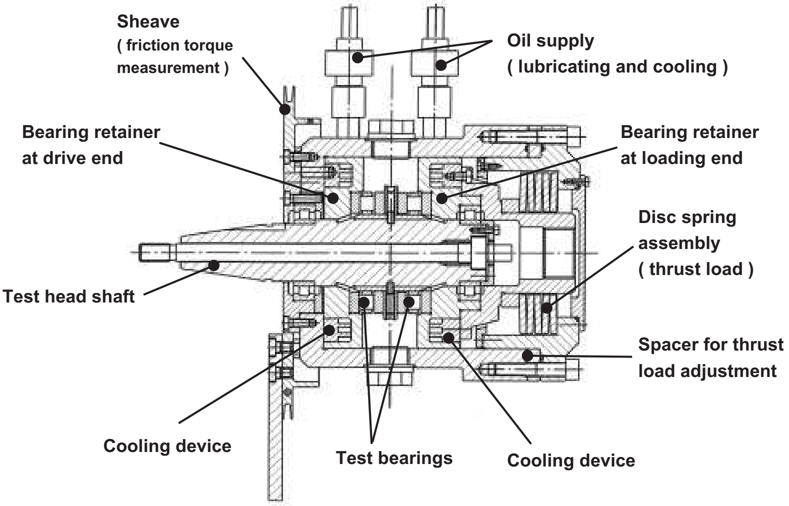


**Fig. 19.12** Schematic diagram of the FE8 test head (oil test).

Bearing geometries of 60 mm (inner race diameter) have sufficient bearing size and mass to determine lubricant-dependent mass losses of roller elements, bearing races, and the cage with sufficiently precise values for repeatability and reproducibility. Variation of cage material (e.g. polyamide, brass, or steel) even enables determination of the lubricant-dependent wear protection of different materials on steel contacts for greases and lubricating oils.

The current FE8 roller bearing test apparatus is also suitable for testing bearing fatigue life (pitting load capacity) in mixed-lubrication regimes. Investigations have been reported elsewhere [19.40]. FVA project no. 504 [19.41] evaluates new test procedures reported elsewhere [19.42] with modified test heads. The most important common test standards and test machines for performance tests of lubricated bearings are summarized in Table 19.9.

**Tab. 19.9** Test standards for roller bearing applications.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| FAG roller bearing test apparatus FE8 | Test using the FAG roller bearing testing apparatus FE8 | |
| | General working principles | DIN 51819 |
| | Testing of lubricating greases | DIN 51819, Part 1 |
| | Testing of lubricating fluids | DIN 51819, Part 2 |
| | | DIN 51819, Part 3 |
| | ZF bearing pitting test | ZF 702 232/2003 |
| | Determination of oil influence on pitting load capacity of cylinder roller thrust bearings on mixed-lubrication | VW TL52512/2005<br>VW TL52182/2005 |
| FAG roller bearing test apparatus FE9 | Test using the FAG roller bearing testing apparatus FE9 | |
| | General working principles | DIN 51821 |
| | Test method A/1500/6000 | DIN 51821, Part 1 |
| | | DIN 51821, Part 2 |
| FAG spindle bearing test apparatus WS 22 | Test using WS22 spindle bearing testing apparatus for lubricating grease | FAG specification |
| SKF grease test machine ROF | Testing of lubrication greases at high temperature and high speeds | Test method - TS 14 |
| SKF roller bearing grease test machine R2F | Testing of lubricants using SKF-R2F roller bearing grease test machine | E-DIN 51806 |
| | General working principles | E-DIN 51806, Part 1 |
| | Test under heating condition | E-DIN 51806, Part 2 |
| Oscillating test bench | Standard test method for fretting wear protection by lubricating greases | ASTM D 4170 |
| Rolling bearing performance test apparatus | Determination of rolling bearing performance of lubricating grease | IP 168/95 |
| FAG noise test apparatus | Noise inspection of lubricating greases (computer-aided) | FAG-QV3.102FB MGG 11 |
| SKF noise test apparatus | Noise inspection of lubricating greases (computer-aided) | SKF specification MVH 90 B |

### 19.4.2
### FAG Roller Bearing Test Apparatus FE9

The FAG roller bearing test apparatus FE9 has been developed for testing greases under application-relevant operating conditions. The service life of the grease is determined from the operating temperature, the load, and the rotational speed of the test bearing. A schematic diagram of the FE9 test machine and cap assembly are shown in Fig. 19.13.

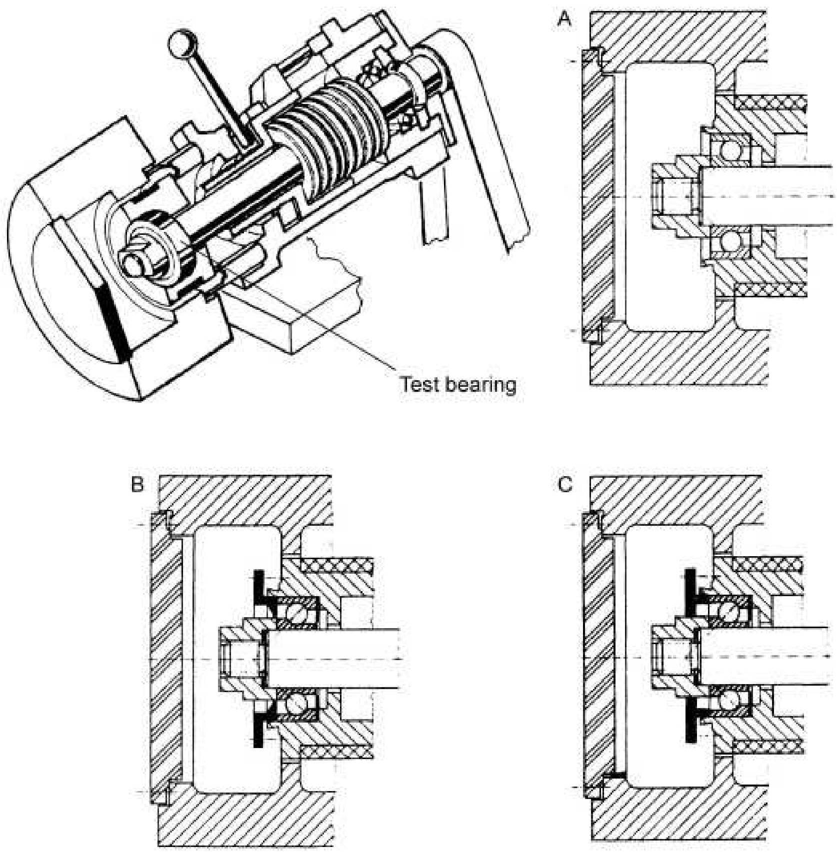


**Fig. 19.13** Schematic diagram of the FE9 test head (grease test) and cap assembly. A. Open cap assembly, B. Covered cap without grease reservoir assembly, C. Covered cap with grease reservoir assembly.

The machine is specified in DIN 51821 part 1 [19.37, 19.38]. The test method A/1500/6000 has been standardized in DIN 51821 part 2 (Table 19.9). The angular contact ball-bearing in the test head is filled with approximately 2 mL grease. The test bearing is loaded with a thrust load (axial test force) of 1500 N at a speed of 6000 rpm. The test temperature can be selected within the range 120 to 200 °C (maximum) and is applied to the test bearings by means of heating elements. These high temperatures of the tested greased bearing facilitate oxidation of the lubricant. Failure of a lubricant in the test bearing results in an increase in the required power input of the drive motor. Exceeding a given limit results in automatic shut-off of one test head, which means the duration of one test run has been determined. For one FE9 test, statistical evaluation is performed for five test runs on five different test heads, all operated under identical conditions; five (slightly) different test results (run-times) are used for statistical determination of the 50 % probability of failure by the method of Weibull [19.20]. The average duration is regarded as the service life of the grease. The test conditions are regarded as having been selected correctly if 50 % probability of failure occurs after 100 to 200 h. After >300 h the test run usually stops automatically.

In a deviation from the standard, the speed can be reduced to 3000 rpm and the thrust load enhanced to 3000 or 4500 N to perform tests under intensified mixed-lubricated conditions. In practice, under all operating conditions it is accepted that at an operating temperature >100 °C, increasing the operating temperature by 10 to 15 degrees halves the service life of the grease in roller bearings. It is not useful to run wear or fatigue endurance tests using the FE9 machine, because the size of the test bearings is too small for gravimetric determination of wear or pitting and this leads to a relatively large spread of test results. Under FE9 test conditions (application-related operating conditions), the nominal bearing life of the loaded test bearing (calculated and statistically validated in terms of fatigue life in catalogues of bearing manufacturers) is, furthermore, much longer than the service life of greases measured and determined in the FE9 apparatus.

## 19.5
## Performance Tests for Synchronizer Applications

### 19.5.1
### Area of Application

Manual transmissions are a common source of complaint from car drivers because of poor or hard shifting. For that reason, OEM pays increasing attention to the friction properties of the synchronizing elements. Component harmonization and mass production imply that the same gearbox model must accommodate a wide range of different driving patterns. In the field there are large variations between minimum and maximum operating conditions and, therefore, different expectations of shifting characteristics. More compact vehicles with smaller engines and better fuel economy require a wider range of gear ratios. Larger gears are used for the lower ratios, increasing the demand for synchronizers operating between shifts. There is more inertia to be stopped but not much more space to accommodate the synchronizer. Hence the development of double and triple cone synchronizers, multiplying the number of friction surfaces, enabling design to be kept compact. The demand on friction is, however, increased, because there is more energy to dissipate in a tighter volume [19.43–19.45].

### 19.5.2
### Function of the Synchronizer

Synchronizer components are housed in the gearbox and its role is vital in the shifting process in a synchronized gearbox. It is used to accelerate the gear to be engaged so that it can quietly mesh with the driving gear when rotating speeds are synchronized. Synchronizers can be described as mechanical brakes. They are commonly conical in shape and have grooves to break the oil film, to provide some cooling, and to furnish a complicated surface pattern for best braking grip. Many different designs have been developed over the years to enable smooth and rapid gear engagement. Several factors potentially play a role in their performance, including machining and manufacturing processes. Synchronizer material is certainly an important factor in providing the correct friction properties in constant evolution. Its composition, component geometry and manufacturing conditions are jealously guarded secrets from all the different suppliers on the market. Typical shifting time ranges from a few tenths of a second in a passenger car to less than 50 ms in racing vehicles. Comfort is less of a priority in the latter application, which enables use of more direct systems that would otherwise be rejected as too harsh by the average driver. Although the details of the operation are complex, they are totally transparent to the driver, except when the synchronizer does not perform its duty properly, causing gear clashing [19.46]. The customer's expectations increase as rapidly as the technology improves. New solutions must constantly be developed or further enhanced to provide faster and smoother shifting. There is therefore a need for standardized test rigs. Figure 19.14 gives examples of synchronizer systems in current use.


Single and double cone synchronizers

Triple cone synchronizers

**Fig. 19.14** Synchronizer systems currently in use.

### 19.5.3
### Standardized Test Rigs and Test Methods

A very limited range of three standardized rigs, so-called "Synchromesh", for performance evaluation are currently available commercially. These are the μ-comb rigs and the FZG SSP180 rig. Testing conditions with this equipment always try to match today's gearbox operating environment, particularly in terms of dynamic performance and performance optimization. For both types of rig the test components can either be taken out of regular production or can be accurate pre-measured and selected parts. Use of a hardware set of known performance (for example the Audi, New Venture Gear, ZF or DaimlerChrysler synchronizers) enables determination of the effects of different lubricants on synchronizer endurance for manual and dual-clutch gearboxes in automotive applications defined by wear and change in the coefficient of friction. As fluid formulations change to address new or greater performance requirements in other areas of the transmission, information provided by these test procedures will enable lubricant formulators to determine whether synchronizer performance will remain acceptable.

Subjecting these units to thousands of engagements serves to test synchronizer durability. Different types of synchronizer and friction material are used by the OEMs; area of application currently affect service life. It is not possible to create one general standard test procedure for approval purposes. OEMs require a common test procedure which enables understanding of oil manufacturers' test results. The test procedures given by CEC L-66-T-99 for the SSP180 are suitable for use as "screening tests" for this purpose [19.47]. Different types of synchronizer can be used and a variety of procedures are available depending on the application (passenger car or heavy duty vehicles, and/or materials, summarized in Table 19.10). Gear clashing, synchronizer wear, or synchronizer seizure are the main types of failure observed for this equipment, in agreement with most field experience [19.48].

**Tab. 19.10** Test standards and synchronizer materials for oil tests.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| μ-Comb synchronizer testing machine | Audi ML 285 brass, single cone, passenger car<br>Audi ML 310 molybdenum, single cone, passenger car<br>Audi ML 310 Carbon 5010, double cone, passenger car<br>VW DK 67, HS45, double cone, passenger car<br>ZF TK 89, triple cone, HS45 | ZF, Audi, VW, New Venture Gear, Getrag, BMW, Ford, GM, Fiat OEM Specifications |
| FZG SSP180 synchronizer testing machine | AUDI B 80 brass, single cone, passenger car<br>ZF BK 117 HS45, single cone, light truck<br>ZF BK 119 molybdenum, single cone, light truck<br>DC AK 177 molybdenum, single cone, HD Appl. | CEC L-66-T-99 |

#### 19.5.3.1 **μ-Comb Synchronizer Testing Machine**

The μ-comb synchronizer testing machine was developed by the Hörbiger Company in Schongau (Germany) [19.49]. This machine uses a single synchronizer set and operates up-shift during testing. To keep operating conditions as close as possible to the real gearboxes, the rotation speed changes direction every 200 revolutions. Different types of synchronizer can be used and different procedures are available depending on the application (passenger car or heavy-duty vehicles, and/or materi-

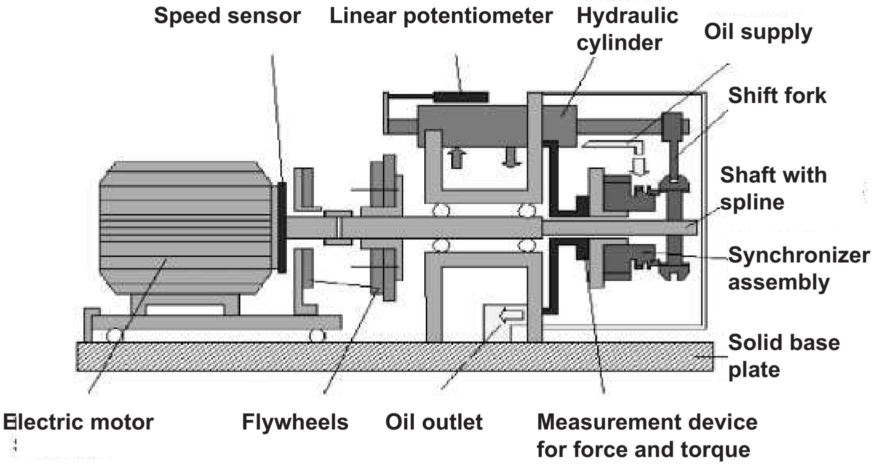


**Fig. 19.15** Schematic diagram of the μ-comb synchronizer testing machine.

als). The rig characteristics are well suited for measurement and development of double and triple cone synchronizers. The most widely recognized version is the small μ-comb option. A bigger version of this testing machine, the μ-comb truck, is used for testing synchronizers of commercial vehicles. A schematic diagram of the operating principle is shown in Fig. 19.15. The main technical data are given in Table 19.11.

**Tab. 19.11** μ-Comb synchronizer testing machines.

| *Technical data* | *μ-Comb* | *μ-Comb truck* |
|---|---|---|
| Synchronizers | 50–120 mm | 100–200 mm |
| Speed difference | 0–4500 rpm | –3500–3500 rpm |
| Axial force | 0–5000 N | 0–5000N |
| Inertia | 0.013–0.036 kg m$^{-2}$ | 0.015–2.5 kg m$^{-2}$ |
| Cycle time | >3 s | >3 s |
| Oil temperature | Room temperature to 120 °C | Room temperature to 120 °C |
| Oil flow rate | 0– 3.5 L min$^{-1}$ | 0 – 3.5 L min$^{-1}$ |
| Total oil volume | 4–6 L | 4–6 L |

This machine uses a single synchronizer set and operates up-shift during testing. The test machine consists of an electric motor, flywheels, actuating hydraulics, an oil heating and circulation system, and a test box. The flywheels are connected to the electric motor via a clutch to ensure a constant and stable source of speed. The flywheels are the load the synchronizers bring to a shift to "speed zero" position to be accelerated after this shifting back to a constant speed by the motor again. This is accomplished by the ring-and-cone synchronizers mounted in the test box. The actuating hydraulics move a shift fork that engages and disengages the unit. During shifting, heated lubricant is sprayed on to the synchronizer unit. Subjecting these units to thousands of engagements serves to test synchronizer durability and friction.

### 19.5.3.2 FZG SSP180 Synchronizer Testing Machine

The most widely recognized tool is the FZG SSP180 synchronizer testing machine, developed by the Hurth Company and now built, distributed, and supported by the ZF in Passau (Germany) [19.50]. This machine uses a complete synchronizer set and operates full up and down-shift during testing. The test machine consists of an electric motor, two flywheels, actuating hydraulics, an oil heating and circulation system, and a test box. The large main flywheel is connected to the electric motor via a belt-and-pulley combination to ensure a constant and stable source of speed. The small flywheel is the load that the synchronizers either bring to zero speed (shift to “A” position) or accelerate to a constant speed (shift to “B” position). This is accomplished by the two ring-and-cone synchronizers mounted in the test box. The rear unit accelerates the load flywheel to synchronous speed while the forward unit decelerates the flywheel to zero speed. The actuating hydraulic moves a shift fork that engages one unit and disengages the other. During shifting, heated lubricant is sprayed on to both synchronizer units.

A schematic diagram of the FZG SSP180 machine is shown in Fig. 19.16. Typical synchronizer engagement traces showing individual shift performance data are illustrated in Fig. 19.17. Data from the engagements are plotted during the course of the test and the trends observed show whether or not performance is degrading. Technical data for the machine are listed in Table 19.12.

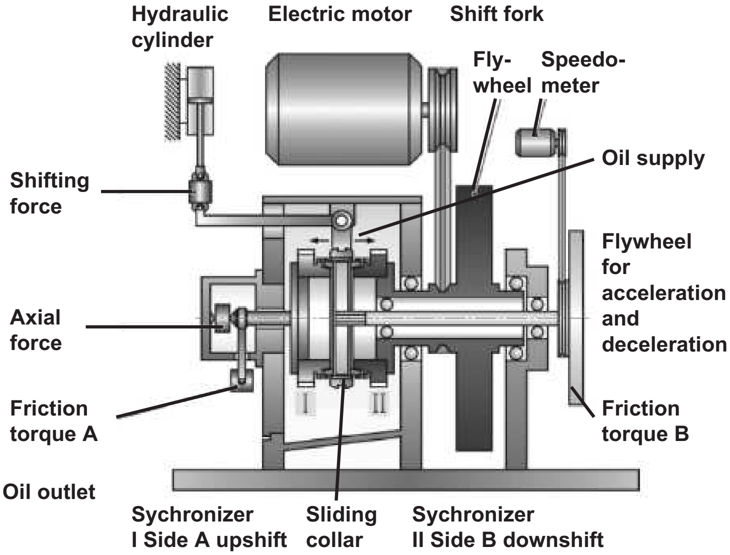


**Fig. 19.16** Schematic of the FZG SSP180 synchronizer testing machine.

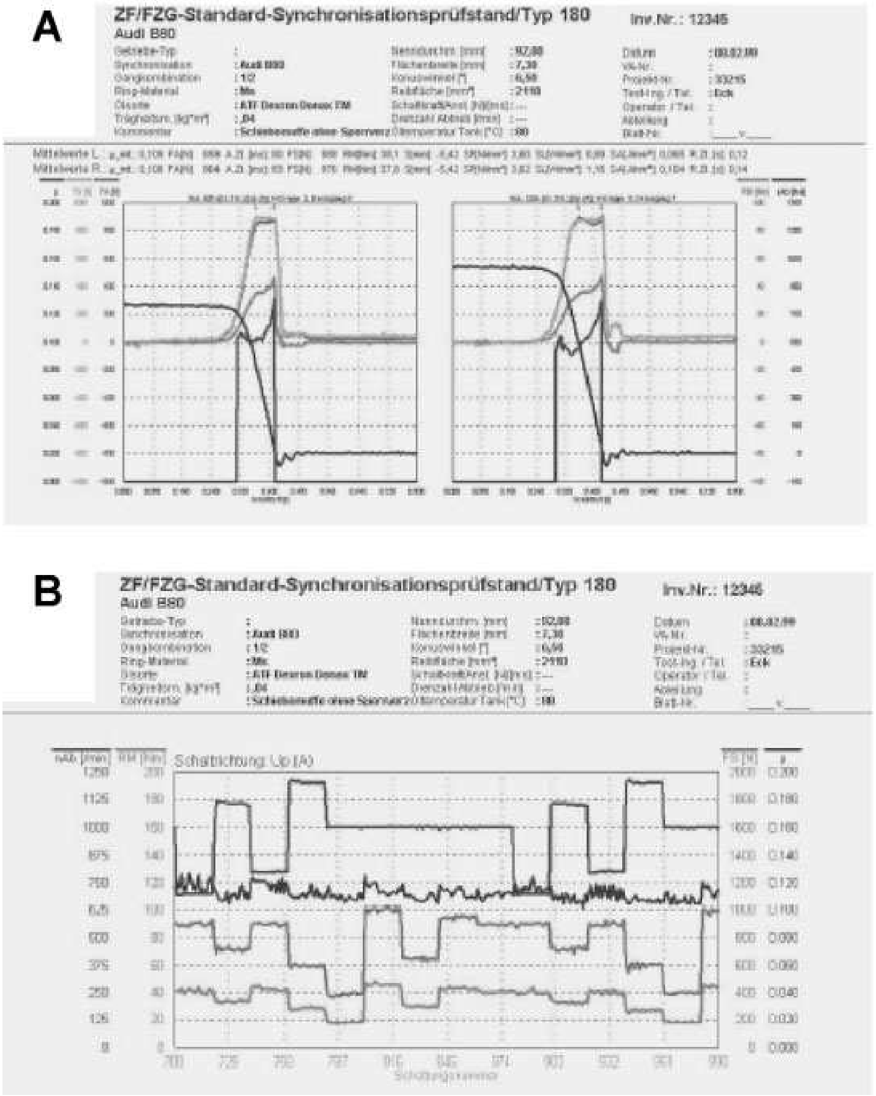


**Fig. 19.17** Typical synchronizer engagement traces showing individual shift performance data. A. Single shifting; B. Trend data.

**Tab. 19.12** FZG SSP180 synchronizer testing machine.

| *Technical data* | *FZG SSP180* |
|---|---|
| Synchronizers | 40–200 mm |
| Speed difference | 0–5000 rpm |
| Axial force | to 4000 N |
| Inertia | 0.04 – 0.12 kg $m^{-2}$ |
| Cycle time | >4 s |
| Oil temperature | 10–120 °C |
| Oil flow rate | 2–9 L $min^{-1}$ |
| Oil volume | 8–10 L |

## 19.6
## Performance Tests for Automatic Transmissions

### 19.6.1
### Area of Application

The testing of fluids for application in automatic and semi-automatic transmissions or in axle gears with limited slip differentials is another aspect of the study of friction and friction materials, especially of friction disks. To ensure today's requirement of smooth gear shifts and braking with limited slip differentials or wet brakes, self-excited vibration or shudder, which may occur, must be avoided. The wet friction coefficients and the constant wet friction durability of the friction characteristics in the sliding contacts of the rotating friction disks are important aspects of Driveline's effective and comfortable torque-transfer process.

The most common use of these friction disks is in the wet clutches of automatic transmissions (AT) used in automobiles and trucks. The disks are also used in all other applications that include wet brakes (speed-controlled brakes), lock-up clutches, continuously slipping torque converter clutches (CSTCC), electronically-controlled converter clutches (ECTCC), launch clutches in motor cycles, and limited slip differentials. Modern all-wheel drives (AWD), which were originally used to improve the off-road capacity of military and other off-road vehicles, have friction disks. In the early eighties, a new market for all-wheel drive performance vehicles emerged with the introduction of the Audi Quattro. The working principle of transmitting torque by use of friction disks is always the same. In all these applications where friction disks operate at low sliding velocities for long periods of time it is important to control the friction characteristics. Many test machines have therefore been designed, especially for testing the fluids and their friction characteristics.

### 19.6.2
### Function of Friction Disks and Fluid

The friction disks are basically working under fully lubricated conditions. The configuration most commonly used is a single-disk or multiple-disk wet clutch as

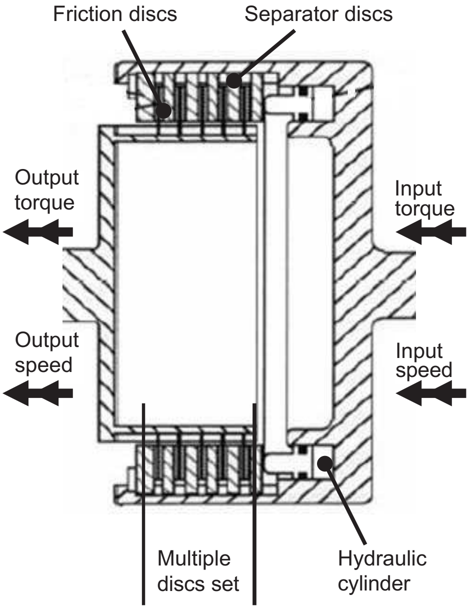


**Fig. 19.18** Schematic diagram of a wet clutch.

shown in Fig. 19.18. This consists of one or more friction disks attached to one shaft fixed by splines. One or more separator disks are similarly connected to the other shaft. When disengaged, the clutch transmits only a small drag torque because of viscous friction, and thus both shafts are free to rotate independently. The clutch is engaged by applying a normal force from the hydraulic cylinder (mechanical or electromechanical actuators are also commonly used), clamping the friction and separator disks together and thereby enabling torque to be transmitted between the input and output shafts. As a result, most research on friction is related to this application. The fluid must cool the disks and maintain their friction characteristics of constant dynamic and static friction coefficients.

Typical friction characteristics for three different fluids, but with identical friction material shown in Fig. 19.19 (similar differences can be seen for a single fluids with different friction materials). To avoid vibrations (commonly referred to as shudder in clutch applications and squeal in brake applications) the relationship between friction and velocity ($\mu$–$v$ relationship) should have a low static coefficient of friction ($\mu_s$) and a higher dynamic coefficient of friction ($\mu_{dyn}$) that increases as the sliding velocity increases [19.51, 19.52]. Oil A will suppress vibrations whereas oils B and C may be susceptible to vibrations because they have a negative slope in some regions. The effect of the relationship between friction and velocity on the anti-shudder performance of wet clutches is well established. The advantage of a positive $\mu$–$v$ slope can easily be shown mathematically from engineering vibration calculations; it should, however, be noted that a positive slope is neither a necessary nor sufficient condition to guarantee friction stability.

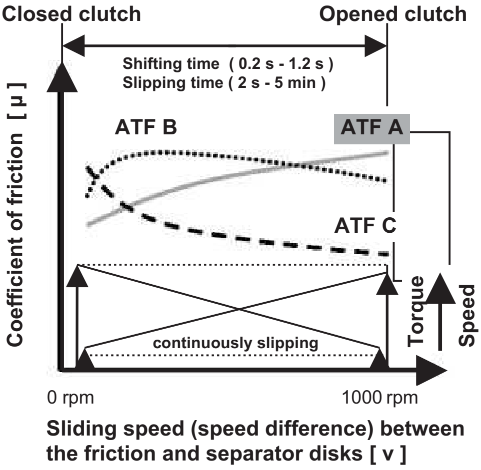


**Fig. 19.19** Schematic diagram of the friction curves of automatic transmission fluids.

### 19.6.3
### Standardized Test Rigs and Test Methods

#### 19.6.3.1 SAE 2 Machine

The SAE 2 machine is probably the most widely used instrument for evaluation of friction performance in North America and Asia Pacific. It has been used for over thirty years [19.53–19.57]. This rig tests a full clutch pack, enabling assessment of the overall performance of lubricant–friction material combinations. The SAE 2 machine can be used in two different ways. In one type of test the large drive motor accelerates the flywheel to 3600 rpm at which point the motor is turned off and the clutch is engaged to stop the flywheel. This procedure is repeated several times a minute for 50 to 100 h to investigate the durability of the fluid. To determine torque capacity the clutch is engaged and the low-speed drive is run at 0.72 or 4.37 rpm. Typical data obtained include the maximum break-away friction and the friction coefficient after some seconds of continuous slip. The applied normal force in the SAE 2 machine is usually well below 10 kN. The JASO M394-95 is a Japanese standard for friction evaluations using the SAE 2 or similar apparatus [19.56]. Researchers have also used modified SAE 2 machines with some success to evaluate friction characteristics at low speeds [19.58] or the friction characteristics of synchronizers.

#### 19.6.3.2 DKA Machine

The DKA Machine operates very similarly to the SAE 2 machine and was developed in Europe [19.9] for research, development, and quality control in the production of additives and finished fluids. A schematic diagram of the DKA machine (ZF friction disk test bench) is given in Fig. 19.20 [19.59]. Technical data for the different types of DKA-1A/1B rig are compared in Table 19.13.

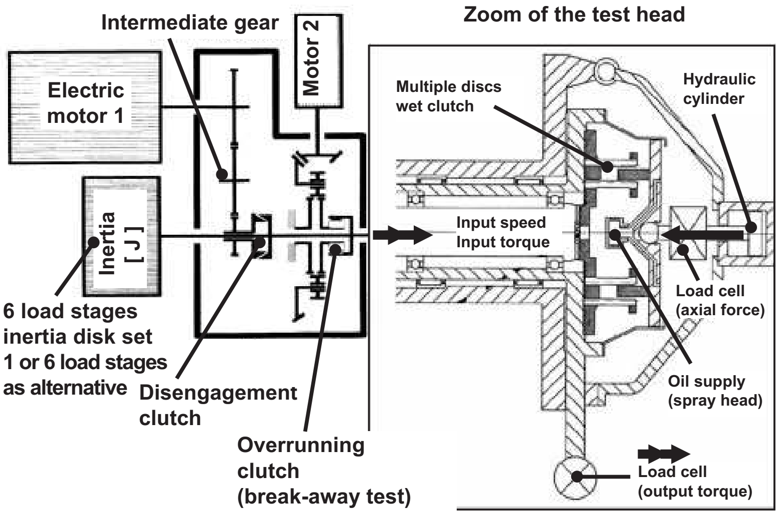


**Fig. 19.20** Schematic diagram of the DKA-1A/1B rig for evaluation of the coefficients of friction of transmission fluids.

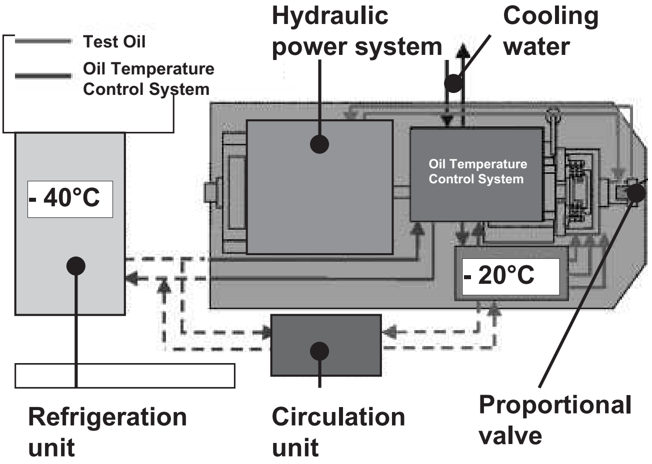
**Tab. 19.13** DKA machine (ZF friction disk test bench).

| *Technical data* | *27 kW DKA-1A* | *50 kW DKA-1B* |
|---|---|---|
| Power supply | 50 kV A | 125 kV A |
| Speed difference | 20–7000 rpm | 20–7000 rpm |
| Break torque | 1500 N m | 2000 N m |
| Break-away torque | 1500 N m | 2500 N m |
| Inertia (var. mass set) | 0.053–0.78 kg $m^{-2}$ | 0.05–1.0 kg $m^{-2}$ |
| Oil temperature | 40–150 °C | 40–150 °C |
| Oil flow rate | 0.2–6.0 L $min^{-1}$ | 0.2–6.0 L $min^{-1}$ |
| Total oil volume | 12–25 L | 12–25 L |

For test runs on DKA machines PC test programs are prepared and control the test bench in automatic mode, including the break-away test where the actuated disk package is accelerated from standstill to a defined break-away speed for measurement of the static friction coefficient. During each test run the friction disks are permanently supplied with a temperature-controlled lubricant. The oil is delivered into the centre of the shaft to simulate the operating conditions found in a vehicle transmission. The friction disks are actuated by means of a servo-cylinder with direct thrust load measuring which enables simulation of the shift pressure characteristics found under actual field conditions. The DKA-1B rig is equipped with two sets of flywheel masses which can be independently connected and disconnected. Measurement accuracy for the DKA machine is also substantially higher than for other systems.

### 19.6.3.3 ZF GK Test Bench 2

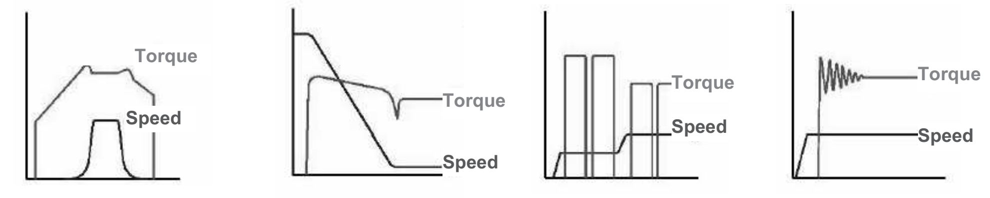
ZF GK test bench 2 is an advanced version of the older ZF GK test bench used for testing wet clutches, torque converters, and their fluids, for example ATF, UTTO, and STOU, engine oils and oils for CVT applications [19.60]. The rig simulates DKA/SAE 2 operation modes enabling limited measurement of the dynamic shifting, quasi-static test and the break-away procedure. The test units are used for research, development, and quality control in production (additive packages in reference oil and finished fluid). Figure 19.21 shows a schematic diagram of the GK rig with the hydraulic system and a separated test oil system enabling oil tempering down to –20 °C using a refrigeration unit for cold-start tests. This compact test oil system has a minimum oil volume of 5 L which enables its use for aged oils from transmissions or from other test rigs or synthetically aged fluids (Sect. 19.3.1.7). The modes of operation of the GK rig are summarized in Fig. 19.22. Figure 19.23 illustrates an example of the change of the friction characteristic of an ATF running a combined oil ageing and speed-controlled opening and closing procedure in a wet clutch on the GK rig. The relationship between friction and velocity (*μ*–*v* relationship) reveals a dramatic decrease of the dynamic coefficient of friction at controlled shifting no. 15 000. This corresponds to a real transmission in a car driving a roadway of 60 000 km under enhanced driving conditions.



**Technical data**

| | |
|---|---|
| **Input power** | **104 kW** |
| **Input speed** | **± 1 rpm - 3.000 rpm** |
| **Torque** | **10 - 1 000 Nm** |
| **Mass inertia** | **0.02 kgm² - 4.5 kgm²** |
| **Pressure controlled** | **0.2 bar - 20.0 bar** |
| **Force controlled** | **0.2 kN - 20 kN** |
| **Oil temperature** | **-20°C - 140°C** |
| **Oil flow rate** | **0.5 l/min - 15.0 l/min** |
| **Total oil volume** | **min. 5 l** |

**Fig. 19.21** Schematic diagram of the ZF GK test bench 2.



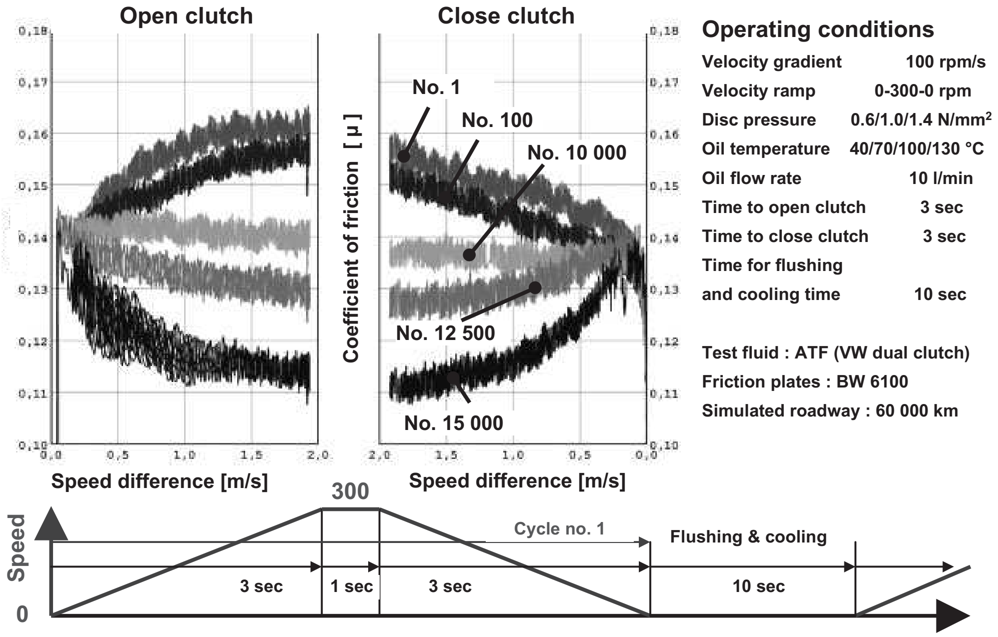
**Opening and closing**
Speed controlled
Torque controlled
Pressure or force controlled

**DKA-operation**
Dynamic shiftings
Quasi-static test
Break-away test

**LVFA-operation**
Determination of
the static friction
coefficient

**( Green- ) Shudder**
Pressure or force
or torque controlled
operating test runs

**Fig. 19.22** Operating modes of the ZF GK test bench 2.



**Operating conditions**

| | |
|---|---|
| Velocity gradient | 100 rpm/s |
| Velocity ramp | 0-300-0 rpm |
| Disc pressure | 0.6/1.0/1.4 N/mm$^2$ |
| Oil temperature | 40/70/100/130 °C |
| Oil flow rate | 10 l/min |
| Time to open clutch | 3 sec |
| Time to close clutch | 3 sec |
| Time for flushing and cooling time | 10 sec |

Test fluid : ATF (VW dual clutch)
Friction plates : BW 6100
Simulated roadway : 60 000 km

**Fig. 19.23** Example of an oil test result run on a ZF GK test bench 2.

## 19.7
## Performance Tests for Continuously Variable Transmissions

### 19.7.1
### Field of Application

A continuously variable transmission (CVT) is a system that enables progressive variation of the transmission ratio by means of a variator. It thus enables selection of an infinite number of ratios between minimum and maximum values. Other transmissions only enable use of a limited set of fixed gear ratios. A manual transmission (MT) usually enables use of five gear ratios, although some gearboxes have six or seven. It is advantageous and more comfortable to use a CVT rather than other transmissions, because the engine will always operate under the optimum conditions and throttle-positions, adapted to varying road conditions and power demands (Sect. 10.4.3.3). With the same car and engine the CVT takes only 75 % of the time of an MT to accelerate to 100 km $h^{-1}$. Perhaps, the most remarkable practical proof of CVT performance was its use in the 800 cV Formula One Canon–Williams–Renault in 1993. Without so much development as the MT version the experimental CVT Formula One was 1 s faster per lap.

### 19.7.2
### Function of Chain, Push-Belt and Fluid

As indicated in the schematic diagram in Fig. 19.24, a continuously variable link chain or push belt in a CVT consists of a first pair of pulleys (input shaft) and a second pair of pulleys (output shaft), each having a fixed pulley, a pulley movable in the axial direction, and a chain or a push belt that is wrapped around these pulleys. The following considerations apply in particular to gears with wrapping elements, for example push belts or link chains, i.e. with a finite number of contacts with the pulleys. In such a CVT the torque is transmitted through friction forces in lubricated contacts between the chain and the cone pulleys. In these circumstances the friction coefficients are in the range $0.07 < \mu < 0.11$, depending on the lubricant and on surface roughness. Thus, very high contact thrust forces are necessary for high torque capacity. These high contact forces deform the pins of the chain links and the pulleys, which should be light compared with the weight of the gears. Because of these deformations, the chain does not wrap around the pulleys in a circular arc, but moves around the pulleys with additional radial motions inwards or outwards.

To these sliding movements in the radial direction a circumferential slip is added; this arises from the longitudinal elasticity of the chain and the changes of the contact radii because of the radial movements. There are also more sliding movements if the ratio is changed. All sliding movements add up to a total slip between the chain and the pulleys. The local friction forces counteract the local sliding movements and cause a change of forces within the chain and thus transmit the torque through the gear. Such relationships for stationary operating states have been very well known for a long time from measurements and theoretical studies

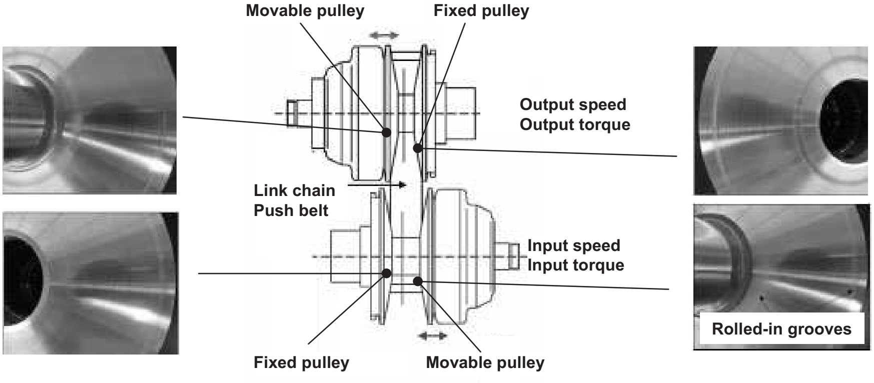


**Fig. 19.24** Schematic diagram of a variator in a CVT gear box.

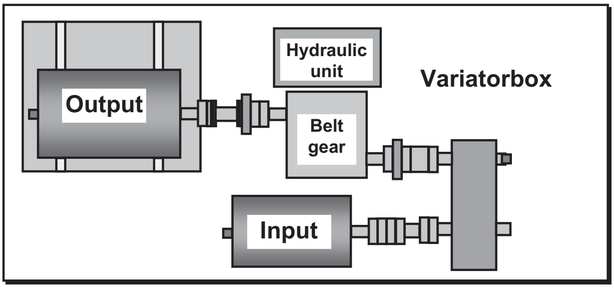
[19.61–19.75]. The construction, optimization, and design of future CVT by the means of available calculation algorithms [19.64, 19.74] require the analysis of the operating points. This is based on measurements on appropriate CVT variator test rigs described in Sect. 19.7.3.

### 19.7.3
### Standardized Test Rigs and Test Methods

All approval tests for current CVT fluids and advanced developments of new formulations are conducted with regular variator components housed in the test gearbox. Two different test rigs are available commercially. Each rig tests a complete variator from production. These rigs are the ZF universal variator test bench [19.76] and the Van Doorne test bench (VDT) [19.77]. The more versatile ZF rig tests both push belt and link chain systems. The VDT rig is specified for the VDT chain system only. Both rigs cover fluid-durability tests and functionality items running different multiple-block programs. The durability items are wear of the belt/chain and pulleys, fatigue of belt/chain and pulleys, and/or the degradation of the fluid (friction characteristics). The functionality items are the torque transmittance (maximum torque capacity), variator efficiency (KpKs evaluation of input and output thrust force/pressure on the movable pulley and the stability of the tribological contact), noise and vibrations, and the cooling properties and/or heat dissipation.

#### 19.7.3.1 **ZF Universal Variator Test Bench**

A schematic diagram of the test rig, test set-up, and technical data is given in Fig. 19.25. The arrangement of the electric motors and belt gear is variable for different CVT systems (push belt or link chain). This test machine can run with a minimum of 8 L test fluid only and enables cold start testing at –20 °C by use of an additional cooling device. A 130-h fluid test procedure has been developed by LUK (manufacturer of link chains for CVT) and ZF for approval of CVT fluids in CFT 23 and 30 gear series for Ford. After running-in (preconditioning of the tribological contact)



| Electric motor 1 – Input machine | | Hydraulic and cooling unit | |
|---|---|---|---|
| Input power | 240 kW | Oil temperature | -20 – 140 °C |
| Input speed | 5 500 / 6 500 rpm | Pressure | 1 bar - 80 bar |
| Torque | 400 / 380 Nm | Oil flow rate | 5 – 9 lmin$^{-1}$ |
| Torque impact | 560 Nm (< 5 500 rpm) | Total oil volume | 8 – 55 l |

| Electric motor 2 – Output machine | |
|---|---|
| Input power | 255 kW |
| Input speed | 1 350 / 6 700 rpm |
| Torque | 1 800 / 349 Nm |
| Torque impact | 2 520 Nm (< 1 350 rpm) |

**Fig. 19.25** Schematic diagram of a ZF universal variator test bench.

the KpKs measurement is conducted temporally for evaluation of the force/pressure input/output and stability of the tribological contact. A so-called zeta-max measurement is also made to find the maximum input/output ratio (zeta), to define torque capacity on the basis of a nondestroying method and for comparison with the with friction coefficient determined by the slip-curve method. A zeta-SF measurement can also be used to determine the coefficient of friction in gear ratio LOW. This value will be used for the calculation and evaluation phase of the fluid test illustrated in Fig. 19.26 to show tribological contact stability by running at constant radii with

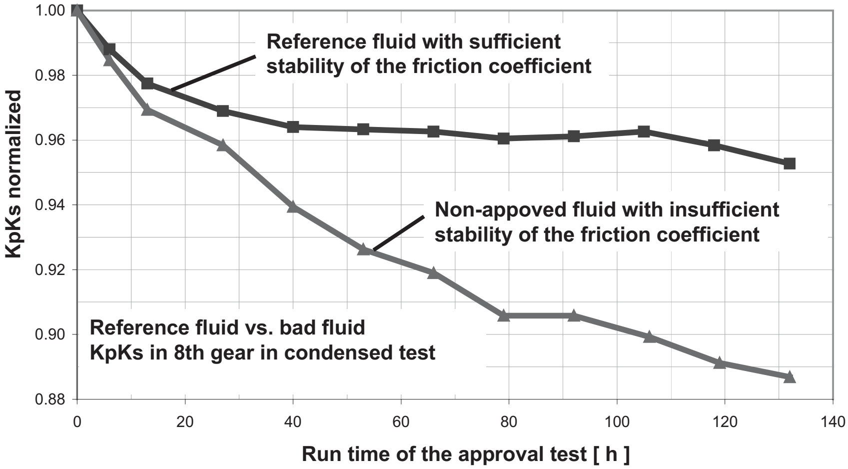


**Fig. 19.26** Test result from the LUK/ZF CVT fluid approval test.

zeta measurement. Between the temporal zeta measurements high-load and high-speed test phases are implemented for 20 h each. This is the so-called steady-state test running in 1st and 8th gear alternately.

#### 19.7.3.2 Van Doorne Test Bench

The fluid test procedure entails 100-h full load testing at nominal torque in three fixed ratios (40 h TOP, 40 h OD (overdrive), and 20 h LOW). LOW and OD are the two most extreme ratios of the variator and TOP is the ratio at which vehicle top-speed is being reached. This durability cycle is being supplemented with wear inspections, wear measurements, and functionality related measurements. To judge the suitability of the fluid tested, all test results obtained for a fluid under investigation are compared with results obtained with a reference fluid. ESSO EZL799 is currently being used as the reference fluid. The fluid tests are being performed with VDTs 24 mm push belt-type 901018 (24/9/1.5/208.8) and ZFST (P811/Alaska) pulleys, using VDT belt boxes and test rigs. A fluid sump volume of approximately 30 to 40 L is used. The tests are performed with regular production belts and pulleys from one production batch, to minimize variation in hardware. Whenever belts or pulleys from other production batches are used, reference tests must to be performed with the reference fluid. A schematic diagram of the test arrangement and a more detailed description of the fifteen steps of the VDT test method are given elsewhere [19.77].

## 19.8
## Performance Tests for Hydraulic Fluid Applications

### 19.8.1
### Field of Application

Hydraulic systems and fluids are crucial components of a healthy operation. Today construction equipment, injection molding machines, steel mills, excavators, forestry equipment, and many other types of industrial and off-road operations rely on hydraulic systems and fluids to keep machinery running. Purposes of the oils in such systems include helping to reduce pump wear, protection against cavitation, and keeping hydraulic systems free from yellow metal corrosion. The oil must enable smooth functioning of valves and extend the life of fluid and filter and must provide a long service life under severe wet and dry operating conditions. Continuing progress in the development of hydraulic systems has led to extended maintenance intervals, increased equipment uptime, and reduced overall maintenance costs, all of which improve production efficiency [19.78]. The demand for anti-wear hydraulic fluids for use in mobile equipment utilizing high-performance hydraulic pumps is increasing rapidly, and equipment breakdowns are costly. Hydraulic equipment failure can be reduced by use of fluids that provide good anti-wear protection in hydraulic pumps. Increased use of ashless (metal-free) additives helps minimize disposal and effluent-treatment costs. The technology also supports a trend toward limiting heavy metal contamination of the environment.

To maintain adequate pump efficiency and wear-protection, correct selection of fluid viscosity grade has become critical. The effect of hydraulic fluid viscosity on the efficiency of vane, gear, and piston pumps emphasizes the effect of viscosity index and shear stability. Analysis of the latest research on pump test machines reveals that selection of the correct hydraulic fluid significantly improves overall efficiency [19.79]. Selection of an appropriate high-VI, highly shear stable, oil can improve pump efficiency compared with a conventional HM (monograde) fluid of the same ISO grade [19.80]. Finally, identification of criteria defining an optimized hydraulic fluid, by use of test rigs and other procedures, enables an equipment operator to easily improve the performance of the system and reduce wear and fuel consumption. Effective tests can be conducted with modern vane, piston, and hybrid pumps.

### 19.8.2
### Function of Vane Pumps

In these pumps several vanes slide in slots in a rotor which rotates in housing or ring (cam ring). The housing may be eccentric with the center of the rotor or its shape may be oval. In some designs centrifugal force holds the vanes in contact with the housing and the vanes are forced in and out of the slots by the eccentricity of the housing. In one vane pump, light springs hold the vanes against the housing; in another design, pressurized pins urge the vanes outward. During rotation, as the space or chamber enclosed by vanes, rotor, and housing increases, a vacuum is created, and atmospheric pressure forces oil into this space, the inlet side of the pump. As the space or volume enclosed decreases, the liquid is forced out through the discharge ports.

### 19.8.3
### Standardized Vane Pump Test Machines and Test Methods

Tests using vane pumps have been the most important wear tests for both water-containing (HFA, HFC, HFD) and non water-containing hydraulic fluids (H, HL, HLP) since the middle of the 1960s. The most important tests for qualification of hydraulic oils used in Vickers vane pumps are used as standardized procedures (Table 19.14) for determination of anti-wear properties using an electric motor driven Vickers V-104-C or V-105-C-10 hydraulic pump. Today's test cartridges are manufactured by Conestoga. Anti-wear performance is determined by measurement of weight loss of pump vanes and cam rings after hours of continuous operation, followed by visual inspection for scoring and scuffing. Modern pumps operate at up to twice their nominal pressure. A diagram of the vane pump's test principle is shown in Fig. 19.27. The Eaton Vickers 35VQ25A pump test (form M-2952-S, revised 8/88) requires 50 h operation at maximum rated pressure and speed. This vane pump can be driven either by an electric motor or by a diesel engine. Associated flow control, reservoir, and heat-exchanger equipment combine to provide substantial flexibility under a variety of operating conditions. In this test the pump must pass three out of five procedures. The test is also required for the Caterpillar TO-4 specification.

**Tab. 19.14** Vane pump tests for hydraulic fluids.

| *Technical data* | *Vickers V-104-C (V-105-C-10)* | *Eaton Vickers 35VQ25A* | *Denison T6CM (HF0, HF2)* |
|---|---|---|---|
| Test duration | 100 h or 150 h | 50 h | 100 h |
| Test temperature | 71–90 °C (160–194 °F) at 13 $mm^2 s^{-1}$ | 93.3 °C (200 °F) | 71 °C (160 °F) for 60 h<br>99 °C (210 °F) for 40 h |
| Maximum pressure | 250 bar (3625 psig) | 207 bar (3000 psig) | 180 bar (2500 psig) |
| Pump speed | 1500 rpm | 2400 rpm | 2400 rpm |
| Flow rate | 22 L $min^{-1}$ (5.8 USgall $min^{-1}$) | 144 L $min^{-1}$ (38.0 USgall $min^{-1}$) | 265 L $min^{-1}$ (70.0 US gall $min^{-1}$) |
| Fluid volume | 70 L (18.5 USgall) | 197 L (52 USgall) | 189 L (50 USgall) |

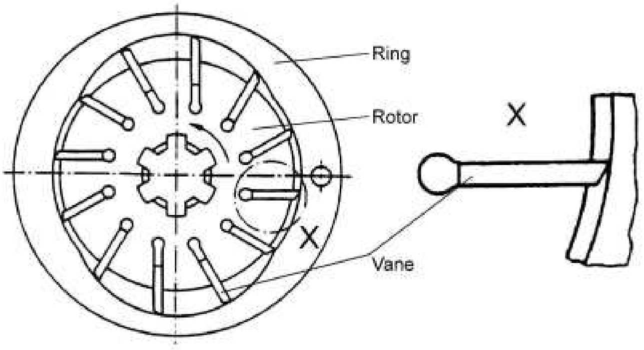


**Fig. 19.27** Test principle of a vane pump.

The Denison test targets in a T6CM pump (TP-30283) evaluate hydraulic fluids in vane pumps at high pressure and at high temperature, with and without the presence of water, by running an intermittent pressure cycle. The test objective is checking the evolution of viscosity, filterability, thermal stability, anti-corrosion fluid capability, and pump wear, with time, with or without water addition. The first test phase is run dry. In test phase II 1 % distilled water is added and the test is then run with the same fluid as in phase I.

## 19.8.4
## Function of Axial Piston Pumps

Axial piston pumps are open or closed-circuit pumps. They can be used with other components in a system to transfer and control hydraulic power. They provide an infinitely variable flow rate between zero and maximum. The pumps are compact, high power-density units, using the axial piston concept in conjunction with a tiltable swash plate to vary the displacement of the pump. Depending from the speed of the engine and the swivel angle the pump delivers a flow and the torque of the drive shaft is the result of the pressure difference between tank and load. Axial piston pumps are divided in two different groups, those with bent-axis design and those with swash plate design. Hydraulic test pumps for fluid testing are usually of bent-axis design, shown in Fig. 19.28. They are often used in mobile hydraulic applications, for example excavators and forest machines. The pistons of a pump of the bent-axis design are connected with spherical joints to the drive shaft and are forced axially in the rotating piston drum. Toggling of each piston must be allowed – otherwise the system becomes kinematically blocked, because the angle of the drum forms an ellipse on the flange of the drive shaft.

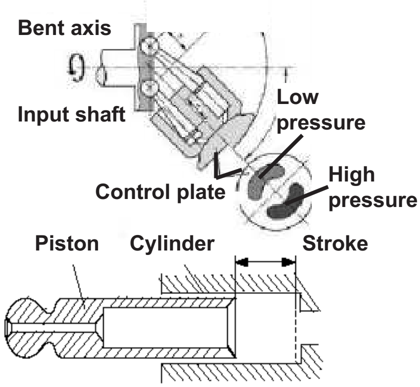


**Fig. 19.28** Principle of an axial piston pump of bent-axis design [19.81].

The stroke of the pistons depends on the swivel angle between drive shaft and drum. The kidney-shaped opening of the control plate, which does not rotate, connects the piston, by high pressure, to the power supply and low pressure or tank. The control plate with its spherical bearing is moved by a bolt, which also has a spherical bearing so that the stroke from the piston depends directly on the swivel angle. The bearing of the pistons minimizes friction between pistons and drum, so the forces between them are very small. As a result of the construction these pump types have a very good efficiency coefficient at low rotational speed. The more complex construction, because a double bearing on the connecting rods is necessary, is detrimental. Driving of the piston drum by the connecting rod and its cyclic change during one rotation, or by a variation of the rotational speed, results in torsion oscillations. The consequences could be wear and damage of the connecting rods and the gear wheels or, at worst, broken pistons.

### 19.8.5
### Standardized Axial Piston Pump Test Machines and Test Methods

Hydraulic fluid performance is evaluated in the Denison P46 piston pump test machine. These tests determine the effects of hydraulic oil on flow, deposits, and wear in a Denison 46 series piston pump. The main equipment used in the procedure are a Cummins L-10 diesel engine equipped with a speed-increasing gearbox coupled with a 46 series pump, a 46 series motor, and a Denison charge pump. Each test is run at 2400 rpm for 60 h at 71 °C and then for 40 h at 99 °C. After 60 h the pump is dismantled and inspected for evidence of distress. After the test run the pump hardware is examined by use of microscope systems. Three-dimensional stereo zoom enables magnification up to 160 times. A real-time color video display facilitates simultaneous viewing of microscopic phenomena [19.82]. Although Denison P46 testing is now obsolete, it is still available in many laboratories worldwide.

The Sundstrand series 22 axial flow pump, driven by a six-cylinder diesel engine, is a highly specialized system that can be used to determine the ability of hydraulic fluids to maintain rated flow at high pressure in the presence of water. Hydraulic pump efficiency can be degraded by a small amount of water in the hydraulic fluid if it causes dynamic corrosion of yellow metals in the pump. This procedure became known as the Sundstrand dynamic corrosion test. The Sundstrand pump is run at high pressure at an outlet temperature of 82 °C for 225 h, with water added after the first 25 h. Evaluations include John Deere fluids, ASTM multipurpose power transmission fluids, and a variety of hydraulic fluids.

The performance of Orenstein and Koppel hydraulic fluid is evaluated in the O and K test machine using an A2F010 “Brueninghaus Hydromatik” piston pump with bent-axis design. This procedure covers hydraulic fluids on piston pumps at high pressure and at high temperature without the presence of water by running intermittent pressure cycle. This endurance test evaluates all the hydraulic equipment utilized, including the fluid. The minimum duration is 5000 h. Pump wear is kept under surveillance by precise measurement of leakage flow after each 500 h. Operating conditions used in axial pump testing of hydraulic fluids are compared in Table 19.15.

**Tab. 19.15** Axial piston pump testing of hydraulic fluids.

| ***Technical data*** | ***Denison P46 (HF1)*** | ***Sundstrand series 22*** | ***O and K Rexroth A2F010 test pump*** |
|---|---|---|---|
| Test duration | 100 h | 225 h | 5000 h |
| Test temperature | 71 °C (160 °F) for 60 h | 99 °C (210 °F) for 40 h | 82 °C (180 °F)<br>80 °C (176 °F) |
| Maximum pressure | 350 bar (5000 psig) | 350 bar (5000 psig) | 350 bar (5000 psig) |
| Pump speed | 2400 rpm | 3100 rpm | 1460 rpm |
| Flow rate | 96.5 L $\mathrm{min}^{-1}$<br>(25.5 USgall $\mathrm{min}^{-1}$) | 94.6 L $\mathrm{min}^{-1}$<br>(25.0 USgall $\mathrm{min}^{-1}$) | 11.0 L $\mathrm{min}^{-1}$<br>(2.9 USgall $\mathrm{min}^{-1}$) |
| Fluid volume | 50 L (13 USgall) | 45 L (12 USgall) | 20 L (5.3 USgall) |

### 19.8.6
### Nonstandardized Specialized Testing, Efficiency Tests

Many universal hydraulic test machines have been developed for testing nonstandard hydraulic fluids or for investigation of the interaction of hydraulic hardware with the fluid. These have flexible capabilities, for example variable drive speeds and drive units up to 400 kW, and enable measurement of inline particle/debris, inline fluid density, and pump driving torque with state-of-the-art computer data acquisition and control. These universal hydraulic test machines are configured so that virtually any type of hydraulic pump can be installed for evaluation of hydraulic fluid or equipment performance under a multitude of conditions. The rigs are mainly used for investigation of the effect of hydraulic fluid viscosity on the pumping efficiency of vane, gear, piston, and double-piston pumps and hybrid pumps. Hydraulic fluids must sustain their minimum viscosity under load and shear. These rigs are particularly important for investigation of the modern phenomenon of temporary shear loss of high-VI fluids.

### 19.8.7
### Hybrid Pump Testing

In 2005 a new Denison test procedure was introduced for evaluation of the effect of hydraulic oil on flow, pressure loss, filterability, and wear using a hybrid T6H20C style piston and vane pump run near rated conditions (TP-30533). The tests are made up of a break-in phase, a dry oil testing phase (no water added), and a wet oil (water added) testing phase. The dry and wet phases are each slightly longer than 300 h. These are cyclic tests with the outlet pressures for both pumps changing every two seconds. The same test pump is used for both wet and dry phases. Test targets are to evaluate hydraulic fluids on vane pumps at 3600 PSI (250 bar) and on piston pumps at 4000 PSI (280 bar), 1700 rpm, high temperature, with and without the presence of water. The pressure cycles are intermittent. As in the older Denison procedures, evolution of viscosity, filterability, thermal stability, shear stability, anticorrosion fluid capability, and pump wear, with or without addition of water, are checked as time passes. This test enables reliable evaluation of the suitability of fluids for use in hydraulic pumps in severe duty applications. Further development steps have enabled improvement of the performance and characteristics of the pumps and the fluids. Test phase I requires use of a fluid of the quality of NAS 1638 class 8 maximum. Phase II tests the same fluid as in phase I, but with 1 % of distilled water added for between 307 and 308 h. The water concentration must be maintained between 0.8 and 1.2 % throughout the duration of this phase. Some water must eventually be added at minimum circuit pressure. These water additions must be recorded. Water is determined by the Karl Fischer method. During this phase a maximum of 2 L of distilled water will be added at pump suction level (30 mL every minute for a total of 1 h). The pump will run at minimum circuit pressure of 100 to 143 PSI (7 to 10 bar). Water decantation, when unexpected, occurs after a prolonged stop. The minimum fixed acceptable time is either one hour or, if it is a detergent fluid, a function of fluid quality. If the vane cartridge is destroyed, it can be removed; the test continues only for HF-1 evaluation. If piston rotating group is destroyed, remove the barrel, pistons, wear plate, and port plate then continue the test for HF-2 evaluation. If a fluid is HF-0 approved it is suitable both for piston pumps and for vane pumps, otherwise approval is HF-1 or HF-2 only.

**Tab. 19.16** Pump tests machines and test standards for hydraulic fluids.

| Test machine | Application | Test method |
|---|---|---|
| Vickers vane pump | Test method for indicating the wear characteristics of petroleum and non-petroleum hydraulic fluids in a constant volume vane pump | ASTM D 2882 |
| | Eaton Vickers 35VQ25A pump test | ASTM D 7043 |
| | Determination of the anti-wear properties of hydraulic fluids – vane-pump method | IP 281/77 |
| | Mechanical testing of hydraulic fluids in the vane pump | DIN 51389 |
| | General working principles | DIN 51389, Part 1 |
| | Procedure A for non-water containing hydraulic fluids | DIN 51389, Part 2 |
| | Procedure B for water-containing, flame-resistant hydraulic fluids | DIN 51389, Part 3 |
| Denison vane pump (HF0, HF2) | Denison T6CM hydraulic vane pump test | Denison specification TP-30283 |
| Denison piston pump (HF0, HF1) | Denison P46 hydraulic piston pump test | Denison specification TP-30560 |
| Denison hybrid pump (HF0, HF1, HF2) | Denison T6H20C vane and piston pump test | Denison specification TP-30533 |
| O and K hydraulic test machine | Determination of the wear behavior and oil ageing of hydraulic oils in an axial piston pump | O and K specification |

## 19.9
## Other Standardized and Non-Standardized Test Methods and Test Machines for Lubricants

In addition to the test machines and test methods described in Sects. 19.2 to 19.8, Table 19.17 summarizes other test procedures still used for mechanical–dynamic testing of lubrication oils. Further standardized test methods used in laboratories throughout the world are described elsewhere [19.37–19.39, 19.83–19.89].

**Tab. 19.17** More test standards for lubrication oils.

| *Test machine* | *Application* | *Test method* |
|---|---|---|
| Roll stability test apparatus | Standard test method for roll stability of lubricating grease | ASTM D 1831 |
| SKF (EMCOR) anti-rust rig | Determination of rust-prevention characteristics of lubricating greases | IP 220/93<br>DIN 51802 |
| Water-washout apparatus | Standard test method for determining the water washout characteristics of lubricating grease | ASTM D 1264<br>IP 215/93<br>DIN 51807, Part 2 |
| ASTM torque test apparatus | Standard test method for low-temperature torque of ball bearing grease | ASTM D 1478 |
| IP low temperature torque apparatus | Determination of the low-temperature torque of lubricating grease | IP 186/93 |
| ASTM leakage test apparatus | Standard test method for leakage tendencies of automotive wheel bearing greases | ASTM D 1263 |
| Sonic probe type apparatus | Standard test method for sonic-shear stability of polymer-containing oils | ASTM D 2603 |
| Shell DeLimon rheometer | Standard test method for determination of delivery resistance using the Shell DeLimon rheometer | E-DIN 51816, Part 1[a)] |
| Evaporation loss tester | Standard test method for engine and hydraulic oils for determination of evaporation loss and sediments | WOLF[a)] specification |
| Incline tribometer | Standard test method for determination of friction coefficients | H. Schmidt specification |

a) Obsolete but still in use

## 19.10
## Interpretation and Precision of Lubricant Tests

Most results from tribological lubricant tests must be interpreted and cannot often be regarded as absolute values. For this reason reference tests and lubricants have particular significance. The precision of all test methods is, usually, substantially affected by the manufactured tolerances achieved by the test machines and test pieces, by the measurement and control systems used , by mechanical elasticities, by and other factors which depend on the operating conditions and on ambient conditions.

The precision of a test method is evaluated from data on the repeatability and reproducibility of the test results and, in Germany as an example, the demands made on these data are standardized in accordance with DIN 51848 [19.90]. The repeatability of a test result is applicable for one observer and one test machine on which it was performed. The reproducibility of a test result is extremely important in relation to compliance with the test standards and specifications of a lubricant. High repeatability is a basic prerequisite for satisfactory reproducibility of a test result. In addition, comparison of existing test conditions with real conditions is of particular importance in assessing the relevance of results obtained in tribological lubricant tests. This requires adjustment of the test conditions to the specific loads, sliding speeds, lubricant temperatures, and materials used in practice. Only when these conditions are taken into account can the test method selected, with the help of mechanical–dynamic lubricant tests, assess the effect of the lubricant.

# Subject Index

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