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Matrix Permeability Measurement of Gas Productive Shales

D.L. Luffel, ResTech Houston; C.W. Hopkins, S.A. Holditch & Assocs. Inc.; and
P.D. Schettler Jr., Juniata College

SPE Members

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Abstract

Three laboratory methods were developed to measure matrix gas permeability (K_m) of Devonian shale cores and drill cuttings at native water saturations. The first method uses pulse pressure testing of core plugs with helium. The second, new method uses pulse pressure testing of core chips or drill cuttings with helium. These methods gave comparable results on 23 companion shale samples from two wells, with K_m = 0.2 to 19 x 10^{-8} md. The third, new method uses degassibility of core plugs with helium and methane, and yielded K_m higher by a factor of 3 to 10. Most of the core plugs tested showed multiple microfractures that remain open at reservoir stress, and these dominate conventional flow tests. These microfractures are parallel to bedding, are coring induced, and are not present in the reservoir. Knowledge of K_m is important in computer simulation modeling of long term Devonian shale gas production, and has been a key to understanding the nature of the natural fracture network present in the reservoir.

Introduction

The Devonian shale is a commercially productive gas reservoir over a wide area of the Appalachian basin. Properties of the shale, especially permeability, have been the subject of a significant amount of research. Permeability results from previous measurements of Devonian shale have ranged from < 0.01 to 800 microdarcies (1 x 10^{-5} to 0.8 md)[1,2]. Two factors have made these measurements of permeability unreliable. First, pulse test experiments have been generally limited to measuring permeability greater than 0.01 µd. Second, laboratory tests show that even when the shale cores are loaded to reservoir stress one or more coring-induced microfractures are usually present that remain partially open. These microfractures dominate flow, so observed permeability exceeds true matrix permeability by several orders of magnitude.

Devonian shale reservoir bulk gas permeability in the Appalachian basin is generally greater than 10 µd based on well flow rates (typically greater than 20 to 30 MCF/D), so natural fractures must play a dominant role in gas productivity. Since matrix permeability (K_m) appears to be very small, why is it important? Figure 1 shows the predicted cumulative production from a typical Devonian shale gas well based on 3D computer modeling. Table 1 shows the fracture spacing, aperture, and other reservoir properties used in the model. These properties are considered typical of those found in Pike County, Kentucky, based on Gas Research Institute study (discussed below)[3]. In Figure 1, for $K_m > 10^{-6}$ md (0.001 µd), recovery is independent of K_m, i.e., productivity is controlled only by fracture properties. For values of $K_m < 10^{-9}$ md, recovery is too low to be commercial. In the range 10^{-9} to 10^{-6} md, K_m is an important factor. Our studies show K_m is in this range.

In 1991, GRI began a research program involving special coring, logging, and testing in the Devonian shale section of two wells in Pike County, Kentucky: Ashland FMC No. 69 and No. 78. A total of 445 feet of conventional oriented

References and illustrations at end of paper.

core was cut while drilling with dry air in the Transition/L. Huron section. Figure 2 shows the sequence of information collected at the wellsite and from experiments conducted in the laboratory to determine reservoir properties, especially permeability. Reservoir gas permeability was determined using special short interval open-and cased-hole production tests[3]. Natural fracture spacing, azimuth, and dip were determined from the FMS log, borehole camera, and detailed whole core description to characterize the fracture network and fracture permeability in the reservoir. In order to determine the matrix gas permeability of the shale, a fairly extensive laboratory program was initiated. Since, we were attempting to measure K_m at levels of 100 to 10,000 times lower than previous methods, and in the presence of microfractures, we chose three separate laboratory methods. This was to provide redundancy and internal validation of results. The three methods chosen were:

1. Pulse pressure testing of core plugs with helium.

2. Pulse pressure testing of crushed core chips with helium.

3. Degassibility testing of core plugs with helium and methane.

Companion core plugs or crushed samples were taken at 14 locations in FMC No. 69 and 31 in FMC No. 78 for these permeability tests. In addition, adjacent crushed core samples were used to measure porosity, native-state gas, oil, and water saturations, kerogen content, and mineralogy, using core analysis methods developed specifically for shale[4,5].

Microfractures From Thin Sections

The number and extent of the microfractures present in each core are needed to interpret K_m from the laboratory tests on core plugs. To aid in this, end trims were taken from each 1 1/2 inch diameter core plug, and fluorescent-dyed epoxy was injected at net in-situ stress of 2000 psig prior to thin section preparation[6]. Each thin section was examined with a petrographic microscope under both transmitted light (plane-and cross-polarized) and reflected light with the appropriate wave length to generate fluorescence from the dyed epoxy. This permits detection of smaller fractures. Figure 3 shows photomicrographs of a sample at 4219 feet, FMC No. 69, with transmitted plane-polarized light and reflected fluorescence of the same view. The major fracture has an aperture of about 10 microns and is parallel to bedding. The reflected fluorescent view shows several smaller microfractures. These are generally less than 5 microns aperture, usually discontinuous, and often clustered around the larger fractures. In general, all of the thin sections showed from 1 to 5 large continuous fractures, 0 to 2 small continuous fractures, and 5 to 20 discontinuous large and small fractures. All of these are parallel to sub-parallel to bedding and are not believed to be present in the reservoir.

Pulse Pressure Tests, Core Plugs

Kamath[7] showed that pulse pressure data from special core tests can be used to calculate fracture and matrix permeability separately. This method requires very small upstream and downstream chamber volumes (a few ml). Kamath reported results only on conventional reservoir rocks, using liquid flow. To determine shale matrix gas permeabilities in the presence of microfractures, new lab equipment, procedures, and interpretation methods were developed[8,9]. For the 1 1/2 inch diameter core plugs used, typical gas-filled pore space at connate water is one ml. For the experimental flow cell, upstream chamber volume is 3.4 ml and the downstream volume is 2.0 ml, the lowest volumes that could be achieved.

Pore space in the core plug is initially pressured to 1000 psig with helium, with sleeve pressure of 4000 psig, for one to two days. A pressure pulse of 40 psi is then introduced from the upstream chamber. Upstream and downstream chamber pressures are recorded for up to 15 hours, with high sampling rate during early time (0.1 sec). An approximate analytical solution was developed to interpret the data[8,9].

Prior to testing shale, the system was validated using a dry, Berea sandstone plug. Permeability tests with helium using either steady-state flow or the new pulse system showed K = 0.0010 md. The plug was cracked lengthwise, reassembled, and retested with the new pulse system. This gave good agreement, with K_m = 0.0013 md, and overall permeability = 0.063 md including the fracture.

Figure 4 shows the pressure behavior for a typical Devonian shale plug, with the interpreted results for gas-filled porosity, and fracture permeability as shown. Table 2 shows the K_m results from 23 plug samples from both wells. These K_m results are not slip corrected, i.e., they represent gas permeability at the mean pore pressure of 1000 psig used in these experiments. Later in the paper, effect of the slippage correction at different pores pressures will be discussed. Figure 5 presents K_m as a function of gas-filled porosity ($Ø_g$). K_m varies from 0.2 to 7.9 x 10^{-8} md, and $Ø_g$ varies from 1.6 to 6.0 percent as measured by these experiments. There is no apparent correlation. The permeability variation however, is rather small, and doesn't appear controlled by rock properties such as total porosity, gas-filled porosity, kerogen, or heavy mineral content. Note that all the core plugs tested come from an organic-rich shale section of the Devonian. Other rock types present in the Devonian include sandy shales and gray shales.

Within the set of 23 plug samples tested, two from FMC No. 78 had no apparent fractures present, as noted in Table 2. During the pulse tests, pressure decay was insufficient during the 15 hour test period to permit a solution for K_m. Each plug was then artificially fractured, reassembled, and retested with K_m results as shown in Table 2 and Figure 5.

Pulse Pressure Tests, Core Chips/Cuttings

In this method shale core samples at in-situ water saturations are crushed, and a narrow sieve cut is used in pulse pressure tests with helium to derive both K_m and $Ø_g$. Advantages of this method are that it is quick to run (roughly one hour), it is less expensive, it can be used on drill cuttings, and since the shale is likely to part along microfractures and bedding planes during crushing, individual chips are unlikely to contain microfractures. A disadvantage is that the test is run at no overburden stress. Note that there are several other shale properties that utilize crushed samples or cuttings in their analyses, such as porosity, fluid content, geochemical properties, mineralogy, and adsorption isotherms. This provides added incentive for developing this new method of permeability measurement, so that drill cuttings alone could provide most of the rock properties for which cores are normally needed.

The lab procedure is as follows. A measured weight of crushed shale (15 to 30 gm) is placed in a sample cell. Helium is then expanded into the sample cell from a reference chamber at 200 psig. Pressure instantly drops to a level dictated by the dead space in the sample cell, then decays with time to a lower pressure as helium moves into the pores within each shale chip. Figure 6 shows a typical test result. The pressure data are then modeled using a reservoir simulator, with the permeability and porosity results as shown in Figure 6. As with the pulse test on core plugs, these permeabilities are not slip corrected, and represent K_m at the mean pore pressure of about 100 psia. Initially, this method was validated using a dry Berea sandstone core plug (as a large, pseudo-chip) with K = 0.007 md based on steady-state helium flow tests. The pulse test showed K = 0.006 md, which is in good agreement. In addition, selected pulse tests were run on shale chips of various sieve cuts (10/20, 20/35, and 35/60 mesh). Figure 7 shows photomicrographs taken to characterize these chip sizes and shapes. The 20/35 mesh size (0.50 to 0.84 mm) was chosen to use for all the samples since it provided best pressure behavior and most symmetrical chip shapes. The simulator model for permeability interpretation assumes the unit chip shape is a cylinder with a diameter equal to the average mesh size and diameter twice the height.

Figure 8 shows K_m versus $Ø_g$ measured on 14 samples from FMC No. 69 and 31 samples from FMC No. 78. These K_m values are in the range from 0.4 to 45 x 10^{-8} md, which is roughly in the same range as, but somewhat higher than found by the pulse tests on the companion plugs. Note that the plugs were tested at 3000 psi net stress and 1000 psig pore pressure while the chips were tested at ambient stress and 100 psia pore pressure. In conventional, low permeability reservoir rocks this would cause the chip samples to show a gas permeability higher than in stressed samples by a factor of over 100. There is, however, some evidence (to be discussed) that gas permeability in shales is rather independent of overburden stress.

Figure 8 shows a fair correlation (with best fit line shown) between K_m and $Ø_g$ for the core chips in the black shales, as contrasted with no correlation found from the plug tests. Correlation of K_m from chip tests with K_m from individual companion plugs is generally rather weak. Overall, chip K_m is higher than plug K_m on average by a factor of four. As will be shown in the next section, the slippage correction for shales is so large that K_m is approximately proportional to the reciprocal of the mean pore pressure. The pulse tests on plugs were run at about 1000 psig mean pore pressure, and the pulse tests on the chips were run at about 100 psia, or a difference of a factor of 10. K_m for chips should, therefore, be 10 times higher. Probably calculated K_m values for the plug tests are too high because more microfractures are present than detected visually.

Also, Figure 8 shows K_m and $Ø_g$ results from tests on seven drill cuttings samples collected while drilling with reverse circulation. This method[10] of air-drilling provides large cuttings size compared to dust-sized particles from normal air-drilling. Results on the drill cuttings are comparable to those from the core chips. Figure 8 also shows results from six gray shales that suggest a different trend, with somewhat higher K_m for a given $Ø_g$.

Degassibility Tests, Core Plugs

A new degassibility method has been developed to measure shale matrix gas permeability to helium or methane in the presence of gas desorption[11,12]. Gas adsorption accounts for 30 to 80 percent of the total gas stored in Devonian shales. In this method, a shale core plug is held in a rubber sleeve at 2000 psi net stress, and equilibrated (soaked) at 500 psia with gas for several hours. A small dead volume (12 ml) at one core face is then exhausted rapidly (< 2 sec) to a vacuum and the cell sealed. Pressure rebound is measured, from which gas produced into the dead volume from the core is calculated, as shown in Figure 9 for a Devonian shale sample using methane. This is plotted as a function of a square-root-time scale. Early time, straight-line slope of the produced gas is used to calculate specific degassibility, G:

$$G = M/(2A\Delta pt^{1/2}) \quad \text{.......(1)}$$

In order to account for the effect of microfractures, their added surface area must be included in the calculation of G.

An independent measurement is made of the total adsorption isotherm on a companion crushed shale sample as shown in Figure 10. From the slope of the isotherm (S), which varies with pressure, the permeability can be derived:

$$K = 3140\, z\, RT\, \mu\, G^2/PS \quad \text{.......(2)}$$

For the methane degassibility results shown in Figure 9, calculated $K_m = 16 \times 10^{-8}$ md.

Prior to conducting the degassibility tests on shale plugs, validation tests were made on a Vycor plug. Vycor is a porous glass with average pore diameters of 0.004 to 0.007 microns, porosity of 28 percent, and gas permeability of 1 to 10 µd. Helium degassibility tests were run at sleeve pressure of 3000 psig and soak pressures of 260, 500, and 700 psia. From the tests, K, K_∞, and b (Klinkenberg factor) were derived, with $K_\infty b = 0.50$ md-psi. Steady state flow tests were also run at 1000 psig sleeve pressure using air and seven mean pore pressures from 23 to 65 psia with $K_\infty b = 0.67$ md-psi (with b adjusted to helium)[13]. The Vycor plug was then fractured and the degassibility tests repeated using helium with matrix $K_\infty b = 0.37$ md-psi.

The reason that $K_\infty b$ product is used for the above comparisons rather than K_∞ alone is as follows. In the Klinkenberg expression

$$K = K_\infty \left(1 + b/P_m\right) \quad \text{.......(3)}$$

if $b \gg P_m$ (such as in very low permeability shales), then

$$K P_m \approx K_\infty b \quad \text{.......(4)}$$

For a given net overburden stress (NOB), $K_\infty b$ is a constant for a particular core and K is proportional to $1/P_m$, so the $K_\infty b$ product is a useful parameter to characterize the rock. Figure 11 shows that degassibility behavior of Vycor using helium at NOB pressures of 1000, 2000, and 3000 psi, all at the same soak pressure of 260 psia, is independent of NOB stress. From equations (1) and (2) this leads to the conclusion that $K_\infty b$ is independent of NOB for this Vycor plug. This suggests that as NOB increases, K_∞ decreases, but b increases such that $K_\infty b$ is relatively constant. This effect has not yet been verified for shale cores. However, this would explain why K_m measured with the pulse tests on shale plugs at 2000 psi NOB stress and K_m measured with pulse tests on shale chips at ambient stress are roughly the same.

Table 3 shows the results of degassibility tests conducted on ten 3/4 inch diameter shale core plugs from FMC No. 69 using a sleeve pressure of 2000 psig and a soak pressure of 500 psia. K_m ranges from 0.67 to 199.7×10^{-8} md , with a median of 19.7×10^{-8} md (helium only). This compares to a median K_m of 2.0×10^{-8} md from the helium pulse tests on core plugs, and a median K_m of 6.2×10^{-8} md for the helium tests on shale chips, all from essentially companion samples from FMC No. 69. Note that for three plugs in the degassibility tests (Table 3), no microfractures were found. It is possible that microfractures are present, but not visible under a normal microscope, in which case calculated K_m is too high.

Table 3 also presents a comparison of K_m derived from degassibility tests using both helium and methane for plugs 2 and 18. This shows that K_m from methane is lower than K_m from helium by a factor of 4 to 8. Most of this difference is due to the lower b factor for methane. Methane b is lower than helium[13] by a factor of about 3, so apparent K_m (Equation 4) should be lower by 3, also.

Conclusions

1. Three methods have been used to measure matrix permeability of Devonian shales. Two of these are newly developed: pulse pressure testing of shale chips with helium, and degassibility of shale plugs using helium and methane.

2. From pulse tests on core plugs (23 in two wells) and pulse tests on companion chip samples with helium (45 in two wells), there is fair agreement showing $K_m = 0.2$ to 45×10^{-8} md. Degassibility tests with helium (10 core plugs in one well) shows $K_m = 0.67$ to 200×10^{-8} md and K_m higher than from pulse tests on plugs and chips by a factor of 3 to 10 on average.

3. In this range K_m is one of the controls on long term gas production in the Devonian shale even though the major flow network in the reservoir is through the natural fractures.

4. The degassibility process is important to understand since it mimics the dual process of desorption and gas transport that occurs in the reservoir.

5. Most of the core plugs tested showed multiple microfractures that remain open at NOB stress. These are parallel to bedding, are coring induced, and are not believed present in the reservoir. However, these microfractures dominate conventional pulse permeability tests on cores.

6. For these very low permeability rocks, the product $K_\infty b$ appears to be roughly a constant for a specific core, independent of NOB stress, and $K_\infty b \approx KP_m$. This explains why K_m from chip tests at ambient stress $\approx K_m$ from plug tests at 3000 psi NOB stress.

Nomenclature

A	=	degassing surface area, cm^2
b	=	Klinkenberg constant, psi
G	=	specific degassibility, mols/cm-atm-$sec^{1/2}$
K	=	permeability, md
K_m	=	matrix permeability, md
K_∞	=	Klinkenberg corrected permeability, md
M	=	gas produced during degassibility, gmols
P	=	average pressure, atm
P_m	=	mean pressure for Klinkenberg correction, psia
Δp	=	pressure drop causing degassing, atm
R	=	gas constant, atm-cc/g mol-°K
S	=	isotherm slope, gmol/cm^3-atm
t	=	time function, sec
T	=	absolute temperature, °K
z	=	gas compressibility factor,
μ	=	gas viscosity, cp

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Table 1

Reservoir Properties for Model Results Shown in Figure 1

Parameter	Value Used
Interval thickness	100 feet
Well drainage area	80 acres
Initial reservoir pressure	650 psia
Gas porosity	2.8%
Adsorbed gas	
Langmuir volume	110 SCF/ton
Langmuir pressure	540 psia
Apparent total adsorbed gas porosity	10.7%
Natural vertical fractures	
Spacing, X direction	2.5 feet
Spacing, Y direction	3.9 feet
Aperture	6.3 microns
Reservoir permeability	2.2 md-ft
Propped fracture	
Radius	200 feet
Conductivity	Infinite
Total gas in place	2.0 BCF

Table 2

Pulse Permeability Results on Shale Core Plugs

Sample Number	Core Depth	Gas-Filled Porosity %	Km 10^{-8} md	No. of Fractures
FMC No. 69				
5	4201.2	2.5	0.8	10
8	4218.2	4.4	4.0	4
16	4262.5	2.0	0.5	9
18	4269.5	4.3	2.1	3
21	4290.5	3.4	0.5	9
22	4298.1	2.3	0.6	8
23	4302.1	1.9	1.2	4
27	4321.5	3.1	2.0	5
29	4333.0	1.6	5.9	1
31	4345.1	3.2	4.3	1
33	4364.3	3.9	7.9	2
39	4399.0	1.7	2.5	2
FMC No. 78				
1	4152.2	1.7	1.7	3
2*	4167.1	6.0	1.2	3
3	4175.2	3.8	0.4	4
6	4200.3	4.6	2.8	1
12*	4260.3	5.4	0.2	2
15	4273.8	5.1	2.8	1
17	4282.4	4.3	3.0	2
21	4316.8	4.4	5.5	2
27	4365.4	2.5	1.7	1
28	4367.7	4.8	1.1	3
30	4377.8	3.8	0.4	2

*Core plug was artificially fractured

Table 3

Matrix Permeability Results from Degassibility Tests on Shale Core Plugs from FMC No. 69

Sample Number	Core Depth	Km 10^{-8} md	No. of Fractures	Gas Used
2	4183.4	63.0	3	Helium
2	4183.4	16.1	3	Methane
5	4201.3	1.9	5	Helium
8	4218.2	21.7	5	Helium
18	4269.9	1.27	5	Helium
18	4269.9	0.15	5	Methane
21	4290.7	17.7	3	Helium
22	4298.1	103.4	0	Helium
29	4333.0	0.67	0	Helium
33	4364.3	16.3	2	Helium
36	4376.2	101.4	1	Helium
39	4399.0	199.7	0	Helium

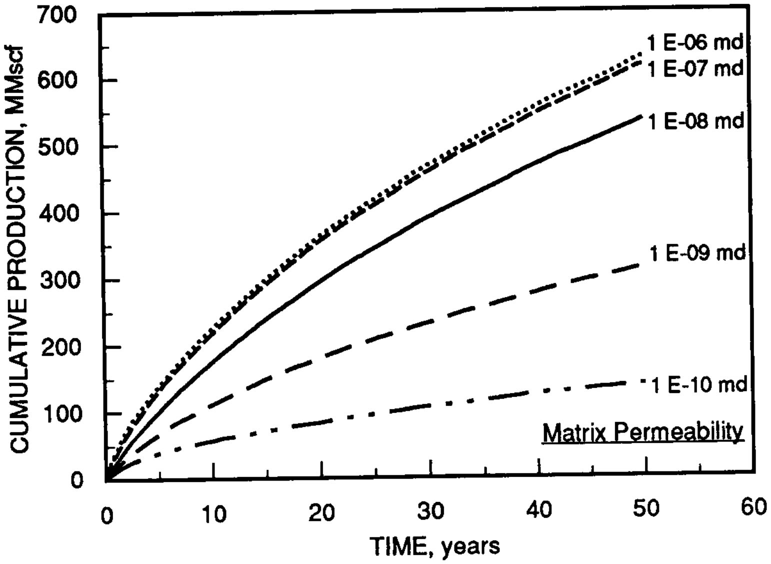

Fig. 1 - Effect of matrix permeability on cumulative production for typical Devonian shale well, Pike County, Kentucky, based on computer simulation

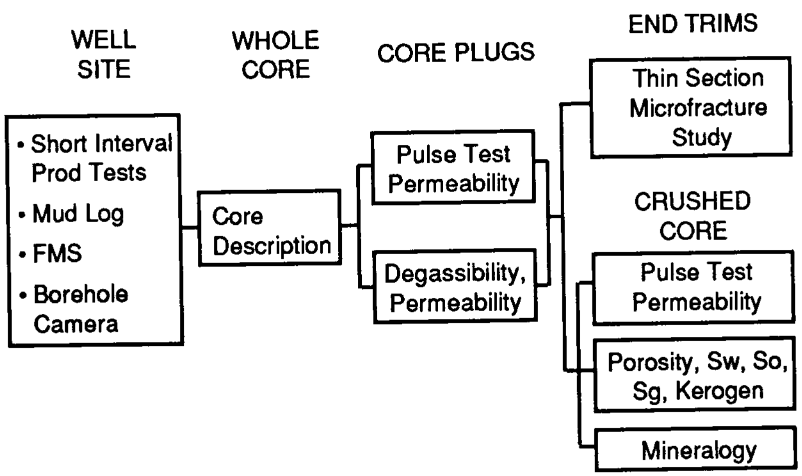

Fig. 2 - Sequence of experiments to link core analyses to reservoir permeability

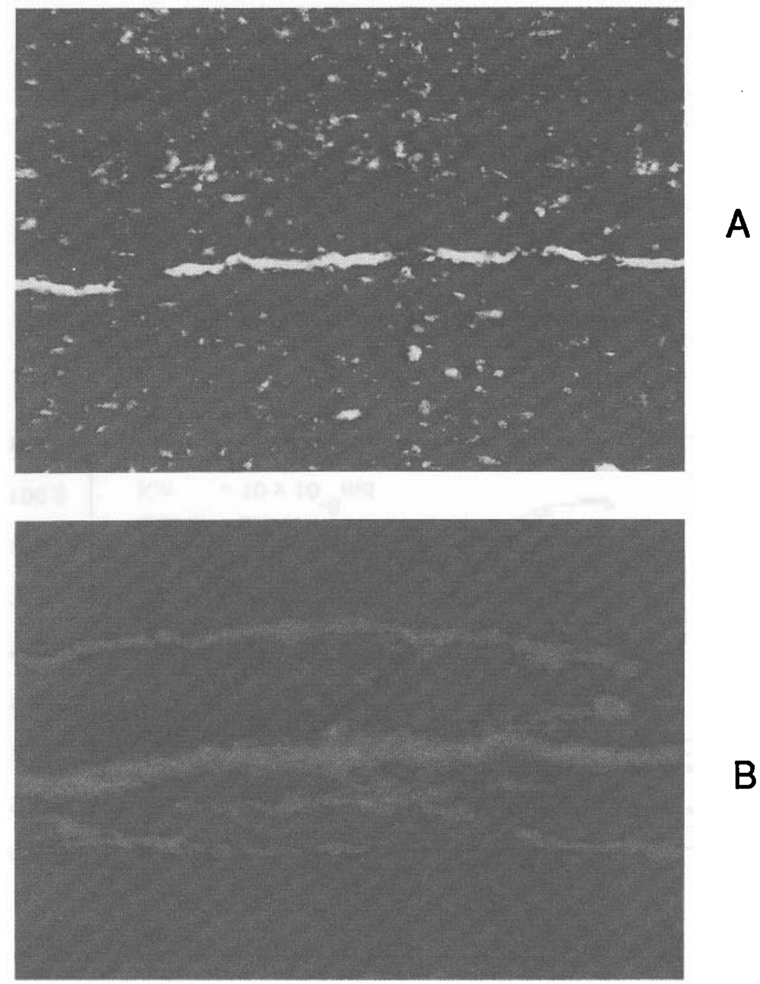

Fig. 3 - Photomicrograph of thin section from 4219 Feet, FMC 69. Photo A with plane polarized light, photo B with green-light fluorescence. Vertical dimension of photos is 0.44 mm.

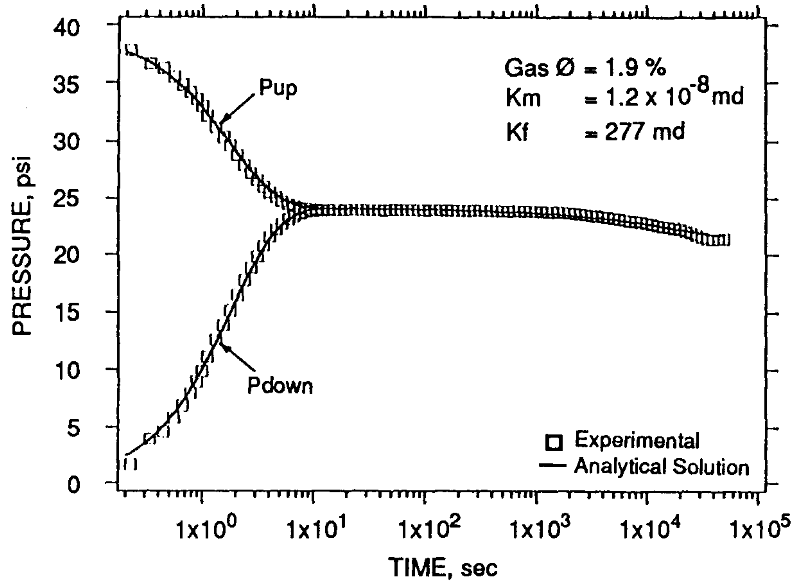

Fig. 4 - Pulse test pressure response, Lower Huron shale core, FMC 69

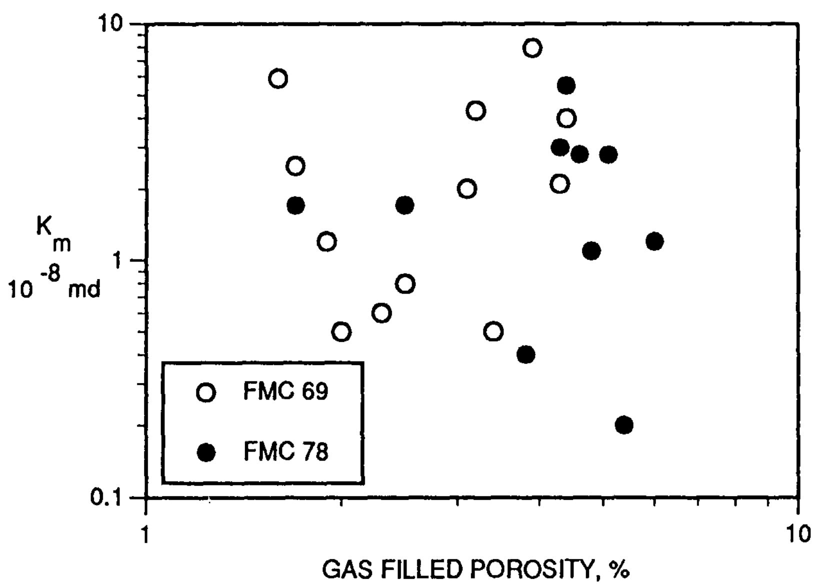

Fig. 5 - K_m varies from 0.2 to 8 x 10^{-8} md, unrelated to porosity

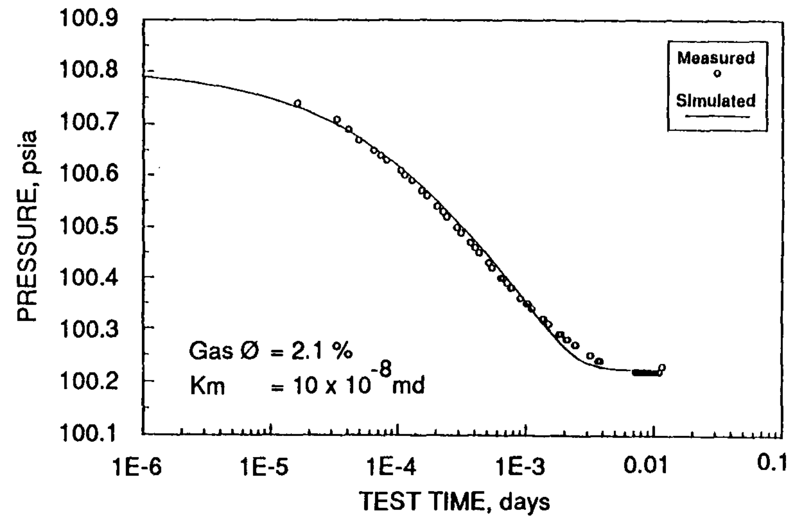

Fig. 6 - History match of 20/35 mesh crushed shale, FMC 69

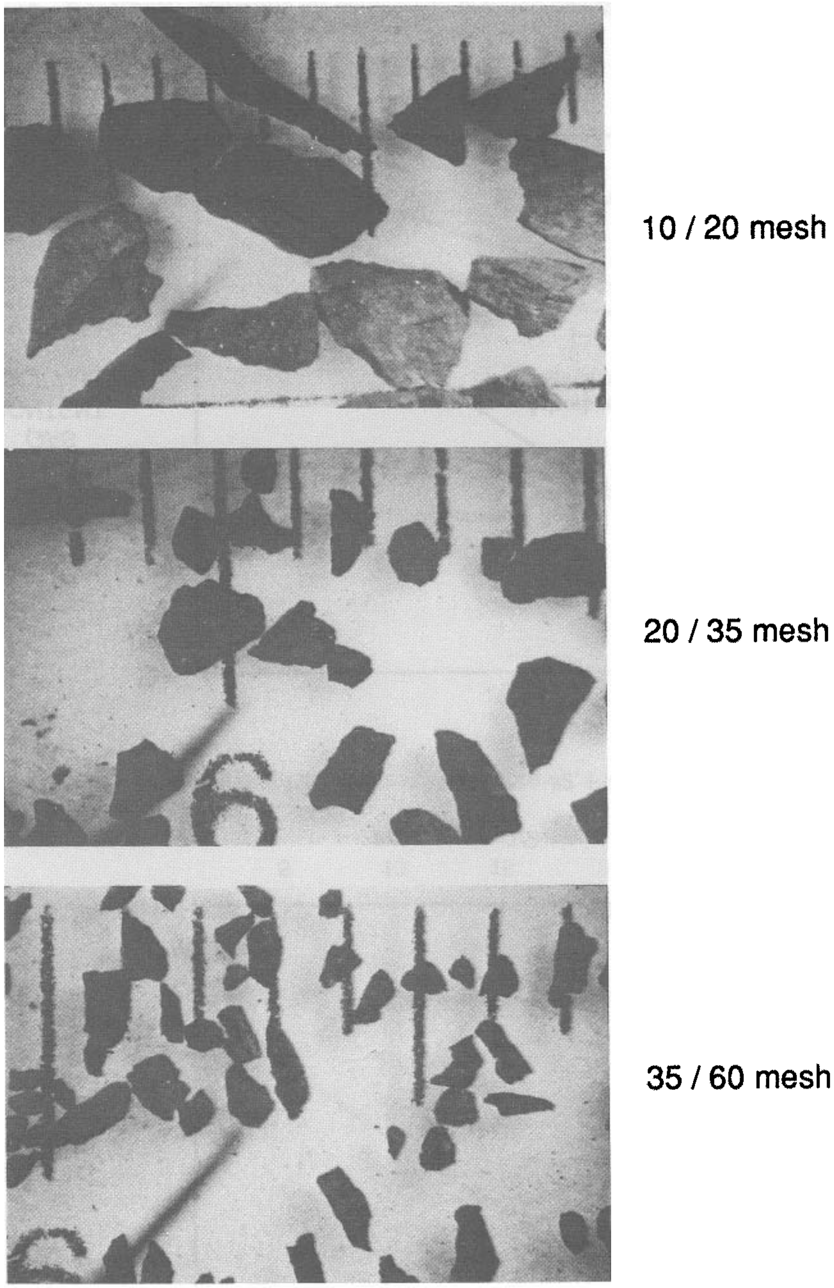

Fig. 7 - Photomicrograph of crushed shale core chips of different sieve cuts, FMC 69. Small ruler scale units = 1 mm

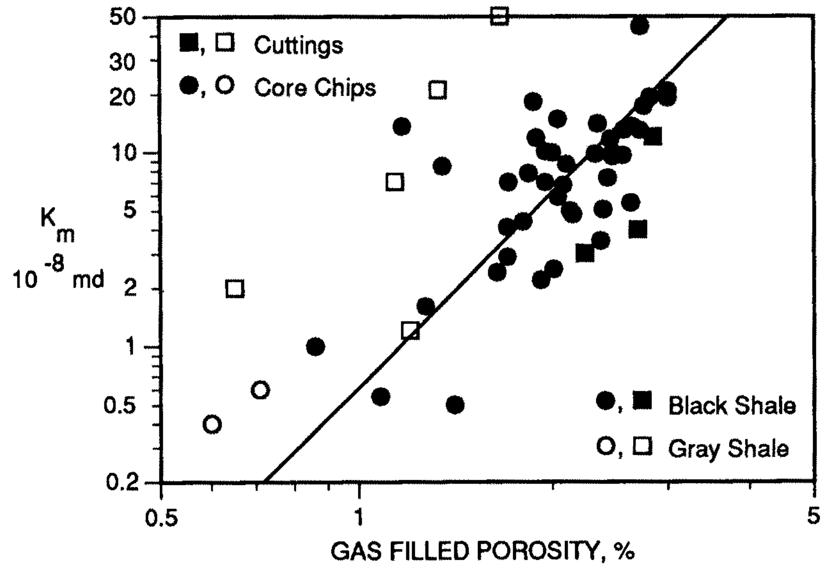

Fig. 8 - Pulse permeabilities on crushed shale chips using helium correlate with porosity

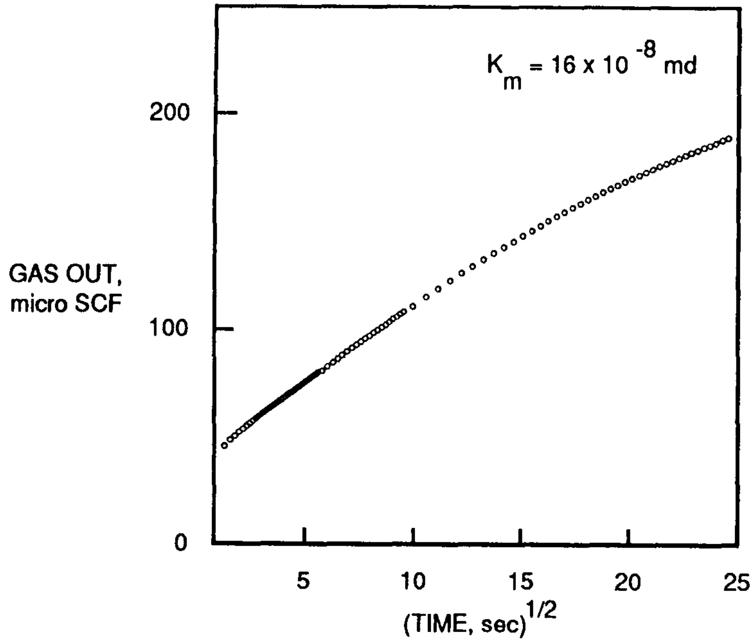

Fig. 9 - Methane degassibility test, shale core #2, FMC 69

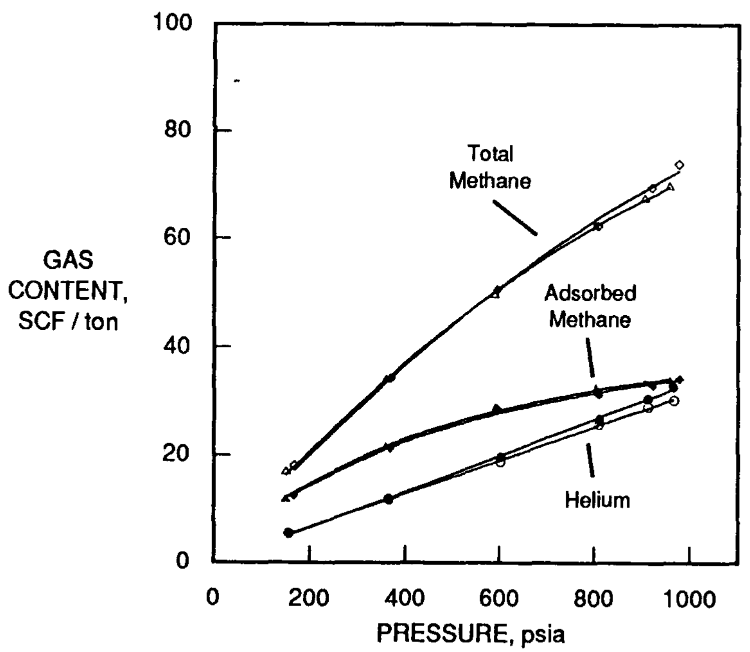

Fig. 10 - Desorption isotherms for companion crushed sample #2, FMC 69

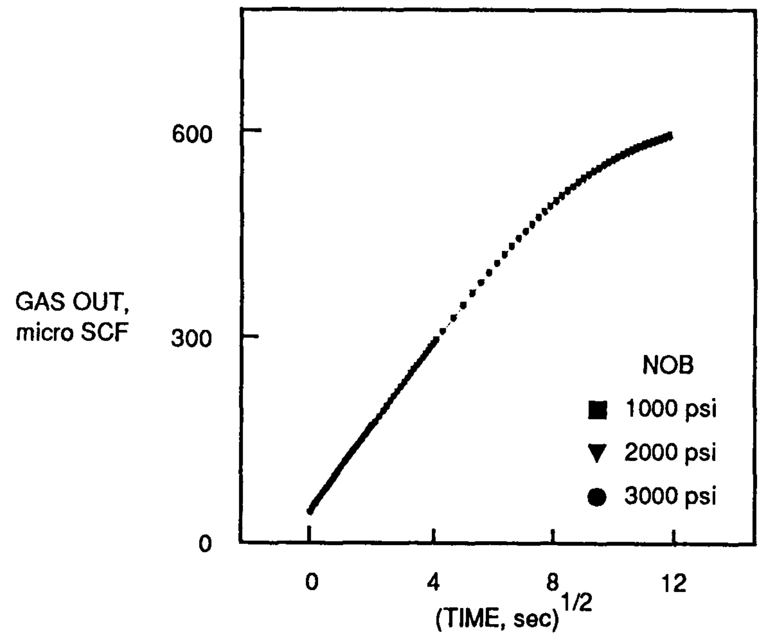

Fig. 11 - Degassibility of Vycor plug with helium is not affected by NOB stress