

STEADY STATE STRESS DEPENDENT PERMEABILITY MEASUREMENTS OF TIGHT OIL BEARING ROCKS

Shreerang S. Chhatre,¹ Edward M. Braun,² Somnath Sinha,¹ Matthew D. Determan,¹ Quinn R. Passey,¹ Timothy E. Zirkle,¹ Alex C. Wood,¹ Jeff A. Boros,¹ Daniel W. Berry,¹ Sergio A. Leonardi,¹ and Ryan A. Kudva¹

1. ExxonMobil Upstream Research Company, Houston TX, United States
2. Consultant, Houston TX, United States

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ABSTRACT

In the last few years, US tight oil production has increased significantly due to advancement in horizontal drilling and completions, hydraulic fracturing, along with favorable economics. For tight reservoirs, matrix permeability plays a key role in determining the drainage area for each well and the optimal well spacing. Currently, the industry practice is to measure permeability after crushing these oil bearing rocks. Permeability on crushed rock samples may not represent the *in-situ* properties. Many publications have reported the limitations of crushed rock measurements in characterizing tight rocks.

In this work, we present a steady-state method to measure liquid permeability on intact core plugs from tight oil bearing rock under reservoir stress conditions. We image the samples using CT scanning to evaluate their physical integrity. We measure the permeability of plugs while injecting a liquid and applying back pressure to compress and dissolve gases. We flow a liquid which is miscible with the remnant oil in the sample, and thereby perform permeability measurements on as-received samples, consequently avoiding the time-consuming step of cleaning such low permeability samples. We measure steady-state liquid permeability at various net confining stresses to simulate reservoir pressure drawdown and buildup.

Our results indicate that we can flow multiple pore volumes of the test liquid through a tight oil bearing sample in a few days to weeks. We can accurately measure steady-state liquid permeability for tight oil core plugs from a few microDarcies to tens of nanoDarcies. The permeability loss over time at constant net confining stress (*i.e.*, “stress creep”) is significant and this creep can last for a few weeks. The steady-state permeability strongly depends on the applied net confining stress and the stress history. Large permeability declines are observed as the applied stress is increased, and little recovery occurs as the stress is relieved. We validated the measurement technique by using a set of capillary standards that are not stress-sensitive and for which the permeability could be calculated using Darcy’s law and Hagen–Poiseuille equation.

INTRODUCTION

In the past few years, oil production from tight oil bearing rocks and shales in the US has increased from less than 1 million barrels/day in 2010 to more than 3 million barrels/day in the second half of 2013 [1]. The Bakken and the Eagle Ford regions are at the forefront of this tight oil production boom [2] [3]. Commercially viable initial oil production rates are achieved by a combination of hydraulic fractures and natural fractures in such tight reservoirs. However, once the fractures are depleted, the production needs to be supported by the flow of hydrocarbon from the reservoir matrix to these fractures. Therefore, matrix permeability becomes a key input in reservoir depletion planning.

Even though some laboratories have started using intact plugs, most commercially available measurements are conducted on crushed rock, using modified forms of the Gas Research Institute (GRI) method. This provides gas permeability as a proxy for oil permeability of tight oil bearing rocks [4]. A number of recent publications, including a round-robin study conducted by ExxonMobil reported that crushed rock permeability measurements have large variability depending on the vendor used for these measurements [5]. Therefore, there is a clear need for standardization of permeability measurement techniques for tight rocks. Further, such crushed rock measurements are conducted without applying any overburden stress. A crushed, unstressed state may not represent the *in-situ* condition of tight reservoirs.

An alternative to experimental measurements of permeability is to create a digital image of rocks using microscopy or CT scanning and to perform flow simulations on the resulting pore space. Due to the complex pore architecture and small pore sizes of tight rocks, a FIB-SEM based Digital Rock Physics approach has limited success in determining matrix permeability of such tight rocks [6].

We previously reported a steady-state technique for measuring gas permeability on tight intact core plugs with reservoir net confining stress (NCS) applied, both at room temperature and at reservoir temperature [7] [8]. In our recent publication, we have extended this work to present liquid permeability measurements on tight rocks from the *Vaca Muerta* formation, which is located in the *Neuquén* Basin in the western part of Argentina [9]. In this companion paper, we address various issues and learnings with steady-state liquid permeability measurements on intact tight rocks from a permeability range of about 10 microDarcy (μD) to about 30 nanoDarcy (nD).

PROCEDURE

Steady state method for measuring liquid permeability

We use core plugs in the as-received state from two cored wells in the *Vaca Muerta* shale play, located in the *Neuquén* Basin in the western part of Argentina. Helium porosity for plugs at similar depth was 4-17%, and total organic carbon (TOC) range was 0-12 wt.%. Our liquid permeability apparatus for tight rocks uses a constant pressure differential steady-state approach, similar to the previously reported apparatus for gas permeability of

tight rocks [7] [8]. Permeability measurements are conducted on core plugs with 1” or 1.5” diameter and greater than 1” length. Core plugs are mounted in core holders and confining pressure is applied using a sleeve fluid. A computer-controlled pump maintains constant upstream pressure by injecting a liquid. The downstream pressure is controlled using a back pressure regulator capable of handling two-phase flow. The flow rate of the measurement fluid is computed from a log of the piston position vs. time at a constant upstream pressure. Once the desired upstream, downstream, and confining pressures are applied, the following three phenomena occur concurrently, and the fluid throughput rate stabilizes slowly with time.

1. **Forcing the gas in the pores into solution at high pore pressure:** When high pore pressure is applied, the gas in the pore space is compressed and forced into solution. The dissolution of gas can be accelerated by displacing air in the pore space with methane, and then introducing liquid hydrocarbon at high pore pressure. Outlet flow rates are also periodically measured using from change in fluid volume in the pump vs. time, or by recording the weight of produced fluid vs. time.
2. **Stress creep:** Measured core permeability decreases with time at a constant sleeve pressure, a phenomenon called stress creep, which will be discussed later in detail.
3. **Miscible displacement of reservoir oil by injected fluid:** The core plug in the as-received state contains remnant oil, water and gas. Therefore, at least one pore volume of fluid is injected before permeability of the rock is recorded. The core saturation step can be long depending on the permeability and core dimensions (Figure 1). During core saturation, the average viscosity of the fluid is changing, and therefore permeability measurements are not recorded.

Time scale to flow one pore volume

For core analysis measurements on high permeability rocks, cores are cleaned using a sequence of solvents, and then “absolute” permeability of the core is measured by ensuring that only one fluid phase is flowing in the core. In tight rocks with permeabilities from 10 nD to 10 μ D, the time required for such flow through cleaning is significantly greater. The time (τ) required for one pore volume of a fluid with viscosity μ to flow through a core plug with length ℓ , porosity ϕ , and permeability k , when a pressure differential of ΔP is applied is calculated using Equation 1 and plotted in Figure 1.

$$\tau = \frac{\phi\mu\ell^2}{k\Delta P} \quad \text{Equation 1}$$

In the lab, we typically apply a pressure drop of 200 to 800 psi across a core plug, and therefore in about 3 weeks, one pore volume of fluid can flow through cores with permeability as low as 10 nD (the shaded area in Figure 1). However, cleaning these cores, with multiple solvents, and many pore volumes of each solvent, would take an impractically long time. Therefore, permeability measurements reported in this work begin on core plugs in the “as-received” saturation conditions. From Equation 1, we also realized that fluid throughput in terms of pore volumes scales inversely with the square of the core plug length. Therefore, for tight rock permeability measurements, we prefer to work with shorter plugs that are about 1” long.

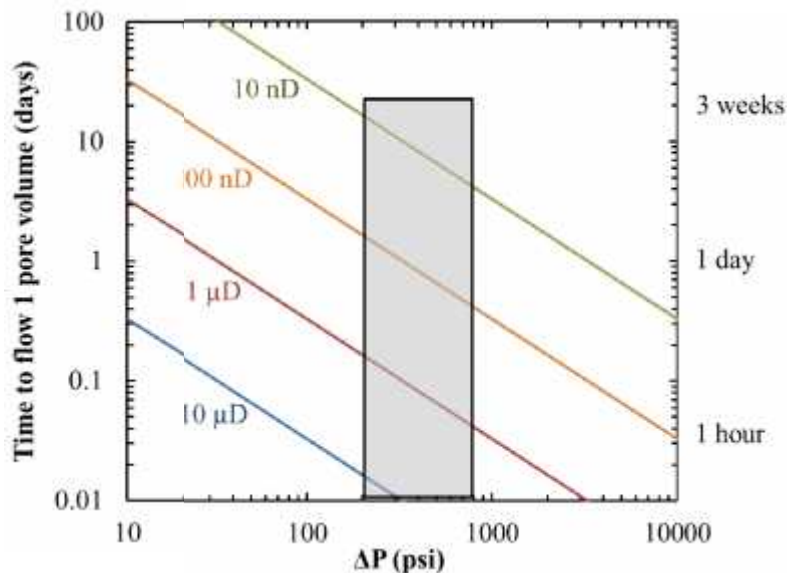


Figure 1. Time to flow one pore volume of fluid with viscosity of 0.6 cP (corresponding to toluene at room temperature); through a 1 inch long core plug with 5% porosity is plotted against the pressure difference across the core plug for tight rocks with permeability from 10 μ D to 10 nD. Range of pressure differences and experimental time per core plug is highlighted in the grey rectangle.

Choice of permeability measurement fluid

The choice of permeability measurement liquid was dictated by the following criteria:

1. High miscibility of the injected fluid with reservoir oils to avoid asphaltene precipitation in tight pores
2. Low viscosity to minimize the time required to flow through tight rocks (Equation 1)
3. Low water solubility to avoid the displacement of immovable water in the pore space of tight rocks
4. Chemical inertness to minimize interactions with rock surface, especially with swelling clays and organic matter in the rock
5. Chemical inertness to minimize laboratory chemical safety concerns

Based on these criteria, we have chosen to run the liquid permeability experiments using either decalin (decahydronaphthalene, 2.4 cP at room temperature), or toluene (0.6 cP at room temperature). Both are non-reactive hydrocarbons with low water solubility, and generally do not cause asphaltene precipitation [10].

Constant pressure difference test preferred over constant rate test

In our measurements, we set a fixed pressure drop across the core and measure the flow rate. This approach is faster than a constant rate method, which can suffer from slow equilibration due to high fluid compressibility and low mobility [11]. Also, as injection pressure must remain several hundred psi below the sleeve pressure, a constant pressure difference approach is clearly favored over a constant injection rate approach. Table 1 shows that, if constant rate measurements were to be used, the pressure drop would equilibrate with a time constant (τ) of hours to days, adding additional time to an already slow experiment.

Table 1. Sample calculation of time constant for pressure equilibration for tight rocks, assuming upstream pump volume of 50 cc containing a fluid with compressibility of 1×10^{-5} /psi, viscosity of 1 cP and a core plug with 1 inch length and 1 inch diameter

k	Core permeability	1,000	100	10	nD
$R = \Delta P/q = \mu \ell / kA$	Flow resistance	1.2×10^5	1.2×10^6	1.2×10^7	psi/(cc/min)
$\tau = RC$	Time constant	1	10	100	hours

In the constant rate mode, the measured pressure drop would approach the equilibrium value with a time constant ($\tau = RC$, Table 1), where $C (= C_o V_o)$ is the upstream capacitance of the pump, R is the flow resistance ($R = \Delta P/q = \mu \ell / kA$), μ is the fluid viscosity, ℓ is the plug length, k is the permeability, A is the cross sectional area of the plug, V_o is the pump volume, and C_o is the oil compressibility. For an exponential variation, 3 to 5 time constants have to pass before the pressure drop can be accepted as an equilibrium value. For a core of 1,000 nD, the time constant is 1 hour, so a permeability measurement could be conducted after waiting for a few hours. However, for tighter cores with 100 nD permeability, the wait period increases to 2 days, and the measurements become experimentally impractical if a large throughput of such measurements is desired. Also, when we begin a permeability measurement on a core plug in a lab, we do not know whether the permeability is 10 nD or greater than 1,000 nD. Therefore, the experimental protocol has to be designed to accommodate response of the rock assuming the lowest measurable permeability.

CT scanning of the core plugs to select intact plugs for permeability measurements

Tight liquid reservoirs have a wide range of natural fracture density. Coring and core handling processes may induce additional fractures due to depressurization and drying of the core. Therefore, in this work, we measure permeability of intact rocks, *i.e.*, rocks with no fractures detected down to about 15 μm , which is the resolution of our CT apparatus. We could not directly visualize whether there were any fractures smaller than 15 μm . However, the measured permeability values were too small for fracture dominated flow.

We believe that permeability measurements on such intact rocks will characterize the “matrix” permeability of a reservoir. Permeability estimates obtained using well tests might be higher than core-based matrix permeability due to the presence of natural and artificial fractures in the reservoir. The initial production rate will be strongly influenced by the efficacy of the fractures. However, as these fractures are depleted, the matrix will support the production, and the matrix contribution will become increasingly important.

Darcy’s law and uncertainty in measured permeability

For laminar flow, the permeability, k , of a core plug with length ℓ , and cross-sectional area A , when a fluid with viscosity μ is flowing through the plug with volumetric flow rate q , when a pressure drop ΔP is applied across the length of the core plug is defined using Darcy’s law (Equation 2).

$$k = \frac{q\mu\ell}{A\Delta P} \quad \text{Equation 2}$$

In the lab, we measure the viscosity (μ) of the fluid and dimensions of the core plug. The flow rate (q) is computed from the slope of cylinder volume of a computer-controlled pump vs. time, and the pressure difference is measured using pressure transducers. Out of all the measured physical quantities, the uncertainty in the measured flow rate is generally the main source of uncertainty. At very low flow rates corresponding to permeability ≤ 30 nD, the motion of a pump barrel due to thermal expansion of the fluid in the pump barrel due to small temperature fluctuations in the lab become significant. Therefore, for extremely tight samples, longer measurement times are required to improve confidence in the measurements.

Applied stress state during tight rock permeability measurements

In the reservoir condition, rocks are subjected to three principal stresses along three orthogonal directions, and the stresses are not generally equal in magnitude. In this work, we use a hydrostatic sleeve pressure (σ_s). The net confining stress (NCS) can be estimated using the sleeve pressure (σ_s), mean pore pressure (P_p), and the Biot coefficient (η) using the Terzaghi relationship (Equation 3) [12].

$$\sigma_{NCS} = \sigma_s - \eta P_p \quad \text{Equation 3}$$

The Biot coefficient (η) depends on the mechanical properties of rock minerals and the rock itself. Few experimental measurements of the Biot coefficient on clay-rich shales indicate η varies from 0.5 to 1 depending on the mineralogy and overburden stress [13] [14] [15]. In this work, we assume the Biot coefficient to be 1. Therefore the net confining stress is calculated by taking the difference between the sleeve pressure and the mean pore pressure. We keep a constant mean pore pressure and change the sleeve pressure to quantify the effect of stress on permeability. So, even if our simplistic assumption of $\eta \sim 1$ is not accurate, our reported NCS values will be off by a constant offset given by $(1 - \eta)P_p$.

Effective sealing of tight rock plugs with rubber sleeves

Tight oil bearing core plugs have low permeability, and as a result, even a miniscule leak between the rubber sleeve and the rock surface can create a significant bypass for the fluid and it will result in anomalously high values of measured permeability. To avoid this bypassing, we use Viton rubber sleeves of 1/16th inch thickness with 75 durometer and apply a sleeve pressure of at least 2, 000 psi higher than the average pore pressure. Using this technique, we have measured practically zero (< 10 nD) permeability for some core plugs indicating that our method provides adequate sealing.

Minimization of leaks during permeability measurements

Flow rates corresponding to permeability of tight oil bearing rocks are extremely small, in the range of 1×10^{-3} to 5×10^{-5} cc/min or less. These rates are comparable to leak rates during permeability measurements of conventional high permeability rocks. Therefore, special wrenches had to be developed to tighten fittings in order to get a leak rate

substantially lower than the flow rates during tight rock permeability measurements. In these experiments, leak checks were performed often, and leak rates corresponding to permeability < 10 nD were obtained before initiating the liquid flow through tight rocks.

Simplified permeability measurement setups during stress aging

The permeability of tight oil bearing rock samples was found to depend on applied stress, stress creep and stress history. The permeability changed over a time period of multiple weeks, so it became impractical to tie down computer-controlled pumps when the rock was still in a transient state. In order to enhance the permeability measurement throughput, stress aging stations were designed using simpler pumps, back pressure regulators (bpr) and balances. Core samples were subjected to appropriate overburden stress and pore pressures, and the permeability was tracked using daily change in the weight of a fluid containers attached to the upstream pump and to the outlet of the bpr. These stations provided a good estimate (within 50 to 100 nD) of how the permeability was changing with time. Once the permeability stabilized, the core plugs were transferred to computer-controlled pumps for accurate measurements of the steady state permeability.

Capillary standards for the validation of permeability measurement apparatus

We introduced capillary standards in order to validate low permeability measurements [7]. These standards are built using stainless steel cylinders of outer diameter D (1" or 1.5"), with a glass capillary embedded at the center with inner diameter d (5 to 50 μm , Figure 2). The permeability can be computed from first principles and it is given by

$$k_{theory} = \frac{d^4}{32 D^2} \quad \text{Equation 4}$$

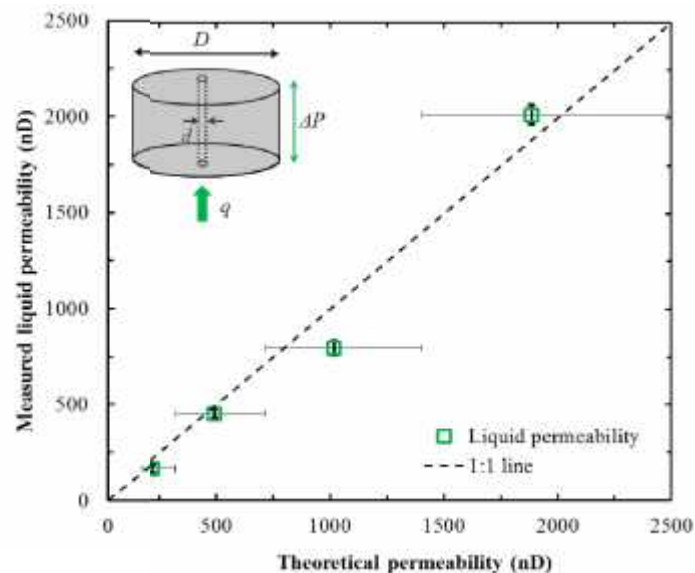


Figure 2. A comparison of measured steady-state liquid permeability values for standards against permeability computed using Equation 4 show a good agreement [9].

The permeability standards allow us to validate our experimental setup. The permeability of a standard is not affected by (1) the applied stress, as the stainless steel billet is relatively incompressible, and (2) the fluid used for measurements. The pore volume of a capillary standard is very low due to the small inside diameter of the bore, therefore permeability measurements of capillary standards can be done quickly. In Figure 2, measured liquid permeability for four capillary standards is plotted against the corresponding theoretically computed permeability [9].

The measurements agree well with the theoretical values, within the uncertainty corresponding to a tolerance of 1 μm in the internal capillary diameter (horizontal error bars). The uncertainty in theoretical permeability (δk) scales with the cube of the capillary diameter (d), and therefore, the horizontal error bars get progressively wider at higher d . The relative error in the permeability decreases with increasing permeability *i.e.* $\delta k/k \propto 1/d$. The vertical error bars are typically 10 to 30 nD, representing the standard deviation in measured permeability values. A successful validation of the experimental setup provides confidence that the setup is measuring correct permeability values for tight rocks. These capillary standards are being used by us and many industry colleagues to validate their tight rock permeability measurement setups.

RESULTS AND DISCUSSION

Effect of stress creep on liquid permeability measurements

Once an experimental apparatus is designed based on considerations outlined in the previous sections, we can accurately measure steady state liquid permeability and understand the effects of stress and stress creep. A continuous decline of permeability with time at constant NCS (termed stress creep) and the effect of applied NCS on the permeability is plotted for a core plug from the *Vaca Muerta* formation in Figure 3(a).

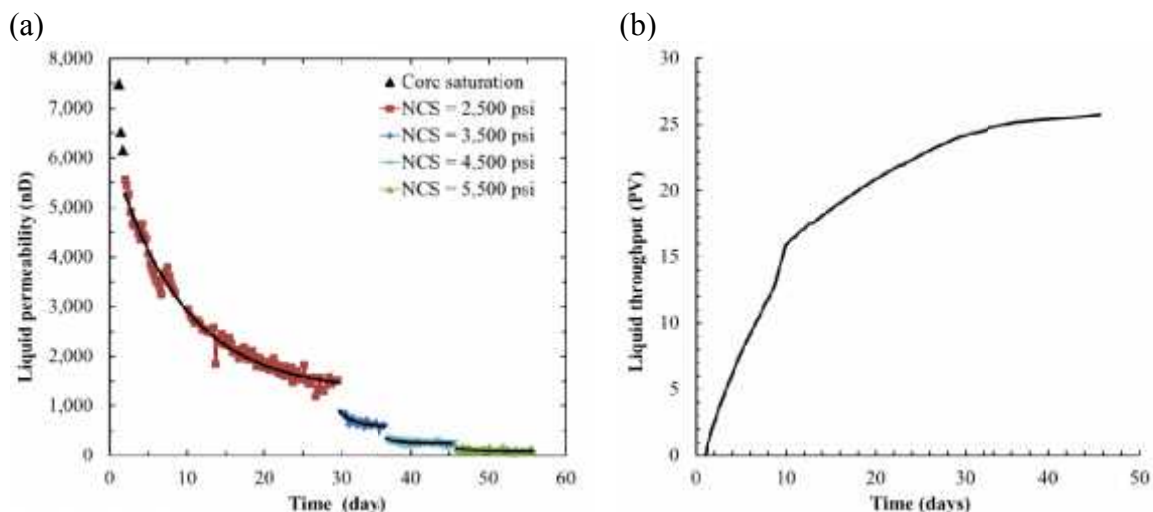


Figure 3. (a) Measured liquid permeability (nD) decreases with time at a constant net confining stress (NCS in psi), *i.e.*, creep, and the steady-state value of liquid permeability decreases with increasing NCS [9]. Liquid throughput in pore volumes vs. time for the same test is plotted in (b).

The first three data points (\blacktriangle in Figure 3(a)) are measured when less than one pore volume of toluene has been pumped through the core, so they are not considered as liquid permeability measurements. When liquid throughput is less than one pore volume (PV), some gas flow might occur in the core and the viscosity of in-situ oil might be different from the carried liquid. Over the next 30 days, numerous pore volumes of toluene flowed through the rock (Figure 3(b)), and the measured liquid permeability monotonically declined from 5,500 nD to 1,500 nD, at a constant NCS of 2,500 psi. When liquid throughput exceeded a couple of pore volumes, the mobile phase in the rock was almost pure toluene; therefore the fluid viscosity did not change appreciably with time. A model prescribing an exponential relationship of permeability with time provided good fit to the experimental data. The asymptotic value of this model was considered as the steady-state permeability for the rock at 2,500 psi. Change in measured permeability was not caused by increasing throughput of toluene or decalin, as long as throughput > 2 PV. After 30 days, NCS was increased from 2,500 to 3,500 psi, which resulted in an instantaneous drop in permeability from 1,500 to 850 nD. Further gradual decline with time at constant NCS was observed for higher NCS values.

If we had not waited for such a long time, we could have erroneously interpreted the steady state liquid permeability of this core plug at 2,500 psi as 7,500 or 5,500 nD; which is substantially higher than the asymptotic value of 1,290 nD. In the lab, we have accommodated this slow response of permeability with time at constant NCS by stress aging rocks for 4 weeks before measuring permeability. Permeability measurements on stress-aged samples are much faster and can be completed in a few days. The degree of stress creep was found to be a function of the stress history of the sample, as shown in Figure 4(a) and (b) for the first stress state and the subsequent stress states respectively.

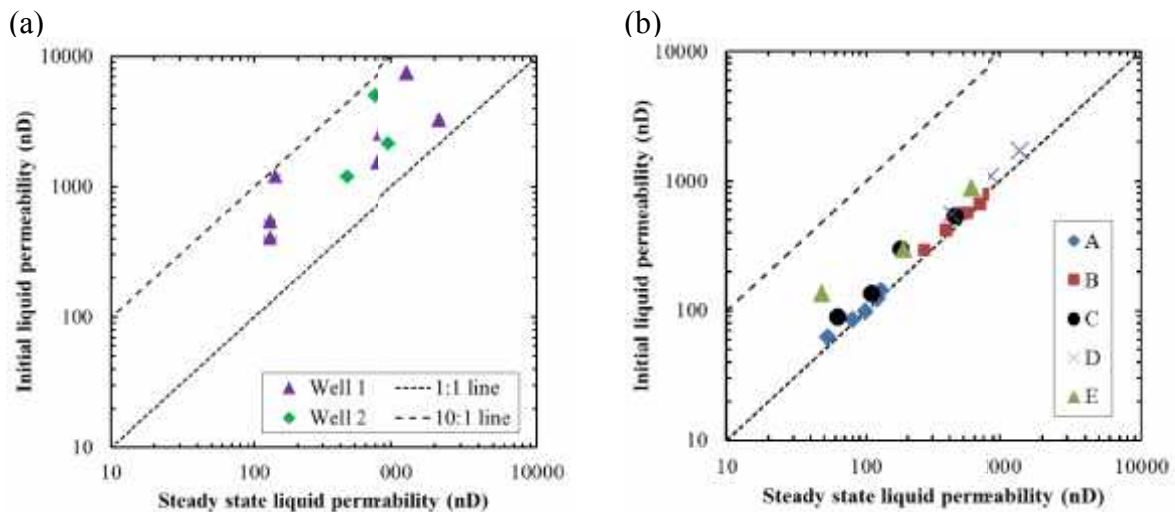


Figure 4. Initial liquid permeability based on first 6 to 24 hours after a sample is mounted vs. steady state liquid permeability for the same core plug under the same NCS are shown. In (a) the stress creep is more severe for the first stress state for 10 core plugs from two wells [9], as compared to (b) where subsequent stress states during drawdown for 5 core plugs from well 1 are considered.

Typically, the highest permeability declines were observed when samples were stressed for the first time, as shown on a cross-plot in Figure 4(a) for 10 core plugs from two wells from the *Vaca Muerta* formation. The initial liquid permeability is typically higher than the steady state permeability by a factor of 2 to 10, *i.e.*, the points lie between the dotted line (corresponding to 1:1) and the dashed line (10:1) in Figure 4(a).

For this data set, the first stress condition corresponds to NCS of 2,000 or 2,500 psi; whereas subsequent stress states consist of a reservoir drawdown (NCS-NCS_i) of up to 4,000 psi in steps of 1,000 psi. For subsequent stress states, the decline from “initial” to “steady-state” permeability values was lower, as indicated by the clustering of points closer to the 1:1 line in Figure 4(b). The core plugs were already aged to the first stress state (NCS = 2,500 psi), so the reduction in permeability from this “initial” state for the second stress condition (NCS = 3,500 psi) to the steady state value was less severe.

The reduction in permeability due to stress creep over a time scale of weeks indicates the importance of stress aging the sample before permeability measurement. Therefore, we recommend that tight oil permeability data be interpreted with care by considering

- (1) Whether the analysis was conducted on crushed-rock or intact samples
- (2) If the analysis was conducted on intact core plugs, were there any cracks in the plug
- (3) How much NCS was applied on the core plug and the stress history of the plug
- (4) Whether any back pressure was applied to compress or dissolve gas in the plug
- (5) Whether fluid throughput before reporting permeability was more than 1 PV, and
- (6) How much time was the plug aged before reporting steady state permeability

Effect of net confining stress on tight rock permeability

The previous section focused on the effect of stress creep on the permeability measurement. Whereas in this section, the focus is on how NCS affects steady state permeability, *i.e.*, once all the transient effects of stress creep have been eliminated. During production from a tight reservoir, the pore pressure is decreased substantially, while the overburden stress is not altered. As a result, the rocks experience higher NCS than the initial NCS before production started. Rock permeability decreases with increasing NCS, and this effect is more pronounced for tight rocks [13].

Steady state liquid permeability as a function of NCS during drawdown and buildup are plotted in Figure 5(a) and (b) respectively for core plugs from well #1 from the *Vaca Muerta* formation. A model prescribing an exponential dependence of permeability (k) against NCS provides good fit to the permeability data, both during drawdown as well as buildup (Equation 5).

$$\frac{k}{k_i} = e^{-\gamma(NCS - NCS_i)} \quad \text{Equation 5}$$

Steady state liquid permeability monotonically decreases with increasing NCS during drawdown, *i.e.*, for plug B (■ in Figure 5), 760 nD when NCS = 2,500 psi down to 260 nD when NCS = 6,500 psi. However, during buildup, the permeability only partially recovers to 440 nD at 2,500 psi. This permeability hysteresis with stress and partial recovery can be quantified by comparing the slopes of the drawdown and buildup lines

on the semi-log plots in Figure 5(a) and Figure 5(b) respectively. The magnitude of the slope of the best fit lines on a semi-log plot is termed as permeability modulus (γ , Equation 5). For core plugs A to E, permeability modulus was calculated to be 2×10^{-4} to 6×10^{-4} /psi for drawdown, and 1×10^{-4} to 2×10^{-4} /psi for buildup data.

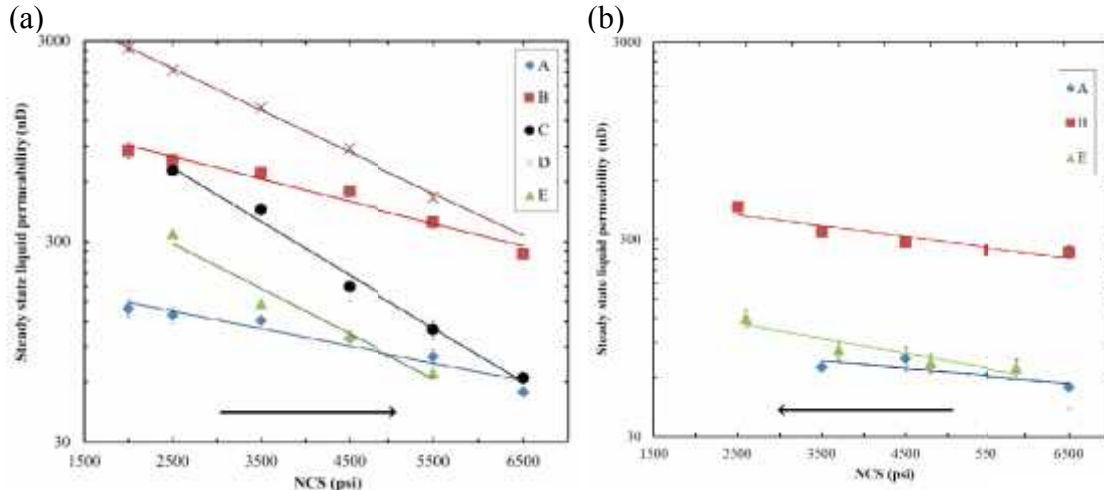


Figure 5. Measured oil permeability decreases with increasing NCS, and the extent of permeability impairment with stress is shown on a semilog scale during drawdown (increasing NCS) in (a) [9], and during buildup (decreasing NCS) in (b) for core plug samples from the same tight-oil well

CONCLUSION

In this work, various experimental core analysis aspects to measure liquid permeability for tight oil bearing rocks are outlined. Crushed rock permeability measurements do not provide measurements representative of the reservoir as the rock fabric is destroyed and no confining stress is applied during such measurements. A set of questions was posed to ensure quality control of permeability data on tight oil bearing rocks. An experimental apparatus capable of measuring low flow rates was commissioned in order to measure steady state liquid permeabilities from tens of microDarcies down to tens of nanoDarcies. Flow measurements using capillary standards were used to validate the apparatus, and these standards are available for use by others.

Steady state liquid permeability measurements from about $10 \mu\text{D}$ to 30 nD were performed at reservoir net confining stress, using inert hydrocarbons like toluene or decalin as the injectant. Gradual decline in permeability at constant NCS (stress creep) was observed, and experimental best practices to accommodate the creep using a simple stress-aging apparatus are outlined. Stress dependence on steady state liquid permeability was quantified using a permeability modulus. Strong permeability decline was observed with increasing NCS, and the permeability recovered only partially with decreasing NCS, *i.e.*, permeability hysteresis was observed.

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