TITLE PAGE

Title: Seismic and Rockphysics Diagnostics of Multiscale Reservoir Textures

Type of Report: Quarterly Technical

Reporting Start Date: October 1, 2003

Reporting End Date: December 31, 2003

Principal Investigator: Prof. Gary Mavko

Date of Report: February 2004

DOE Award Number: DE-FC26-01BC15354

Submitting Organization: Stanford University, 651 Serra Street, Suite 260, Stanford, CA
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ABSTRACT

As part of our study on “Relationships between seismic properties and rock microstructure”, we have studied relationships between clay cements and velocity. Acoustic waves were propagated through this arrangement of quartz discs and clay. We monitored water content by weighing. Pore size distribution was measured by NMR experiments. We report here waveforms of P- and S-wave signals transmitted through the air-coupled, dry clay-coupled, and cemented clay-coupled quartz discs. We find that the state of the clay changes the wave propagation characteristics in a very pronounced manner.

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INTRODUCTION

In this quarter, we submit results of our research on effects of clay cements on velocity. In an attempt to understand the effects of clay on wave propagation, we have investigated velocity changes in clay as dries in contacts.

PAPER 1

WAVE PROPAGATION AND NMR EXPERIMENTS IN CONTACT CLAYS

Paper 1: Total of seven figures in paper

WAVE PROPAGATION AND NMR EXPERIMENTS IN CONTACT CLAYS

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ABSTRACT

In this paper we present results of our experiments in clays and quartz discs. Two discs were placed in contact with and without anything in the contact zone and acoustic waves were propagated through them. The contact zone between the discs was filled with thin layers of dry, clay slurry that was allowed to dry slowly, and air. Acoustic waves were propagated through this arrangement of quartz discs and clay. We monitored water content by weighing. Pore size distribution was measured by NMR experiments. We report here waveforms of P- and S-wave signals transmitted through the air-coupled, dry clay-coupled, and cemented clay-coupled quartz discs. We find that the state of the clay changes the wave propagation characteristics in a very pronounced manner.

INTRODUCTION

Cementation of granular media brings about significant changes in microstructure, physical properties (e.g., porosity and permeability), and stiffness. These changes depend primarily on the cement type and its location. Dvorkin et al. (1991) have shown that the effect of cementation on the stiffness and elastic moduli of granular media can be modeled using idealized cement deposition schemes. Thus cement deposition in pore spaces reduces porosity and permeability significantly without changing elastic properties. On the other hand, even a small amount of cement deposited in contact zones increases the stiffness and elastic moduli significantly without a large change in porosity and permeability. This increase in stiffness and elastic moduli also depends on the type of cement. Experimental data on velocity measurements in clean and in clay-bearing sandstones by Han et al. (1986) has shown that, at same porosity, sandstones with quartz cement have higher velocity than those with clay cements. On the basis of the experimental data, Han et al. (1986) derived empirical relations for velocity variations with porosity and clay content.

Clay minerals can affect acoustic impedance considerably. du Bernard and Prasad (2003) have shown that the microstructural impedance is lower in the clay contacts in sandstones. They found a direct relation between ultrasonic impedance and microstructural impedance measurements in clean and in clay-bearing sandstones. Prasad et al., 2002 found that significant signal damping occurs after applying a 0.05 mm thick clay layer in the contact region between glass discs. The effect is more pronounced in the S-waves than in the P-waves.

This study was conducted to understand the mechanisms behind clay cementation in rocks. Specifically, we have investigated changes in acoustic properties as the clay dries and changes from a damping to a cementing agent and correlated them to changes in internal pore structure of the clay. We present the results of wave propagation experiments through an idealized contact between two quartz discs with and without clay in the contact zone. We find that addition of a water saturated clay layer to quartz discs dampens waves significantly. However, as the clay dries, it acts as a strong cementing agent and enhances wave propagation through the clay-cemented quartz discs.

METHODS

The measurements were made on two optical quality quartz discs of 25.4 mm in diameter and about 12.3 mm in length. The surface of the discs was finished to 1/10 of a wavelength. At the frequency of measurements, for all practical purposes, the contact between the discs was a perfect contact. The samples used were dry kaolinite powder and kaolinite powder mixed with distilled water. Three types of contact conditions were simulated between the two quartz discs. Table 1 gives a summary of all the experiments reported in this study.

1. In the first test, the discs were air-coupled, that is, they were placed in contact without any couplant between them. These tests were denoted as “air-coupled”. Note that the term “air coupled” refers to the contact between the two quartz discs. It does not refer to the contacts between the discs and transducers; these were coupled with a high-viscosity bonding medium in all tests.
2. The second test was made with dry clay in the contact. A small amount of sieved kaolinite powder was placed on one of the quartz discs and the other disc was placed on top of the clay. There was no measurable change in length of the assembly on addition of the clay powder. These tests were called “dry clay-coupled”.

3. The third test was made with clay – water slurry placed in the contact. The slurry was prepared by mixing measured quantities of sieved kaolinite powder with distilled water. The quartz – clay assembly was sealed with heat-shrink tubing put over two o-rings to ensure that no water could escape during the acoustic and NMR measurements. The water content was monitored carefully by weighing the cells with an analytical balance that had a repeatability of ± 0.1 mg. These tests were called “cement clay-coupled”. A control acoustic test was run with wet slurry in the contacts. Acoustic measurements were made as the clay slurry dried with time. The transducers were not removed during this test run to make the NMR tests and the water content was not monitored. The test run was made to check repeatability of the acoustic coupling between the quartz disk assembly and the transducers.

Table 1: List of the measurements and samples used in this study

Sample	Acoustic	NMR
Dry quartz discs	Air coupled	
Dry clay	Dry clay coupled	As powder
Wet clay		As slurry
Wet clay in contact allowed to dry with time	Sample A	Sample A
	Sample B	Sample B
	Sample C	

During drying of the clay slurry in the cells, the following steps and time required for each step were observed carefully to ensure repeatability:

1. The sample was weighed to record the amount of water lost.
2. Acoustic measurements at 1 MHz – these typically took about 30 minutes to complete.
3. NMR measurements – depending on water content in the slurry, these measurements took between 1 – 6 hours.
4. The sample was weighed again after the NMR measurements to ensure that no water was lost during Step 3.
5. Acoustic measurements were repeated to ensure that the signal did not change in during Step 3.
6. The jacket was perforated and cells placed in oven again for drying.

Ultrasonic Measurements

The pulse transmission technique (Birch, 1960) was used for V_p and V_s measurements. The experimental setup consisted of a digital oscilloscope and a pulse

generator. The sample was jacketed with heat-shrink tubing to allow controlled drying of the contact slurry. 1 MHz Panametrics P- and S-wave transducers were used for the pulse-transmission experiments. A high-viscosity bonding medium (Panametrics SWC) was used to bond the transducers to the sample. Travel-time was measured after digitizing each trace with 1024 points at a time sweep of 5 μ s, thus allowing a time resolution of about 5 ns or about 0.2% error in velocity. Actual error in velocity measurement is estimated to be around 1% due to operator error in picking first arrival. The system delay time was measured by taking head-to-head time. The system delay time was confirmed by measuring a standard aluminum cylinder.

NMR Experiments

T2 relaxation curves were collected for two kaolinite-water slurries between quartz discs to provide information about the pore-scale location and size of the water in the clay as the samples dried. In each sample a thin layer of clay slurry (sample A: 38.2 mg clay + 21.9 mg water; sample B: 27.9 mg clay + 26.6 mg water) was spread between two quartz discs. Unfortunately, the heat-shrink sealing process partially dried the samples, so it was not possible to collect NMR data on the thin clay layers when they were fully saturated. A separate slurry was prepared for NMR with 0.3451 g clay and 0.4383 g water to approximate the initial condition of the clay between the quartz discs; this sample is referred to as the standard. NMR measurements on the standard slurry were carried out using a Teflon cell of known volume. T2 data were also collected for a 46.5 g sample of the 'dry' clay containing only residual bound water. NMR data were collected for partially dried sample A with water contents of 0.55 and 0.39. The sample B dried at a faster rate, so NMR data were collected only with water content of 0.17.

T2 data were collected using a Maran Ultra NMR spectrometer at 2 MHz. The standard CPMG pulse sequence was used with 1600 echoes and a 0.2 ms echo spacing between points. To maximize the signal-to-noise ratio, 30,000 to 60,000 scans were stacked for the samples with the quartz discs; 5000 scans were stacked for the standard. The relaxation delay between scans was 0.5 s. The noise in the data was determined from the last 4% of the decay curve, where the signal has decayed to zero. To reduce computing time for inverting the decay data, the 1600 linearly spaced points were averaged to create 237 points (approximately equally spaced on a log scale). Since the o-rings contributed slightly to the NMR signal, T2 data were also collected for the rings alone; all NMR data sets for samples A and B were corrected for this contribution.

NMR Data Processing

NMR relaxation data can be described as a sum of exponential functions. The equation describing multi-exponential relaxation is

$$I(t_i) = I_i = \sum_{j=1}^M a_j e^{-\frac{t_i}{T2_j}}, \quad i = 1, 2, 3, \dots, N, \quad (1)$$

where the N data I_i are measured at times t_i , and a_j is the unknown amplitude of the component at relaxation time $T2_j$. A set of M $T2_j$ values is chosen to contain the expected values of $T2$; M is 200, large enough not to bias the solution into a small number of relaxation times. We used a Tikhonov regularized least squares algorithm (referred to as the L2 fit) implemented by Whittall and Mackay (1989) to invert the data.

Equation 1 is a Fredholm equation of the first kind, and thus has no unique solution (e.g., Istratov and Vyvenko, 1999). Many models – which can be quite different from each other – fit the data equally well. For decay curves with low signal-to-noise ratio we found it prudent to explore the extent of non-uniqueness to avoid the pitfalls of over-interpreting a single relaxation time distribution constructed by a single inversion algorithm. We used a weighted linear programming (LP) technique (Whittall and Mackay, 1989) to obtain relaxation time distributions which either maximized or minimized amplitudes within localized intervals of relaxation times. The LP method gives the minimum amplitude required and the maximum allowed for the relaxation times within the interval. These bounds are not approximations; they are absolute bounds on the set of all relaxation time distributions having an equal or smaller misfit to the data. We used this method to communicate a sense of how much variation was possible for the relaxation time distributions obtained for our samples.

In addition, the pore size distribution was calculated from the $T2$ distribution using the following relationship between $T2$ and surface area – to – volume ratio (S/V) of the water-filled pore:

$$\frac{1}{T2} \approx \frac{1}{3} + \rho_2 \left(\frac{S}{V} \right), \quad (2)$$

where ρ_2 , the surface relaxivity, is a measure of the ability of the clay surface to enhance relaxation. Note that the surface relaxivity of kaolinite could be calculated using the $T2$ data from the standard slurry sample because S/V was known (i.e., calculated from measured porosity, clay grain density, and specific surface area). For the samples in

which the pores are not fully saturated with water, ρ_2 remains constant and the effective size of the water layer (d) in the pores can be estimated as $6/(S/V)$.

RESULTS

Figure 1 shows the experimental setup along with a comparison of P- and S-waves through the quartz discs with a couplant and with a wet-clay layer between them from Prasad et al., 2002. The signals displayed show signal damping after applying a 0.05 mm thick clay layer in the contact region between the glass discs. The effect is more pronounced in the S-waves than in the P-waves. The amplitude of both P- and S-signals decreases significantly, in particular amplitude of the S-signal.

Figure 1: P- and S-waves through glass discs as shown on the right (from Prasad et al., 2002). Gray lines depict signals traveling through glass only and black lines represent signals through two glass discs separated by a 0.05mm layer of clay. Signals deteriorate on addition of clay to the contact area between the glass discs. The shear wave marked by a thin black line was made with the transducers at cross-polar position to ascertain shear arrival (thick black line).

The tests in this study were made to investigate the transition stages in clay cementation of contact surfaces. Figures 2 and 3 show P- and S-wave signals from the cement clay-coupled tests. In these tests, the acoustic signals were propagated through the quartz discs with wet slurry in the contacts. The slurry was slowly allowed to dry out. There is a dramatic change in signal quality as the slurry dries out. The amplitude of both P- and S-waves increases as the slurry dries. S-waves are not recognizable for large water content in the slurry. The signals displayed in Figure 2 were measured while keeping the disc-transducer assembly undisturbed, so as to ensure a constant coupling with the transducer. In Figure 3, the signals were measured before and after subsequent weighing and NMR measurements. Thus, the disc-transducer assembly was removed to allow the water content measurements and a fresh couplant was applied before each measurement. The similarity between signals in the two figures shows that effects due to a change in coupling between disc and transducer are negligible.

(a)

(b)

Figure 2: (a) P- and (b) S-wave signals through the quartz cells with clay slurry in the contacts measured with the transducers in place. The different signals are for various stages of drying marked by increasing numbers. As drying progresses, P-waves get attenuated and S-waves become more prominent showing competing effects of partial saturations in P-waves and cementation in S-waves. The arrow marks the signal that shows cementation effects recognized by an increase in P-wave amplitude and onset of S-wave propagation.

(a) (b)
Figure 3: (a) P- and (b) S-wave signals through the quartz cells with clay slurry in the contacts. The different signals are for various stages of drying and correspond to the NMR measurements.

Figure 4 shows P- and S-wave signals from the dry clay-coupled tests. In these tests, the acoustic signals were propagated through the quartz discs with a thin layer of dry clay powder in the contacts. Although the waves are still propagated through the discs there is a marked decrease in signal quality. S-waves are difficult to recognize and both P- and S-waves are greatly reduced in amplitude. Furthermore, the signals are shifted towards lower frequency content.

(a)

(b)

Figure 4: (a) P- and (b) S-wave signals through the quartz cells with dry clay in the contacts.

Figure 5 shows P- and S-wave signals from the air-coupled tests. In these tests, the acoustic signals were propagated through the quartz discs with only air in the contacts. Although the waves are still propagated through the discs there is a marked decrease in signal quality. Just as in Figure 4, S-waves are difficult to recognize and both P- and S-waves are greatly reduced in amplitude.

Figure 5: (a) P- and (b) S-wave signals through the quartz cells with clean contacts.

Next, we compare the signals from all three, the air-coupled, the dry clay-coupled, and the clay cement-coupled tests. P-waves are shown in Figures 6 and S-waves in Figure 7. Although both clay layers have comparable water contents, the clay cement-coupled tests show much higher signal quality as compared to the dry clay-coupled tests. Thus there is a difference in the cementation strength of clay minerals when they are deposited as slurries on a substrate and allowed to dry. The cements formed by this deposition are stronger than those generated by dry clay powder in the contact. Also, the clay cement appears to be significantly more competent for S-wave propagation (Figure 7b) than for P-waves (Figure 6b).

(a)

(b)

Figure 6: Comparison between air, dry clay, and cement clay in the contact for P-wave signals through the quartz cells. (a) Air coupled (black) and dry clay coupled (red) contacts and (b) cement clay coupled. The air- and dry clay couples signals are quite similar and much lower in energy as compared to the clay cement signals.

(a) (b)
Figure 7: Comparison between air, dry clay, and cement clay in the contact for S-wave signals through the quartz cells. (a) Air coupled (black) and dry clay coupled (red) contacts and (b) cement clay coupled. The air- and dry clay couples signals are quite similar and much lower in energy as compared to the clay cement signals.

COMPARISON WITH NMR RESULTS

The T2 distributions (L2 fits) for the extreme cases of the clay-water slurry and ‘dry’ clay are shown in Figure 8. In each case, the signal-to-noise ratio of the relaxation data was high (107 and 83 respectively) so there is little ambiguity in the interpretation of the T2 distributions. The approximate size of the water layer within the pores is shown on the upper horizontal axis. The ‘dry’ clay contains residual water (~3 monolayers) on the silicate surface approximately 1.5 nm thick and the pore size in the slurry is approximately 0.6 μm. Note that the bound water in the slurry is not visible in Figure 8 at this scale. Thus the discs with the clay-water slurry can be considered to be water coupled. P-wave signals are thus strongest in the initial stages and no shear signals are propagated in Figures 2 and 3 (signals 1S00 and 2S00, respectively).

Figure 8: The T₂ relaxation distribution and estimated size of the water layer within the clay for the initial clay slurry and the dried clay containing only bound water. The vertical scale of the dry clay has been expanded to facilitate comparison of T₂ values.

In comparison, the T₂ distribution for the cement layer in sample A (water content = 0.55) is shown in Figure 9. The clay layer contains a wide range of sizes for the pore water intermediate to those of the slurry and surface bound water; the average water layer size is 56 nm. In sandstones, water with relaxation times greater than 33 ms is considered to be free water (i.e., moveable); water with T₂ less than 33 ms is considered to be “capillary bound” water associated with clays (Straley, et al., 1997) – this cutoff is shown for reference in Figure 9. Most of the water in sample A is either capillary water or surface bound water.

Figure 9: The T₂ relaxation distribution and estimated size of the water layer within the clay for partially dried sample A (water content = 0.55). The T₂ cutoff of 33 ms (~0.2 μm) for capillary bound water is shown for comparison.

Considering that the decay curve for sample A was relatively noisy (S/N ~ 20) we found it prudent to explore the extent of non-uniqueness to avoid the pitfalls of over-interpreting the structure shown in Figure 9. Figure 10 shows the minimum required

amplitude and maximum allowed amplitude for the T₂ distribution of sample A within T₂ intervals for free (> 33 ms) and capillary bound water (1.8 – 33 ms). The surface bound water (< 1.8 ms) does not show at this scale. Notice that the minimum required amplitude (red line) for T₂ > 33 ms is zero. This indicates that there might not be any water in sample A with thickness greater than 0.2 μm. In fact, the capillary bound water for sample A with water content 0.55 could account for 27 – 100% of the water present (see Table 2).

Figure 10: The minimum required (red) and maximum allowed (blue) amplitudes for T₂ values > 33 ms (free water) and T₂ = 1.8 – 33 ms (capillary bound water). The amplitudes (black) within the same intervals obtained from the L2 fit (in Figure 6) are shown for comparison.

Table 2: Pore Water Location and Thickness Determined from NMR Measurements

Sample	Water Content	Average Water Thickness (nm)	Fraction as Capillary Water
slurry	1.27	600	0 – 6%
A	0.55	56	27 – 100%
A	0.39	56	34 – 100%
B	0.17	7	29 – 97%

Table 2 shows the average water layer thickness and the fraction of pore water with thickness less than 0.2 μm (i.e., T₂ < 33 ms) as a function of water content of the drying clay cement. The average thickness did not change (~ 56 nm) as sample A dried from 0.55 to 0.39 water content, but was only 7 nm for sample B with 0.17 water content. The fraction of pore water with thickness less than 0.2 μm was relatively constant for all three samples.

The acoustic signals corresponding to these water contents are shown in Figure 11. Note the 5-fold increase in the shear signals and the increasing P-wave amplitude in the

Sample B with water content = 0.17. It is possible to recognize onset of shear waves in Sample A with 0.39 water content, however the signal is weak and not very prominent.

Figure 11: Acoustic signals corresponding to the NMR measurements shown in Table 2. The sample name and its saturation are given in the legend. A definite shear signal is measured in the sample with 0.17 water content.

ACKNOWLEDGEMENTS

M.P.'s work was performed under the auspices of the National Science Foundation (Grant No. EAR 0074330) and the Department of Energy (Award No. DE-FC26-01BC15354).

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