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Assessing Tensile Strength of Unconventional Tight Rocks Using Microwaving

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1. Abstract

Rock tensile strength is a critical parameter needed to design and model hydraulic fracturing (or crack initiation and propagation) in oil and gas reservoirs, especially in tight organic-rich shale. Conventional methods for measuring rock tensile strength are either time consuming (direct tensile strength method) or unreliable (indirect method), and require precision "machined" samples and heavy mechanical instruments. We propose a new technique that is simple and rapid for measuring the tensile strength of rocks with low permeability using microwave heating.

Our method is developed based on the fact that when the temperature of water in a confined space, e.g., within a tight rock, increases, water cannot freely expand and consequently the pressure within water-filed pores quickly elevates to the point where it exceeds the rock's tensile strength such that the rock breaks. Microwave heating can rapidly increase the temperature of water in the rock sample due to the relatively large dielectric loss of innate water. This method works well for rocks with low permeability where pressure leak off during the rapid heating is negligible. This paper presents both the theory and results of our initial laboratory tests for the microwaving heating method.

The test results demonstrate that microwaving cracks and sometimes pulverizes shales and tight sandstones with water content. When the sample is pulverized, likely 100% of the light hydrocarbons will be released. Hence, the proposed method also may provide insight into total recoverable light hydrocarbons per kilogram. We believe that microwave (or electromagnetic wave (EM)) heating approach, for some tight reservoirs, has the potential to be further developed to provide a practical waterless fracturing technology.

2. Introduction

Tensile strength is the maximum stress a rock can withstand when stretched before failing. It is a key parameter in designing hydraulic fracturing to produce unconventional reservoir. Mechanical test on natural rock samples always involves large instruments and complex procedures in part because significant force is required to break the rock. The tensile strength of rock is usually measured with a Brazilian Test. In this paper, we propose a new and relatively simple method to measure rock tensile strength using microwave heating. Microwave heats matter with large dielectric losses; therefore, it can rapidly raise the temperature of water in a core sample. As a result, the water pressure is elevated to the point where the rock breaks, the tensile strength. This method works for rocks with low permeability because the pressure leak off from the rock during the rapid heating is negligible.

In this paper, we first establish a theoretical treatment including the important factors that determine the pressure of trapped water when temperature is raised. We will then present some initial experimental results. Finally we discuss the potential of using microwaving as a technology to fracture tight rock by heating the water to elevate the pore pressure in water-filed pores.

3. Method

3.1 Concept Outline

When water is heated, the temperature increasing leads to pressure and/or volume change. As water is heated in tight rocks of low permeability, its volume is confined by the rock matrix and thus cannot expand significantly. As the result, the water pressure increases rapidly. When the water pressure reaches the rock tensile strength, the rock fails. Therefore, we can use this concept to estimate the tensile strength of tight rocks. To calculate the water pressure in the rock pores, we also need to account for the compressibility of rock matrix since any equilibrium state is a balance between the confined water and rock matrix.

3.2 Equation of State for water

The pressure increase attributed to heating water in the rock sample is obtained from the equation of state (EOS). The EOS for water can be expressed as:

$$z = \frac{p_w V_w}{R T_w} \tag{1}$$

With R = 0.461526 kJ·kg^{-1}·°K^{-1} is the specific gas constant of ordinary water (Cooper, 2007), T_w is the temperature in degrees Kelvin, p_w is the pressure in MPa, and V_w is the volume of water in m^3. It is easier to use molar density of water in our calculation, so we convert Eq. (1) to

$$z = \frac{p_w}{R T_w \rho_w} \tag{2}$$

where ρ_w is the density in kg/m^3.

As a most important substance, water has been thoroughly studied and many different formulations for water EOS have been proposed. Here we use the EOS published by the International Association for the Properties of Water and Steam (IAPWS) (Cooper, 2007; Wagner and Pruß, 2002) based on experimental data, covering the following temperature and pressure ranges:

$$0°\text{C} \le T \le 800°\text{C}, \quad p \le 100\ \text{MPa} \tag{3a}$$

$$800°\text{C} \le T \le 2{,}000°\text{C}, \quad p \le 50\ \text{MPa} \tag{3b}$$

These temperature and pressure ranges cover the full range of application and experiments discussed and anticipated here. The IAPWS formula for Eq. (2) contains several dozens of parameters and is generally handled using a computer program. We developed a computer program using Matlab for this purpose based on an open source program (Holmgren, 2006). The program can calculate any required property for water in the pressure and temperature range defined by (3a) and (3b).

The EOS of water in small pores may deviate from the above equation. The correction requires significant amount of experimental data to evaluate. Thus, we use the EOS discussed above in this study, which is not expected to introduce significant errors for practical applications.

3.3 Pressure increase with constant water density

First we consider a simple or ideal scenario where the rock is strong enough so that rock does not deform under an elevated water pressure. In this case, the water volume in the pore does not change and the density of water remains constant. The net outcome of temperature increase is to raise the water pressure. This temperature-dependent pressure can be obtained from the water EOS directly at constant water density.

The calculated pressure build-up according to temperature is shown in Fig. 1, with initial condition of water at room condition of 1 ATM and 20°C. In this ideal situation, the pressure increases rapidly. The tensile strength of shale rock ranges between 300 and 1,600 psi (Abbas et al., 2015; Lin, 1983). From Fig. 1, it can be seen that the pressure

reaches 1000 psi when the temperature increases only approximately 10°C. We note that for an infinite bulk modulus the pressure increase does not depend on the volume of water in the rock as long as the water filled porosity is not zero.

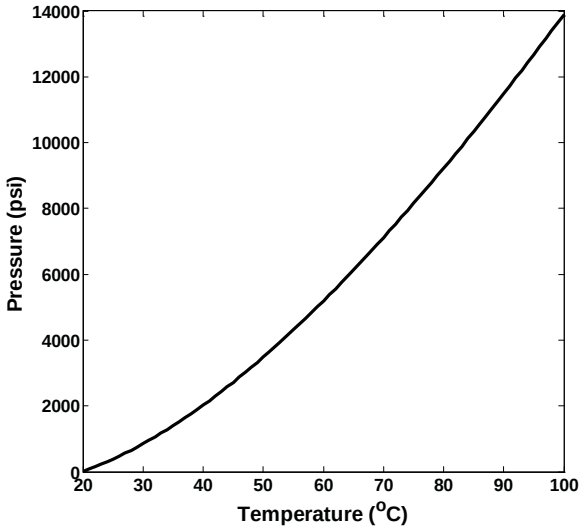

Fig. 1. Temperature-dependent pressure for water in a rock without leak off and volume expansion with initial condition at 1 ATM and 20°C.

3.4 Pressure change in compliant rock

In this section, we consider that the rock compressible under the pressure exerted by the heated water. This allows the water to expand when heated. The rock compressibility and the associated water expansion reduce the rate of pressure build-up due to the water heating.

Fig. 2 illustrates the scheme how the elevated water pressure in multiple pores compresses the rock matrix. All the rocks considered in this paper are tight with fine grains and small pores. The perturbations to the *in situ* stress field due to the presence of the water-filled pores are localized to within a few radii of the pore (Jaeger et al., 2007) . Therefore, change in the bulk rock volume owing to the elevation of the pore water pressure can be practically ignored. In this case, the volume change of the rock matrix caused by the water compression should be equal to the expansion of the trapped water volume, or

$$dV_m = -dV_w \tag{4}$$

where d indicates the volume change; V_m and V_w are the volume of matrix (solid phase) and water, respectively. The pressure exerted on the rock matrix, p_m, equals the pore pressure from the water, p_w. The differential form of this equality is

$$dp_m = dp_w \tag{5}$$

Under the pressure, the volume change of the rock matrix depends on the matrix (solid phase) modulus K_m as

$$dV_m = -\frac{V_m^0}{K_m} dp_m \tag{6}$$

where V_m^0 is the initial rock solid-phase volume, i.e., the rock solid-phase volume at time zero. From Eqs. (4), (5) and (6), we obtain

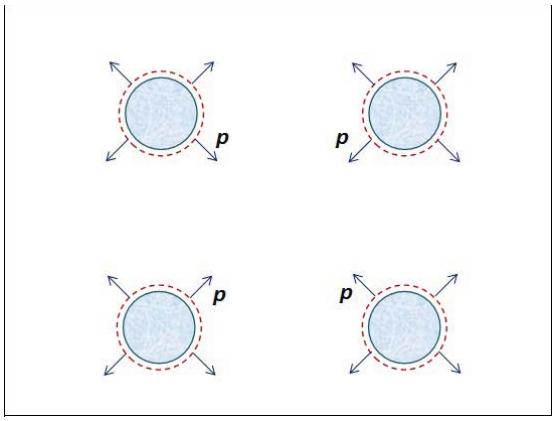

Fig. 2. A schematic illustrating the effect of elevated pore pressure due to the expansion of water on the compressible rock matrix. Dashed circles depict minor pore expansion.

$$dp_w = \frac{K_m}{V_m^0} dV_w \tag{7}$$

Using the following relationships

$$V_w^0 = x_w V_r^0 \tag{8a}$$

$$V_m^0 = (1 - x_w) V_r^0 \tag{8b}$$

where x_w is the water content defined as the volumetric fraction of water (This includes water in pores as well as water in shale interlayers and may not be equal to the water saturation in the "conventional" pore space.) to the total rock and V_r^0 is the initial bulk volume of a rock sample. Eq. (7) becomes

$$dp_w = \frac{K_m}{(1 - x_w) V_r^0} dV_w . \tag{9}$$

Further using

$$dp_w = p_w^t - p_w^0 \tag{10a}$$

$$dV_w = V_w^t - V_w^0 \tag{10b}$$

where the superscript t and 0 indicate time t and time 0, Eq. (9) becomes

$$p_w^t = p_w^0 + \frac{K_m x_w}{(1 - x_w)} \left(\frac{V_w^t}{V_w^0} - 1\right). \tag{11}$$

The water volume can be converted to density and Eq. (11) becomes

$$p_w^t = p_w^0 + \frac{K_m x_w}{(1 - x_w)} \left(\frac{\rho_w^0}{\rho_w^t} - 1\right). \tag{12}$$

Eq. (12) shows that the pressure of the heated water depends on the water density, the water volume fraction and the bulk modulus (specifically, the rock matrix solid-phase modulus). The water density is a function of temperature and pressure according to Eq. (2). Therefore, we can combine Eq. (12) and Eq. (2) to obtain the water pressure at a given temperature when the rock bulk modulus and water content are known.

Again, the analytical form of Eq. (2) contains many parameters; hence, a Matlab program was developed to solve Eqs. (2) and (12) using bisection method (Arfken and Weber, 2013). The calculation error was set to be within one psi for pressure.

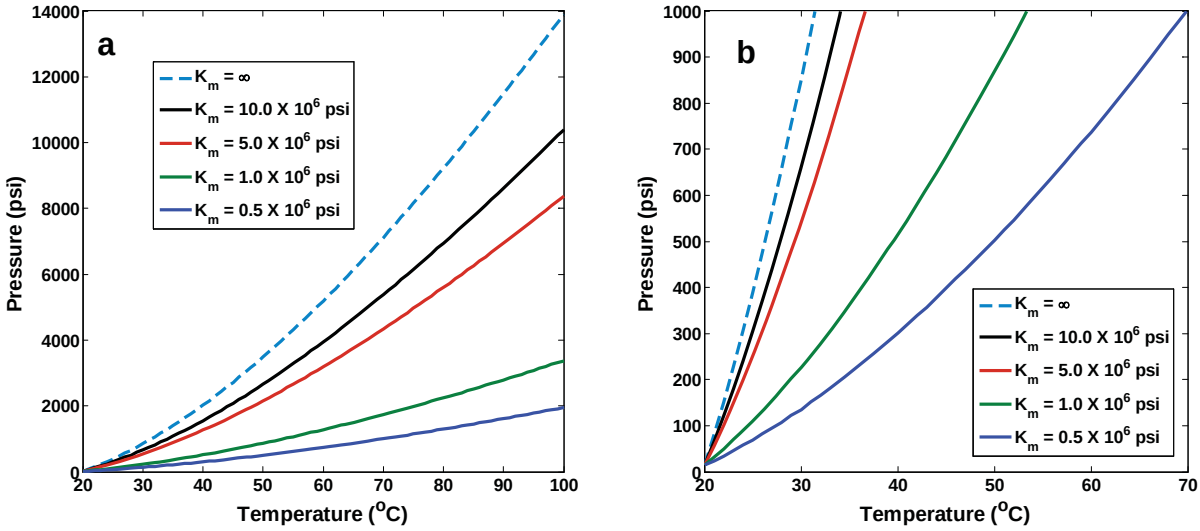

Fig. 3. Pressure dependence on the temperature of the water inside the rock pores with different moduli as shown in the legend. Fig 3b is a blow-up to show the smaller pressure range. The total water volume is 10% of the bulk rock volume.

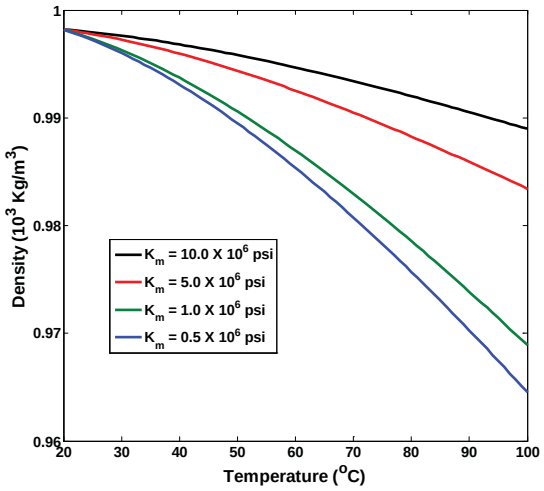

Fig. 4. The density of water in the rock pores versus temperature for different rock matrix moduli, corresponding to Fig. 3a.

Shown in Fig. 3a is the water pressure build-up for different bulk moduli for a rock with 10 p.u. water at initial condition of 1 ATM and 20°C. It indicates that the rate of pressure build-up significantly depends on the rock's modulus. Smaller bulk moduli allow the water volume to increase, and as a result, the pressure build-up with temperature increase is less. Fig. 3b shows an enlarged section of Fig. 3a which is relevant to our experiment. In Fig. 4, the density change for water in the pore is plotted according to the temperature, corresponding to Fig. 3. If the water volume is only 10% of the rock volume then; a 4% change of water density will only result in about 0.4% solid-phase volume change of the rock matrix.

3.5 Tensile strength measurement

This section presents the relationship of measuring tensile strength using elevated pore pressure and traditional test. The rock tensile strength measured using this method equals to the pore pressure at the point where rock fails by the pore pressure. On the other hand, in a traditional tensile test, a tensile stress equals to the largest stress a rock can withstand while being stretched before failing.

In unconventional rocks, the portion of water-filled pores is small; therefore, it is justifiable to ignore interactions between individual pores when investigating stress distributions near a pore. Also for simplicity, we study a two-dimensional problem with a pore being represented by a circle with radius R as illustrated in Fig. 5.

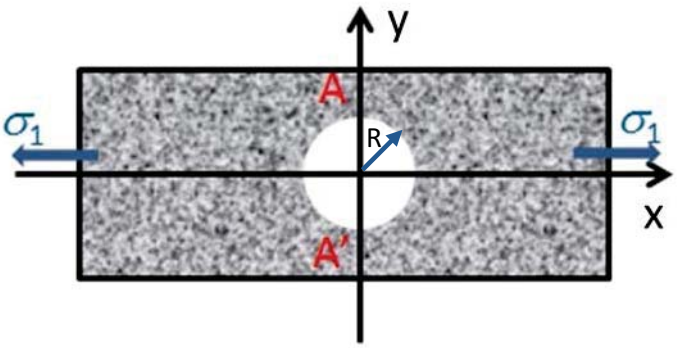

Fig.5. Stress distribution near a circular pore with radius R in an infinite two-dimensional medium under far-field tensile stress σ_1. The pore pressure inside the pore is p_w.

For the case shown in Fig. 5, the largest tensile stress occurs along y axis. Thus, we focus on tensile stress distribution along y axis, given by (Jaeger et al., 2007)

$$\sigma_x = \sigma_1\left(1+\frac{1}{2}\left(\frac{R}{y}\right)^2+\frac{3}{2}\left(\frac{R}{y}\right)^4\right)+p_w\left(\frac{R}{y}\right)^2 \tag{13}$$

The above equation is applied to the region $y \geq R$. It is obvious from Eq. (13) that the maximum tensile stress occurs at point A ($x = 0$, $y = R$) and A' ($x = 0$, $y = -R$) in Fig. 5 and is:

$$\sigma_x^{\max} = 3\sigma_1 + p_w \tag{14}$$

A rock sample under test fails when the maximum stress exceeds the "tensile strength" of the rock, the rupture stress, σ_c :

$$\sigma_x^{\max} = \sigma_c \tag{15}$$

which is an intrinsic property of the rock matrix.

For traditional tensile tests obtained with the "pulling" method, the tensile strength, S_{tr}, is determined (with no pore pressure, $p_w = 0$) and equals the stress σ_1 when the corresponding rock sample fails. Thus, we have from Eqs. (14) and (15),

$$S_{tr} = \frac{1}{3}\sigma_c \tag{16}$$

For the microwave heating tests, the tensile strength, S_{mw}, is determined (with no external forces, σ_1 = 0) from the pore pressure, p_w when the corresponding rock sample breaks. From Eqs. (14) and (15), we have

$$S_{mw} = \sigma_c \tag{17}$$

From Eqs (16) and (17), we obtain

$$\frac{S_{mw}}{S_{tr}} = 3 \tag{18}$$

Therefore, the tensile strength measured from microwave heating test, assuming round pores, is three times larger than that from traditional tensile tests. While Eq. (18) is obtained under an idealized condition, it indicates that rock tensile strength measured with microwave heating maybe significantly larger than that from the traditional measurements, as a result of different rock micromechanical models assumed for the two different test conditions. Note that the interpretation of the traditional "pulling" method is based on the continuum mechanics and an assumption of macroscopic homogeneity of a rock sample under test. Consequently, it does not consider the stress concentration near a pore when determining the tensile strength

3.6 *Microwaving tight rock samples*

Microwave radiation quickly heats materials with substantial dielectric loss. An estimate of power dissipated into the homogeneous materials is given by

$$P_{av} = \frac{1}{2}\omega\varepsilon_0\varepsilon_r^{''}\int_V E \cdot E^* \, dV \, . \tag{19}$$

Where P_{av} is the average power, ω is the microwave frequency, E is the electric field strength, E* is the conjugate of electric strength, $\varepsilon_0 = 8.85 \times 10^{-12}$ F/m, and $\varepsilon_r^{''}$ is the relative dielectric loss which is part of relative complex dielectric permittivity, ε_r^*:

$$\varepsilon_r^* = \varepsilon_r^{'} + i\varepsilon_r^{''} \, . \tag{20}$$

$\varepsilon_r^{'}$ is the relative dielectric constant. Eq. (19) is integration over the entire sample. If the rock sample contains several different minerals and water, to calculate the power adsorbed by the rock then one needs to consider all of the rock's constituents. If we know the fraction composition of minerals and their dielectric loss, Eq. (19) becomes

$$P_{av} = \frac{1}{2}\omega\varepsilon_0\sum_i(\varepsilon_{r,i}^{''}\int_V E \cdot E^* \, dV) \tag{21}$$

Where summation i is over all the dielectric loss components from minerals and fluids. The wavelength of microwaves we used is approximately 11 cm and is much larger than the mineral grain size and pore size; therefore, the electric field E over different component can be considered the same. As the result, integration over the space in Eq. (21) is the same for all the rock's constituents. This can significantly simplify the calculation and Eq. (21) becomes

$$P_{av} = \frac{1}{2}\omega\varepsilon_0\int_V E \cdot E^* \, dV\sum_i \varepsilon_{r,i}^{''} \tag{22}$$

Some typical dielectric constants are listed in Table 1 (Church et al., 1988; Peng et al., 2014; Shen, 1985).

Table 1. Relative dielectric constants of typical minerals in shale. The number listed in the parenthesis in the first column is the frequency the measurement was conducted.

Rock/Mineral	Dielectric Constant (ε_r')	Dielectric Loss (ε_r'')
20 Ohm Salt water[a] (1.1 GHz)	79	5.6
1 Ohm Salt water[a] (1.1 GHz)	77	21
0.1 Ohm Salt water[a] (1.1 GHz)	59	167
Dolomite[b] (1 GHz)	7.41	1.80E-03
Calcite[b] (1 GHz)	8.94	4.20E-04
Quartz[b] (1 GHz)	3.89	5.33E-04
Pyrite[c] (2.45 GHz)	83	17

[a] Shen, 1985; [b] Church, 1988; [c] Peng, 2014;

4. Experiments

Samples and preparation. Microwaving test were performed on a tight Tennessee sandstone, five Mancos outcrop shale plugs, and clay disks. Saturation of the dry Tennessee sandstone sample was achieved by first vacuuming the plug for more than 40 hours and then imbibing 2% KCl solution under vacuum condition. The plug was in the solution for a week before the microwaving experiment. The montmorillonite clay was from Panther Creek, Colorado. A clay disk was made by pressing the powder at pressure of 70,000 psi using an Angstrom 4451AE Briquet Press. No treatment was done on the Mancos shale plugs.

Water content determination. Water content was measured using low field NMR (2 MHz or 13 MHz) by comparing the measured NMR signal from the sample to known amount of water. The NMR signal was acquired using a CPMG spin echo method (Carr and Purcell, 1954; Meiboom and Gill, 1958).

Microwaving. A Monowave-300 microwave from Anton Paar was used for the majority of microwaving experiments. For all experiments on Monowave-300, a constant power mode with output power at 200 watts was used. The maximum microwave time was set to be 40 sec. The microwaving experiment was immediately terminated when the rock failure is detected. Rock failure was detected from the sound with an attached microphone to the microwave. The sample temperature was measured using an infrared thermometer.

Some qualitative microwaving tests were performed with a common household microwave (Hamilton Beach Household Microwave, Model P100N30AP_F4).

5. Results and discussion

Fig. 6. Tight Tennessee sandstone before and after microwaving. 20.1 seconds microwaving completely obliterates the rock plug.

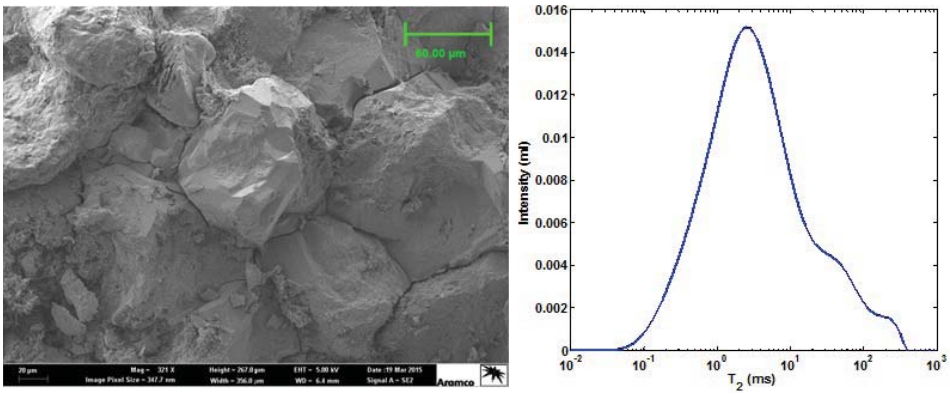

Fig. 7. SEM image of a snap-broken face of the Tennessee sandstone (see Fig. 6) and NMR T_2 spectrum of fluid in the sample before microwaving experiment.

Fig. 6 shows a tight Tennessee sandstone sample before and after microwaving. The grain size is approximately 60 to 70 μm from the SEM images (Fig. 7 left). The plug was initially dry and had no NMR detectable water content. After vacuum saturation, it contained 6 p.u. (percent unit, i.e. volume percent of water to the core plug) water based on the NMR measurement (Fig. 7 right). The NMR T_2 spectrum is shown on the right in Fig. 7. No permeability measurements were made, but the sample is extremely tight under visual and microscopic examination. Immediately after microwaving, the sample temperature was 51°C, measured with an infrared thermometer. The rock matrix contains 97.4% quartz and traces of muscovite and clay, from XRD analysis. Therefore, we use the quartz modulus $K = 5.4\times10^6$ psi (Jaeger et al., 2007) for the matrix modulus of this sample. Using this matrix modulus, a water content of 6 p.u., and the initial room pressure of 14.5 psi, we obtain the pressure build up shown in Fig. 8 for the temperature range of interest from 20°C to 100°C. We interpret the "tensile strength" of this sample to be the heated water pore pressure at 51°C of 1776 psi. This value is in the range of expected tensile strength for this type of tight sandstone.

The temperature read on the sandstone powder after pulverization probably represents a lower temperature than that of the water at the time the sample failed, because water is heated faster than quartz by the microwaves, and probably the quartz is not in thermal equilibrium with the water. Therefore, the tensile strength calculated using this temperature represents a lower bound. A more accurate method based on the energy input, the water content, and rock conductivity, and dielectric properties is under development.

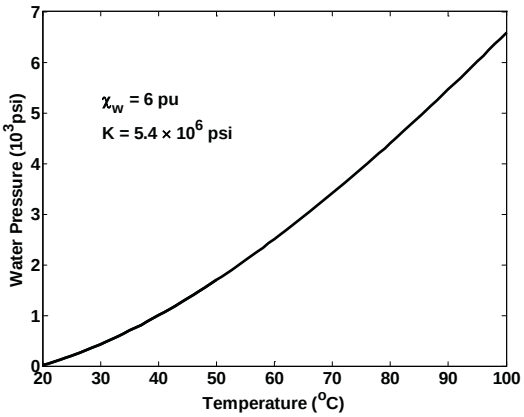

Fig. 8. The pore water pressure build up due to microwave heating for tight Tennessee sandstone with water content of 6 p.u. and matrix modulus $K = 5.4 \times 10^6$ psi. The pore water pressure, 1776 psi, at temperature 51°C when the rock fails, is interpreted as the tensile strength of the rock.

Organic-rich shales generally consist of significant amounts of quartz and/or carbonate, clay, and organic matter. In addition, shales usually contain small amount (1~2wt%) of pyrite, which has a dielectric loss of more than 10, in contrast to less than 0.1 for most other minerals and TOC. The temperature increase for a mineral depends on its dielectric loss. Hence, the temperature in a heterogeneous sample is non-uniform and heat transfer between different minerals and fluids needs to be evaluated for a precise water temperature. The interpretation of microwave heating of shales is much more complicated than that of tight sandstones and is current under investigation. Here we show some initial microwave heating experiments on shale samples. Quantitative estimate of tensile strength in shale samples will be reported in later publications.

Fig. 9 shows the Mancos outcrop shale plug on the left and results after 35 seconds of heating with a household microwave on the right. The water content in this sample is 3.3 p.u.. Microwaving completely obliterated this plug. Fig. 10 shows the microwaving result for a different Mancos outcrop plug. This plug contains 2.1% water. Microwaving left about half of the plug intact (far right in the picture). NMR testing on this half plug found no observable water signal. In addition, microwaving a dry (no NMR detectable water) Eagle Ford outcrop plug for 60 s did not generate any observable fractures, in contrast to the result on shale plugs with water content as shown in Fig. 9. These are consistent with our theory that increased water pressure is responsible for pulverizing the tight rocks.

Fig. 11 shows results of microwaving on clay disk pressed from Montmorillonite powder under 70,000 psi. This clay contains 6 percent water by weight. It took only 9 seconds to fracture the disk in the Hamilton Beach Household Microwave. In contrast, 30 seconds microwaving did not have much effect on a clay disk (data not shown) pressed from Kaolinite without NMR detectable water.

Microfractures in the rocks reduce the tensile strength of rocks which is reflected in the results of traditional method. The effect of microfractures on test results of tensile strength using microwaving method is not very clear and requires further study. However, if the microfractures form a continuous channel to the rock surface allowing water content and pressure to leak off, the water pore pressure cannot increase when heated by microwave. These microfractures then have no effect on the measured tensile strength from microwaving method because the rock breaking will be initiated from those water-filled pores that allow pressure elevation. In this case, microwaving method measured tensile strength only reflects the continuous phase of rock.

Fig. 9. A Mancos outcrop shale plug before (left) and after microwaving (right).

Fig. 10. A Mancos outcrop shale plug before (left) and after microwaving (right).
Half of the plug was intact after microwaving.

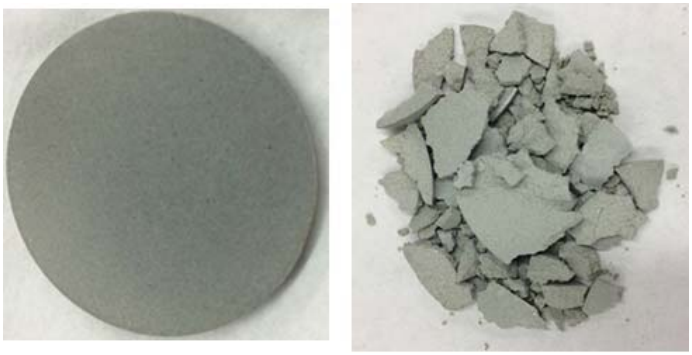

Fig. 11. Left: a clay disk pressed from Montmorillonite powder at 70,000 psi;
right: after 5 seconds microwaving in the household microwave.

The microwaving experiments on the tight sandstone and shales illustrate that the elevation of water pore pressure can easily and effectively pulverize or fracture the tight rocks. The same principle can be applied to subsurface fracturing applications. Microwave or electromagnetic (EM) wave heating in general can be an efficient fracturing technology and has the potential to replace hydraulic fracturing for tight reservoir production. Advantages of the proposed EM technology over the widely used hydraulic fracturing are obvious and remarkable:

1. Foremost, it is water free. This is significant for regions that are rich in tight reservoirs but lack water resource.
2. If EM heating can pulverize the tight rock in the subsurface with any resemblance to the laboratory experiments, a majority of the light hydrocarbons bound in the nanoporous rock system will be released.
3. EM heating avoids formation damage caused by fracturing fluid imbibition into shales when hydraulic fracturing.
4. It is environmentally friendly. It does not require any chemical treatment of the reservoir. In addition, controlling the size of stimulation zone with EM is relatively easy to implement, by setting the EM wavelength and energy density.
5. The EM technology should also work in deep reservoir and hard rocks.

6. Conclusion

A method is proposed to estimate tight rock tensile strength using microwaves to heat trapped pore water and elevate the pore pressure. The tensile strength equals the water pore pressure when the rock fails. We have demonstrated that this method can be used on tight sandstones. Application of this method to shales is more complicated due to the complex mineral composition. Microwaving tight rocks pulverizes them in the laboratory.

Based on the laboratory studies, we also propose that EM heating might be used to fracture tight reservoir for light hydrocarbon production. We believe that with further development, the proposed method might be an attractive water free alternative to the hydraulic fracturing.

7. Acknowledgement

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