EXPERIMENTAL STUDY AND NUMERICAL MODELING OF CRYOGENIC FRACTURING PROCESS ON LABORATORY-SCALE ROCK AND CONCRETE SAMPLES

by

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ABSTRACT

Cryogenic fracturing is a new fracturing concept that uses cryogenic fluids as fracturing fluids. Its mechanism rests on the effect of a thermal shock introduced by cryogen on the hot surface of reservoir rock. The thermo-mechanical properties of rock and failure criteria play very important roles during the cryogenic fracturing process.

The objective of this research is to conduct cryogenic fracturing experiments on concrete and rock samples and build a simulation tool. For the experimental aspect, a tri-axial stress confining system with capability of injecting liquid nitrogen or other cryogenic fluid is built. It is capable of conducting cryogenic fracturing treatment on concrete and rock samples under tri-axial stresses. For modeling, the experimental process is simulated and matched with the actual experiment results. The simulation tool can also predict the distribution of artificial fractured samples. The influences of different confining stress, injection pressure and failure criteria are identified by comparing results from modeling and experiments.

The experiment setup and modeling tool developed can also provide valuable guidance to field applications of cryogenic fracturing technology to select most efficient operation factors.
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LIST OF SYMBOLS

Cross-section area of the specimen  

Biot number of the rock  

Heat capacity of the aluminum inner vessel of calorimeter  

Specific heat of the sample  

Specific heat of water  

Diameter of the specimen  

Displacement discontinuities in normal direction at the crack tip element $i$  

Displacement discontinuities in shear direction at the crack tip element $i$  

Young's Modulus  

Mass or heat flux  

Shear modulus  

Bulk modulus  

Critical stress intensity factor  

Stress intensity factor in the normal direction  

Stress intensity factor in the shear direction  

Mogi-Coulomb intercept  

Thickness of the specimen  

Constraint modulus  

Mogi-Coulomb slope  

Mass of the sample  

$A$  

$B_i$  

$C_{al}$  

$c_{p-sample}$  

$c_{p-w}$  

$D$  

$D_n^i$  

$D_s^i$  

$E$  

$F$  

$G$  

$K$  

$K_{Ic}$  

$K_I$  

$K_{II}$  

$k$  

$L$  

$M$  

$m$  

$m_{sample}$
Mass of water \( m_w \)

Total number of grid blocks \( n \)

Maximum applied load \( P \)

Break down pressure of formation rock \( p_b \)

Formation pore pressure \( p_p \)

Bottom-hole flowing pressure assuming radial injection \( p_{wf,r} \)

Quantity represents mass or energy per volume \( Q \)

Average heat loss rate \( \dot{Q}_{\text{avg}} \)

Sinks and sources \( q \)

Temperature \( T \)

Reference or original temperature \( T_0 \)

An arbitrary subdomain of the system under study \( V_n \)

Compressional wave velocity \( V_p \)

Shear wave velocity \( V_s \)

Half-length of an internal crack \( x_f \)

Temperature change of water \( \Delta T_w \)

Temperature change of the sample \( \Delta T_{\text{sample}} \)

Time used to reach equilibrium \( \Delta t \)

Linear thermal expansion of the rock \( \beta \)

Total number of components \( \kappa \)

Density \( \rho \)

Thermo-elastic stress \( \sigma^{\Delta T} \)

Closed surface by which the subdomain is bounded by \( \Gamma_n \)
Uniaxial compressive strength \( \sigma_c \)

Stress at which a crack start to grow \( \sigma_{cr} \)

Maximum horizontal principal stress \( \sigma_{H\text{max}} \)

Minimum horizontal principal stress \( \sigma_{h\text{min}} \)

Normal stress in \( kk \) direction \( \sigma_{kk} \)

Octahedral normal stress \( \sigma_{oct} \)

Splitting tensile strength \( \sigma_t \)

Reservoir stress at the fracture tip \( \sigma_{\text{tip}} \)

Vertical stress \( \sigma_v \)

Octahedral shear stress \( \tau_{oct} \)

Strain in \( kk \) direction \( \varepsilon_{kk} \)

Poisson’s ratio \( \nu \)

Displacement vector \( \mathbf{d} \)

Body-force vector per unit volume \( \mathbf{f} \)

Identity matrix \( \mathbf{I} \)

Stress tensor \( \mathbf{\sigma} \)

Strain tensor \( \mathbf{\varepsilon} \)

A normal vector on surface element \( d\Gamma_n \) pointing inward into \( V_n \) \( \mathbf{\bar{n}} \)
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CHAPTER 1
INTRODUCTION

Cryogenic fracturing is a new concept that looks to expand and improve on traditional hydraulic fracturing technology. The concept rests on the idea that a frigid fluid can induce fractures when brought into contact with a much warmer rock under downhole conditions. The cold fluid that effectuates such a fracture is known as the cryogenic fluid, or cryogen. When liquid nitrogen is injected into a rock whose temperature is much higher, heat from the rock will quickly transfer to the liquid nitrogen. This rapid heat transfer, better known as a thermal shock, will cause the surface of the rock to shrink, relative to the inner warmer material of the rock and eventually fail in tension, inducing fractures orthogonal to the contact plane of the cryogen and the rock. Liquid nitrogen has a liquid-gas expansion ratio of 1:694 under the ambient condition which, in a confined space, should create a high pressure environment helping to propagate the fractures.

Just as hydraulic fracturing has changed the energy resource development landscape, cryogenic fracturing offers much promise in answering the petroleum industry’s quest to improve both the pace and efficiency of resource recovery. This new technology could potentially increase the effects of fracturing and decrease the cost of fracturing resulting in more formations becoming economically viable. Cryogenic fracturing therefore has the potential to drastically increase oil and gas reserves. Traditional hydraulic fracturing in low permeability formations uses a highly pressurized fracturing fluid that carries proppants to create and prop open a complex network of fractures. These conductive fractures increase the contact area between the reservoir and the wellbore allowing more reservoir fluids to flow into the wellbore and be extracted. Hydraulic fracturing and its associating technologies have drastically changed the United States’ oil and gas production. Without a doubt hydraulic fracturing has revolutionized the exploitation of hydrocarbons in the United States.
Modern hydraulic fracturing technology relies on water-based fracturing fluids, due to the general availability and low cost of water; however, a dependence upon water presents several major shortcomings. First, water can cause significant formation damage, which can occur as clay swelling and relative permeability effects stemming from capillary fluid retention. Formation damage mechanisms inhibit hydrocarbon flow and thus impair production rates and recovery efficiency. Second, water use in large quantities may place significant stresses upon local water resources as well as environments. For example, diversion of water away from other uses, transportation of water to well sites on road infrastructure that was not designed for high traffic volumes, or construction activities associated with pipeline development can all have great impacts on the surrounding community. Finally, the downhole injection of chemicals needed in water-based fracturing programs, including slickwater and gel-based fracturing treatments, can lead to a contentious political climate.

In contrast to hydraulic fracturing, cryogenic fracturing offers potentially greater fracturing capabilities without the issues associated with use of water-based fracturing fluids. As for formation damage, there are no concerns for cryogenic fracturing, since nitrogen will not react with chemicals inside the formation neither as liquid nor as gas. In addition, there is also no flow back requirement after cryogenic fracturing for nitrogen can be miscible with natural gas and has little retention when displaced with liquid hydrocarbons inside the formation. After cryogen treatment, the formation can be regarded as intact from any kind of water or drilling fluid. Cryogenic fracturing can also minimize water consumption in stimulation process, which will save millions of gallons water when compared to traditional hydraulic fracturing operations. Once massively deployed, liquid nitrogen can be obtained by separating and compressing nitrogen from air by commercial air separation equipment at operation sites, which also minimizes the cost for transportation or pipeline development.

The main objectives of this thesis are as followed:

- Investigating fundamental physical processes that happened during fracture initiation and propagation by cryogenic fluid.
• Conducting cryogen treatment on concrete and rock samples under tri-axial confining stress condition to evaluate the effectiveness and feasibility of cryogenic fracturing.

• Measuring basic rock properties of concrete and rock samples to provide input parameters for modeling. Explaining different performance observed on different samples via these basic rock properties.

• Building a mathematical model for cryogenic fracturing. Simulating the process to provide further insight on fracture initiation and propagation processes.

• Provide guidance for future field applications.
CHAPTER 2
LITERATURE REVIEW

Cryogenic fracturing is a relatively new concept to mitigate the side effects and environmental concern in traditional hydraulic fracturing technology. There are several cryogenic fracturing laboratory experiments and field tests that have been done in past thirty years. However, there are not many experiments or theoretical analyses looking into the fracturing mechanism or rock mechanical behavior under low-temperature and high-pressure condition for sedimentary rocks, which are the main composition of petroleum reservoirs. The fracturing mechanism is also tied to the mechanical behavior of rock at cryogenic temperature and the phase behavior of these cryogen fluids.

2.1 Cryogenic Fracturing

Cryogenic fracturing is not a new emerging concept in petroleum industry. Although not much research has been done in this area, King (1983) used gelled liquid carbon dioxide to stimulate tight gas sand formations instead of conventional fracturing fluids such as water or oil. His primary motivation for using liquid carbon dioxide as stimulation fluid is to eliminate the effects of residual fluid in the stimulation of low permeability reservoirs, especially for the low fluid return problem. After the liquid carbon dioxide treatment, the carbon dioxide would evaporate and return to the surface under controlled rates as a gas, resulting in a more rapid cleanup. Since the liquid carbon dioxide is gelled, it has enough carrying capability for bringing proppants into the fractures and holding them open. He used this technique to treat several wells in field. All of the wells showed increased production rate after treatment. Unfortunately, no long-term post fracturing production data is given.

Although the field tests showed successful results in King’s research, the laboratory experiments were absent. The field tests results cannot address whether the fractures are generated due to hydraulic effect or thermal effect from the gelled liquid carbon dioxide.
To further address the fracturing mechanism, McDaniel et al. (1998) conducted several laboratory liquid nitrogen submersion tests on coal samples to prove that cryogenic fracturing may have an advantageous effect on gas production from tight, low-rate coal-bed methane wells. The coal samples experienced significant shrinkage during the submersion tests with creation of micro-fractures orthogonal to the surfaces which were exposed to cryogen. After further multi-cycle cryogen submersion tests, the coal samples were shattered into small pieces. After three cycles of submersion in liquid nitrogen and warming up to the ambient temperature, the coal samples were reduced to grain size particles. This research shows that cryogenic fracturing can effectively increase the production in coal-bed methane formations and may also have a promising effect on other rock formations. Besides the laboratory experiments, McDaniel et al. (1998) also applied cryogenic fracturing to five wells for field tests. However, the results from these wells were mixed: three of them experienced increased production rate, one experienced equivalent production and one experienced decreased production. Among the three wells with increased production, two of them had long term incremental production.

Grundmann et al. (1998) conducted later a cryogenic fracturing treatment in a Devonian shale well with liquid nitrogen. The well showed an 8% increment in the initial production rate when compared to a nearby offset well that underwent a traditional nitrogen gas fracturing treatment. However, there was no subsequent production information available for this well due to a logistical shut-in. Although the result from this well may result from various reasons, such as anisotropic stress conditions and heterogeneous reservoir conditions over short distances, it showed no drawback with cryogenic fracturing technology as opposed to conventional hydraulic fracturing.

Although the field tests have shown some promising benefits from cryogenic fracturing, they did not identify the fracturing mechanisms at work in downhole conditions. In addition, there are also some concerns about the effectiveness of cryogenic fracturing, such as equipment used for injection cryogenic fluids and proppant carrying capability. From research by
Rudenko and Shubnikov (1934) and Fenghour et al. (1998), liquid nitrogen and carbon dioxide lack significant viscosity for carrying proppants into fractures in downhole conditions, which may result inadequate proppants in fractures to hold them open. Gupta and Bobier (1998) concluded that it is possible for cryogenic carbon dioxide to transport adequate amount of proppants by increasing the velocity of the fluid. The turbulence accompanied by the high velocity permitted proppant to be carried efficiently from the wellbore to the perforations or even to the fractures. In addition, with the rubblization effect discovered in the research from McDaniel et al. (1998), the rock formations treated with cryogenic fluid may undergo a self-propping process. The rubblized rock may enable the fractures to stay open against in-situ stress after cessation of treatment pressure.

2.2 Rock Properties at Low Temperature

Rock properties at low temperature are critical for simulation of cryogenic fracturing experiments and they play a vital role in helping us understand the fracturing mechanism. Not much research has been done in this area when compared to the availability of physical properties of rock at higher temperatures. Heins and Friz (1967) tested the thermos-mechanical behavior of three types of rock: limestone, basalt and granite. Their motivation for testing rocks at low temperatures stems from the advent of lunar exploration. The drastic temperature change at the surface of moon (approximately $-250^\circ F$ to $+250^\circ F$) creates a complicated environmental factor for rock mechanical behavior and drew a general interest in the field of rock mechanics in the 1960s. After subjecting three different types of rocks at both room temperature ($75^\circ F$) and liquid nitrogen temperatures ($-320^\circ F$) to the same mechanical tests, the point-load breaking strength and modulus of rupture all show incremental increases at liquid nitrogen temperatures. Limestone, which is an important sedimentary rock in petroleum reservoirs, shows an average 46% increment in the point-load breaking strength and 59% increment in the modulus of rupture at liquid nitrogen temperatures. According to these facts, Heins and Friz (1967) conclude qualitatively that the Young’s Modulus for limestone increases with decreasing temperature.
Similar to aforementioned work, Inada and Yokota (1984) have done vast amount of experiments on the physical properties of granite and andesite with the motivation for Liquefied Natural Gas (LNG) storage. Inada and Yokota (1984) tested samples with different water saturations at multiple temperature points or with multiple cooling-heating cycles in order to obtain the effect of temperature, water saturation and thermal history on the mechanical properties of rock. As a result, both compressive and tensile strengths of rocks are tested at low temperatures with the reduction in temperature, as shown in Figure 2.1. Water saturation increases the tensile strength significantly and increases the compressive strength moderately at low temperatures compared to dry samples at low temperatures. Cooling-heating cycles influence the properties of rocks by significantly reducing the strength of rocks, especially for wet samples because the integrity of the cementing material is strongly influenced by ice. Increasing the number of cycles weakens the rock. In a later research by Inada et al. (1997), samples of granite and tuff are further tested to characterize the effect of cooling-heating cycles on rock mechanical properties. The result is similar: the rock becomes weaker with increasing cycles of thermal hysteresis, including compressive and tensile strength (shown in Figure 2.2), tangential Young’s modulus and Poisson’s ratio. In addition to this finding, all these properties seem to converge to constant values after enough cycles were elicited.

Although these results are not obtained specifically for sedimentary rocks of normal petroleum reservoirs, they give a good guidance for the development for cryogenic fracturing technology. For instance, increasing the number of treatments should weaken the strength of rocks, making them easier to be fractured. Unfortunately, all the studies on rock properties at low temperatures are only available as data points at certain temperatures between the room temperature and the liquid nitrogen temperature. There is no empirical formula to correlate rock mechanical properties with temperature change.
Figure 2.1: Dynamic Young’s Modulus at Low Temperature by Inada and Yokota (1984).

Figure 2.2: Tensile Strength after Undergoing Thermal Hysteresis of Low Temperature by Inada et al. (1997).
2.3 Thermal Failure of Rocks

Thermally induced fracturing is a very well-known and well-established phenomenon in rock mechanics. It was first discovered during large water injection for secondary recovery. Generally, a large temperature change inside the pore volume of rocks will be accompanied by a large physical property changes within the rock matrix, not only the physical properties that are mentioned in last section, but also properties like porosity and permeability. Those changes in rock properties under extreme circumstances lead to thermal failure of rock, creating potential pathways for fluid migration. The current focus of thermal failure research for rocks is thermal spalling, and thermal pressurization by pore fluid. However, as there are generally many processes occurring at the same time during thermally induced fracturing, assumptions are often made to simplify the complex mechanism and to reduce the number of parameters.

In a thermal spalling process, a certain part of rock is rapidly heated causing thin disk-like fragments to be removed from the surface before the rock materials begin to melt. The mechanism of thermal spalling was first described by Preston (1926) and Preston and White (1934). Due to the low thermal conductivity of rocks, a large temperature gradient concentrates at near-surface layer, exerting an associated compressive stress. With pre-existing natural fractures or weaknesses, the compressive stress will cause fractures to extend outward parallel to the rock surface. When the induced fracture propagates to a sufficient extent, the heated fragment will buckle and then fall off from the rock. Thermal spalling has long been of interest as a drilling method for faster rate of penetration, especially for brittle granitic rocks according to Browning et al. (1965). In addition, Germanovich (1997) concluded that the thermal spallability (or easiness to be thermal spalled) depends on not only the material type, but also on the absorbed heat flux. Germanovich also derived an expression for the stress at which the crack due to compression start to grow:

$$\sigma_{cr} = \sqrt{\frac{K}{x_f}} K_{IC}$$  \hspace{1cm} (2.1)
where:

\[ \sigma_{cr} \] is the stress at which a crack start to grow due to thermally induced compression;

\[ K_{Ic} \] is the critical stress intensity factor;

\[ x_f \] is the half-length of an internal crack.

Thermal pressurization results from the discrepancy between thermal expansion coefficients of the pore fluid and the rock matrix when the rock is subjected to temperature differences. The change in the pore pressure will induce a change in the effective mean stress and can lead to shear failure of the rock. When temperature increases in saturated porous materials, the effective mean stress will decrease due to the pore pressure increase, or vice versa. The change in the thermal pressurization coefficient, which is the pore pressure increment due to a unit temperature increase in undrained condition, is dependent highly on the nature of materials, state of stress, range of temperature change, and induced damage according to Ghabezloo and Sulem (2009).

Besides the aforementioned thermal spalling and pressurization studies, both of which are more of fundamental interests, there are also several more engineering-oriented research on thermally induced failures of rocks.

Detienne et al. (1998) presented a simplified model for thermally induced fracturing during water injection. In this model, fracturing pressure is assumed to be equal to the stress at the well. This assumption is proved effective although a more complex formula for back stresses at the well could be introduced. With this assumption, the failure criterion is

\[ p_{wf,r} = \sigma_{tip} \]  \hspace{1cm} (2.2)

where:

\[ p_{wf,r} \] is the bottom-hole flowing pressure assuming radial injection;

\[ \sigma_{tip} \] is the reservoir stress at the fracture tip.
Thus when the flowing pressure is larger than the reservoir stress \( p_{\text{wf,r}} > \sigma_{\text{tip}} \), the radial flow must fracture the formation rock. After matching Equation 2.2 with actual field data, it was shown that this model could predict the daily wellhead pressure and injection rate during a period of three to five years for injection wells in complex sandstone/dolomite reservoirs.

In a later research on CO\(_2\) injection induced fractures, Luo and Bryant (2010) presented a similar model for estimation of the range of safe injection rate to avoid injection-induced fracture initiation around an injection well. In this model, the thermo-elastic stress is calculated from the difference between the original formation temperature and the temperature of formation containing injection fluids using a thermal expansion coefficient. The mechanical properties of formation rock are constants for any temperature. The failure criterion for fracture initiation is adapted from Zoback (2007):

\[
p_b = 3\sigma_{h\text{min}} - \sigma_{H\text{max}} - p_p - \sigma^{\Delta T}
\]

where:

- \( p_b \) is the breakdown pressure;
- \( \sigma_{h\text{min}} \) is the minimum horizontal principal stress;
- \( \sigma_{H\text{max}} \) is the maximum horizontal principal stress;
- \( p_p \) is the formation pore pressure;
- \( \sigma^{\Delta T} \) is the thermo-elastic stress.

In addition to this, Joule-Thomson cooling effect is applied to account for the temperature drop when CO\(_2\) flows through perforations. The model shows that the magnitude of thermo-elastic stress where injected CO\(_2\) enters the storage formation depends on the efficiency of heat transfer between the fluid in the wellbore and the surrounding formation relative to the injection rate. This model also provides an insight that when the injection rate of CO\(_2\) is very large, thermo-elastic stress will have a greater influence on the formation rock.
The thermal failure of rocks is also vital for geothermal reservoirs. The fractures that it leads to are usually referred to as secondary thermal fractures. Zhou et al. (2010) studied the initiation, propagation and interaction of thermal failures within impermeable, hot and dry rocks due to the cooling of a main hydraulic fracture by long-term reservoir fluids circulation. The thermo-elastic model is adapted from Jaeger et al. (2009):

\[
\sigma = 2G\varepsilon + \left(K - \frac{2G}{3}\right)\text{trace}(\varepsilon)I + 3\beta KT I
\]

(2.4)

where:

- \(\sigma\) is the stress tensor;
- \(\varepsilon\) is the strain tensor;
- \(K\) is the bulk modulus;
- \(G\) is the shear modulus;
- \(T\) is the temperature;
- \(\beta\) is the coefficient of linear thermal expansion;
- \(I\) is the identity matrix.

The thermo-elastic equation can also be combined with the momentum and energy balance equations and the heat transfer equation to yield for the following field equation:

\[
G\nabla^2 \mathbf{d} + \left(K + \frac{G}{3}\right)\nabla(\nabla \cdot \mathbf{d}) = -\mathbf{f} - 3\beta K \Delta T
\]

(2.5)

where:

- \(\mathbf{d}\) is the displacement vector;
- \(\mathbf{f}\) is the body-force vector per unit volume.
The fracture propagation model is based on the stress intensity factor (SIF) by Olson and Pollard (1991), which can be computed as a function of the displacement discontinuities at the crack tip element, \( i \):

\[
K_I = 0.806 \left[ \frac{\sqrt{\pi}E}{4(1 - \nu^2)\sqrt{2a}} \right] D_n^i 
\]

\[
K_{II} = 0.806 \left[ \frac{\sqrt{\pi}E}{4(1 - \nu^2)\sqrt{2a}} \right] D_s^i
\]  

(2.6)  

(2.7)

where:

\( K_I \) is the stress intensity factor in the normal direction;

\( K_{II} \) is the stress intensity factor in the shear direction;

\( E \) is Young’s Modulus;

\( \nu \) is Poisson’s ratio;

\( D_n^i \) is the displacement discontinuities in normal direction at the crack tip element \( i \);

\( D_s^i \) is the displacement discontinuities in shear direction at the crack tip element \( i \).

The equivalent SIF based the maximum circumferential stress theory from Erdogan and Sih (1963) is used for fracture propagation criteria:

\[
K_{eq} = \cos \frac{\theta_0}{2} \left( K_I \cos^2 \frac{\theta_0}{2} - \frac{3}{2} K_{II} \sin \frac{\theta_0}{2} \right)
\]

(2.8)

where \( \theta_0 \) is determined by:

\[
K_I \sin \theta_0 + K_{II} (3\cos \theta_0 - 1) = 0
\]

(2.9)

Fracture propagation will take place when \( K_{eq} \) exceeds the material fracture toughness \( K_{IC} \), which is set for certain type of materials. Zhou et al. (2010) found that the thermal fracture propagation for a single secondary thermal fracture has a strong relationship with the temperature change of the secondary thermal fractures and the maximum horizontal in-situ stress, which is shown as Figure 2.3. The cooled secondary thermal fracture (showing
in red in Figure 2.3), assuming that the fluid entering the secondary thermal fracture has the same temperature as that in the main fractures, propagates about 1/3 longer than the uncooled one (showing in blue in Figure 2.3) at time of 2108 seconds (6.2 years). The larger maximum horizontal in-situ stress has a negative effect on the propagation length of the secondary thermal fracture.

Figure 2.3: The propagation lengths of a single secondary fracture for various situations by Zhou et al. (2010).
In this chapter, we present the experiment setup for rock properties measurement on concrete, sandstone and shale samples. Characterizing rock properties, such as porosity, permeability, mechanical modulus, and thermal properties, are crucial for developing quantitative understanding and predictions on geomechanical effects associated with subsurface flows. Rock property measurements have been conducted for three different rock types. These three rock types are concrete, sandstone, and shale. In this section, measurements and results are presented for each rock type. Since dimensional requirements for different measurements are different, Table 3.1 presents the details of these requirements.

Table 3.1: Core Dimension Requirements for Different Measurements

<table>
<thead>
<tr>
<th>Test</th>
<th>Concrete</th>
<th>Sandstone</th>
<th>Shale</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Diameter (in)</td>
<td>Length (in)</td>
<td>Diameter (in)</td>
</tr>
<tr>
<td>Acoustic</td>
<td>2</td>
<td>4</td>
<td>1.5</td>
</tr>
<tr>
<td>Permeability &amp; Porosity</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Brazilian Test</td>
<td>2</td>
<td>1</td>
<td>1.5</td>
</tr>
<tr>
<td>Uniaxial Compression Test</td>
<td>2</td>
<td>4</td>
<td>1.5</td>
</tr>
</tbody>
</table>

3.1 Equipment and Measurement Condition

The basic rock properties measured in this research includes acoustic measurement of mechanical properties, such as Constraint Modulus, Shear Modulus, Bulk Modulus, Young’s Modulus, and Poisson’s Ratio using acoustic transmitter and receiver, permeability and porosity measurements using CMS-300, tensile strength and uniaxial compression strength measurements using an MTS loading frame.

3.1.1 Acoustic Measurement

Acoustic measurement provides velocities of compressional and shear waves inside solid materials. With a known density, the dynamic mechanical modulus and the Poisson’s ratio
can be obtained from these two velocities. The measurement on concrete samples were conducted on cores with 2” in diameter and 4” in length for concrete, 1.5” in diameter and 3” in length for sandstone and 1” in diameter and 2” in length for shale using P-wave and S-wave pulsers/receivers and oscilloscope (DSO-X 2004A by Agilent Technologies). Figure 3.1 shows a schematic of the experiment setup for acoustic measurements. Table 3.2 shows the wave equations and the mechanical moduli equation that are used to obtain the various mechanical moduli. The procedures of acoustic measurement follow the procedure established by Cha and Cho (2007).

![Experiment Setup for Acoustic Measurement by Cha and Cho (2007)](image)

**Table 3.2: Wave Equations and Mechanical Modulus Equations**

<table>
<thead>
<tr>
<th>Property</th>
<th>Equation Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constraint Modulus, M</td>
<td>$M = \rho V_p^2$</td>
</tr>
<tr>
<td>Shear Modulus, G</td>
<td>$G = \rho V_s^2$</td>
</tr>
<tr>
<td>Bulk Modulus, K</td>
<td>$K = M - \frac{2}{3}G$</td>
</tr>
<tr>
<td>Young’s Modulus, E</td>
<td>$E = \frac{G(3M - 4G)}{2M - 2G}$</td>
</tr>
<tr>
<td>Poisson’s Ratio, $\nu$</td>
<td>$\nu = \frac{M - 2G}{2M - 2G}$</td>
</tr>
</tbody>
</table>

where:

$\rho$ is density;

$V_p$ is the compressional wave velocity;
$V_s$ is the shear wave velocity.

### 3.1.2 Permeability and Porosity

Permeability and porosity are measured by CMS-300 manufactured by CoreLab, as shown in Figure 3.2. This equipment uses helium to flow through the core samples under a hydrostatic confining stress. The workflow is shown in Figure 3.3. Permeability and porosity measurements were conducted on three cores for each type of rock samples with 500 psi confining stress at the ambient condition ($65^\circ F$ and 11.87 psia).

![Figure 3.2: Core Measurement System, Model 300 (CMS-300) by CoreLab.](image)

### 3.1.3 Brazilian Test

Brazilian test, also known as the splitting tensile strength test, is used to determine the tensile strength of cylindrical specimens, such as molded cylinder and drilled cores. The test methodology in the laboratory follows the American Society of Testing and Materials (ASTM) standards. The testing equipment used is MTS Damper Landmark loading frame, which is as shown in Figure 3.4. The MTS loading frame can measure both load (force) and displacement at the same time during tests.

During a Brazilian test, a specimen is placed in the middle of the two machine platens, as shown in Figure 3.5. The loading frame is under the strain-control condition during testing (0.5 mm per minute). As the load on the specimen increases, the specimen will split in the
middle along the loading direction due to tensile stress exceeding the tensile strength. Then, the tensile strength of the rock sample is calculated by the following equation:

$$\sigma_t = \frac{2P}{\pi LD}$$

(3.1)

where:

- $\sigma_t$ is the splitting tensile strength;
- $P$ is the maximum applied load indicated by the testing machine;
- $L$ is the thickness of the specimen;
- $D$ is the diameter of the specimen.

### 3.1.4 Uniaxial Compression Test

The uniaxial compression test is designed to measure the compressive strength of cylindrical specimens under no confining pressure or stress. Specimens are loaded axially up to failure or any other prescribed level. The apparatus used for uniaxial compression tests is the
Figure 3.4: MTS Damper Landmark Loading Frame.

Figure 3.5: Testing Setup for Brazilian Test.
same as the Brazilian test. The uniaxial compressive strength of specimens is determined by the maximum loading during testing and the cross-section area using the following equation:

\[ \sigma_c = \frac{P}{A} \]  

(3.2)

where:

- \( \sigma_c \) is the uniaxial compressive strength of the specimen;
- \( P \) is the maximum loading force during testing;
- \( A \) is the cross-section area of the specimen.

The uniaxial compression test for concrete samples and sandstone samples are conducted using the tri-axial loading frame, as shown in Figure 3.6, which uses hydraulic pumps powered by high-pressure air. The test for shale samples are conducted by the same MTS loading frame as for Brazilian tests. The reason for this is because the uniaxial compressive strength of concrete and sandstone samples exceeds the maximum allowable load of the MTS loading frame. However, the tri-axial loading frame can only measure the load (force) during tests and is not as accurate as the MTS loading frame.

Specimens from drilled cores or molded cylinders are prepared by cutting them to the specified length and are thereafter ground and measured. There are high requirements on the flatness of the end-surfaces in order to obtain an even load distribution. The recommended height-to-diameter ratio of specimens is between 2 to 3.

### 3.1.5 Specific Heat

The equipment used to measure specific heat of rock samples includes a calorimeter, a thermal couple, a weight scale, and a data acquisition system. The calorimeter used is a passive calorimeter, which has no heat source, as shown in Figure 3.7. It can isolate the sample from a heat transfer fluid, creating an adiabatic environment. The scale, as shown in Figure 3.8, is used to determine the mass of samples and the amount of heat transfer fluid.
used during measurements. The thermal couple can provide a relatively accurate reading for temperature with a precision of 0.2°C.

The measurements are conducted for samples initially at the ambient temperature (24.8°C or 76.6°F) and the heat transfer fluid, which is water, has a temperature ranging from 50°C to 70°C (122°F to 158°F).

Before the measurement, the calorimeter is calibrated to obtain the heat capacity of the aluminum inner vessel. In addition, a calibration for the heat loss of the calorimeter is conducted from 65°C to 35°C (149°F to 95°F). The heat loss rate as a function of temperature is obtained to mitigate the effect of a non-perfect isolation. The heat loss calibration curve is shown in Figure 3.9.

During a specific heat measurement, heat transfer fluid is poured into the calorimeter at first. After the temperature is stabilized, a rock sample at 24.8°C is dropped into the heat transfer fluid very gently. The temperature change is then recorded during the measurement.
Figure 3.7: Components of Calorimeter by MiniScience.

Figure 3.8: Weight Scale.
Once the temperature reaches a new equilibrium, the specific heat is calculated.

The calculation for the specific heat of samples is based on energy balance. During the measurement, the heat or energy released by the high-temperature object is transferred to low-temperature objects and to the environment, which is considered as heat loss. Thus, the following relation is obtained:

\[(c_{p-w}m_w + C_{al})\Delta T_w = \dot{Q}_{avg}\Delta t + c_{p-sample}m_{sample}\Delta T_{sample}\]  \hspace{1cm} (3.3)

where:

\(c_{p-w}\) is the specific heat of water;

\(m_w\) is the mass of water used in this measurement;

\(C_{al}\) is the heat capacity of the aluminum inner vessel of calorimeter from calibration;

\(\Delta T_w\) is the temperature change of water;

\(\dot{Q}_{avg}\) is the average heat loss rate during the measurement;
$\Delta t$ is the time used to reach equilibrium after dropping the sample;

$c_{p\text{-sample}}$ is the specific heat of the sample;

$m_{\text{sample}}$ is the mass of the sample;

$\Delta T_{\text{sample}}$ is the temperature change of the sample.

A typical temperature profile for a measurement process is shown in Figure 3.10. The time for a dropped sample to reach equilibrium is shaded with brown. Thus, the specific heat of samples is the only unknown in the equation and can be solved for.

![Temperature Profile](image)

Figure 3.10: Temperature Profile for Specific Heat Measurement for Sample Shale-2.

### 3.2 Properties of Concrete

Concrete samples are common surrogates for rocks in laboratory testing, as they are easy to make with consistent properties. In addition, results from concrete samples can also guide the testing of sandstone and shale samples, which are more difficult to obtain.
3.2.1 Specimen Preparation

The concrete samples are made by Type II Portland cement with a constant ratio of water and dry sand. Table 3.3 shows the composition of all concrete samples.

![Table 3.3: Compositions for Concrete Samples](image)

Concrete cores were made using cylindrical molds. We have two different types of molds: plastic molds with 2 inch inner diameter and 4 inch in length and steel molds with 3 inch inner diameter and 6 inches in length. The concrete samples were put into water after one day of curing. The underwater curing lasted one month to reach the maximum strength of concrete samples. After water curing, samples were taken out of water and dried in the ambient condition before use.

For permeability tests, 1 by 1 inch concrete cores were wet drilled with 1 inch inner diameter (ID) diamond impregnated bit from larger cores (3” in diameter and 6” in length). For Brazilian tests, cores were directly cut from originally made ones.

3.2.2 Acoustic Measurement

The results from acoustic measurements are shown in Table 3.4.

![Table 3.4: Averaged Acoustic Measurement Results for Concrete Samples](image)
3.2.3 Permeability and Porosity

Figure 3.11 shows the concrete core samples used for permeability and porosity measurements. The results of these permeability and porosity measurements are in Table 3.5. The average porosity of the concrete samples is 9.56% and the average permeability is around 9 microdarcy (0.009 md).

![Figure 3.11: Concrete Core Samples Used for Permeability and Porosity Measurement.](image)

Table 3.5: Permeability and Porosity Results from CMS-300 for Concrete Samples

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>C-1</th>
<th>C-2</th>
<th>C-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>1.003 inch</td>
<td>1.003 inch</td>
<td>1.003 inch</td>
</tr>
<tr>
<td>Length</td>
<td>0.975 inch</td>
<td>1.121 inch</td>
<td>1.002 inch</td>
</tr>
<tr>
<td>Weight</td>
<td>25.65 g</td>
<td>29.69 g</td>
<td>26.53 g</td>
</tr>
<tr>
<td>Bulk Volume</td>
<td>12.624 cc</td>
<td>14.514 cc</td>
<td>12.974 cc</td>
</tr>
<tr>
<td>Bulk Density</td>
<td>2.032 g/cc</td>
<td>2.046 g/cc</td>
<td>2.045 g/cc</td>
</tr>
<tr>
<td>Pore Volume</td>
<td>1.145 cc</td>
<td>1.334 cc</td>
<td>1.351 cc</td>
</tr>
<tr>
<td>Porosity</td>
<td>9.07%</td>
<td>9.19%</td>
<td>10.41%</td>
</tr>
<tr>
<td>Permeability</td>
<td>$8.15 \times 10^{-3}$ md</td>
<td>$7.31 \times 10^{-3}$ md</td>
<td>$1.07 \times 10^{-3}$ md</td>
</tr>
</tbody>
</table>

3.2.4 Brazilian Test

Figure 3.12 shows concrete samples for Brazilian tests. Figure 3.13 shows a typical load-and-deformation curve for concrete samples during Brazilian tests. The results for the tensile
strength for concrete cores are listed in Table 3.6. The average tensile strength of concrete samples is 2.878 MPa (418 psi).

![Concrete Samples for Brazilian Test.](image)

**Figure 3.12: Concrete Samples for Brazilian Test.**

### Table 3.6: Tensile Strength of Concrete Cores

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>C-1 Br</th>
<th>C-2 Br</th>
<th>C-3 Br</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Diameter</strong></td>
<td>2.032 inch</td>
<td>2.030 inch</td>
<td>2.037 inch</td>
</tr>
<tr>
<td><strong>Thickness</strong></td>
<td>1.011 inch</td>
<td>1.016 inch</td>
<td>1.027 inch</td>
</tr>
<tr>
<td><strong>Maximum Loading Force</strong></td>
<td>7118.281 N</td>
<td>5324.916 N</td>
<td>5676.197 N</td>
</tr>
<tr>
<td><strong>Tensile Strength</strong></td>
<td>3.419 MPa (496 psi)</td>
<td>2.547 MPa (370 psi)</td>
<td>2.677 MPa (388 psi)</td>
</tr>
</tbody>
</table>

### 3.2.5 Uniaxial Compression Test

Figure 3.14 shows concrete samples for uniaxial compression tests. A typical loading force curve for concrete samples is shown in Figure 3.15. Fluctuations in the loading curve were due to the vibration of the hydraulic pump during loading. The detailed results for the uniaxial compressive strength of concrete samples are listed in Table 3.7. The average uniaxial compressive strength of concrete samples is 37.343 MPa (5416 psi).
Figure 3.13: Typical Brazilian Test Deformation Curve of Concrete Samples.

Figure 3.14: Concrete Samples for Uniaxial Compression Test.
Figure 3.15: Typical Uniaxial Compression Loading Curve for Concrete Samples.

Table 3.7: Uniaxial Compressive Strength Results for Concrete Cores

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>C1-5 UC</th>
<th>C1-7 UC</th>
<th>C1-8 UC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>2.047 inch</td>
<td>2.041 inch</td>
<td>2.050 inch</td>
</tr>
<tr>
<td>Thickness</td>
<td>3.99 inch</td>
<td>4.047 inch</td>
<td>4.036 inch</td>
</tr>
<tr>
<td>Maximum Loading Force</td>
<td>17,267 lb</td>
<td>17,813 lb</td>
<td>18,356 lb</td>
</tr>
<tr>
<td>Uniaxial Compressive Strength</td>
<td>36.169 MPa (5246 psi)</td>
<td>37.528 MPa (5443 psi)</td>
<td>38.333 MPa (5560 psi)</td>
</tr>
</tbody>
</table>
3.2.6 Specific Heat

The measured specific heat for concrete samples are ranged from $859 J/(kg \cdot K)$ to $912 J/(kg \cdot K)$, with an average of $891 J/(kg \cdot K)$. These results are listed in Table 3.8.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>C-1</th>
<th>C-2</th>
<th>C-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Heat</td>
<td>$901 J/(kg \cdot K)$</td>
<td>$859 J/(kg \cdot K)$</td>
<td>$912 J/(kg \cdot K)$</td>
</tr>
<tr>
<td>Average Specific Heat</td>
<td>$891 J/(kg \cdot K)$</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.3 Properties of Sandstone

The sandstone samples are gathered from an outcrop of Williams Fork Formation in Western Colorado. This section covers all rock property measurements for the sandstone samples.

3.3.1 Specimen Preparation

The cores were wet drilled from the rock chunks, also from which several 8 by 8 by 8 blocks were cut for tri-axial tests. All cores were dried in the ambient condition for several days before the measurements.

3.3.2 Acoustic Measurement

The averaged results of acoustic measurements for sandstone samples are shown in Table 3.9.

3.3.3 Permeability and Porosity

Figure 3.16 shows the sandstone core samples used for permeability and porosity measurement. The detailed results of permeability and porosity measurement for sandstone cores are listed in Table 3.10. The average porosity of the sandstone samples is 11.47% and the average permeability is around 0.349 md. Both of the porosity and permeability of sandstone samples are larger than the concrete samples.
### Table 3.9: Averaged Acoustic Measurement Results for Sandstone Samples

<table>
<thead>
<tr>
<th>Property Name</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressional Velocity, Vp</td>
<td>7508 m/s</td>
</tr>
<tr>
<td>Shear Velocity, Vs</td>
<td>4868 m/s</td>
</tr>
<tr>
<td>Constraint Modulus, M</td>
<td>124 GPa (17.98 × 10^6 psi)</td>
</tr>
<tr>
<td>Shear Modulus, G</td>
<td>52 GPa (7.54 × 10^6 psi)</td>
</tr>
<tr>
<td>Bulk Modulus, K</td>
<td>55 GPa (7.98 × 10^6 psi)</td>
</tr>
<tr>
<td>Young’s Modulus, E</td>
<td>118 GPa (17.11 × 10^6 psi)</td>
</tr>
<tr>
<td>Poisson’s Ratio,</td>
<td>0.142</td>
</tr>
</tbody>
</table>

Figure 3.16: Sandstone Core Samples Used for Permeability and Porosity Measurement.

### Table 3.10: Permeability and Porosity Results from CMS-300 for Sandstone Samples

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>SS-1</th>
<th>SS-2</th>
<th>SS-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>1.000 inch</td>
<td>1.001 inch</td>
<td>1.001 inch</td>
</tr>
<tr>
<td>Length</td>
<td>1.127 inch</td>
<td>1.094 inch</td>
<td>1.048 inch</td>
</tr>
<tr>
<td>Weight</td>
<td>31.83 g</td>
<td>30.74 g</td>
<td>29.62 g</td>
</tr>
<tr>
<td>Bulk Volume</td>
<td>14.505 cc</td>
<td>14.108 cc</td>
<td>13.515 cc</td>
</tr>
<tr>
<td>Bulk Density</td>
<td>2.194 g/cc</td>
<td>2.179 g/cc</td>
<td>2.192 g/cc</td>
</tr>
<tr>
<td>Pore Volume</td>
<td>1.671 cc</td>
<td>1.610 cc</td>
<td>1.565 cc</td>
</tr>
<tr>
<td>Porosity</td>
<td>11.52%</td>
<td>11.41%</td>
<td>11.58%</td>
</tr>
<tr>
<td>Permeability</td>
<td>0.609 md</td>
<td>0.252 md</td>
<td>0.187 md</td>
</tr>
</tbody>
</table>
3.3.4 Brazilian Test

Figure 3.17 shows sandstone samples for Brazilian tests. Figure 3.18 shows a typical load-and-deformation curve for sandstone samples during Brazilian tests. The results are listed in Table 3.11. The average tensile strength of concrete samples is 2.878 MPa (418 psi).

![Figure 3.17: Sandstone Samples for Brazilian Test.](image)

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>SS-1 Br</th>
<th>SS-2 Br</th>
<th>SS-3 Br</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>1.499 inch</td>
<td>1.497 inch</td>
<td>1.497 inch</td>
</tr>
<tr>
<td>Thickness</td>
<td>0.742 inch</td>
<td>0.751 inch</td>
<td>0.771 inch</td>
</tr>
<tr>
<td>Maximum Loading Force</td>
<td>5372 N</td>
<td>5196 N</td>
<td>4896 N</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>4.769 MPa</td>
<td>4.561 MPa</td>
<td>4.186 MPa</td>
</tr>
<tr>
<td></td>
<td>(691 psi)</td>
<td>(661 psi)</td>
<td>(607 psi)</td>
</tr>
</tbody>
</table>

3.3.5 Uniaxial Compression Test

Figure 3.19 shows sandstone samples for uniaxial compression tests. A typical loading curve is shown in Figure 3.20. The results are presented in Table 3.12. The average uniaxial compressive strength of sandstone samples is 41.457 MPa (6013 psi).
Figure 3.18: Typical Brazilian Test Deformation Curve for Sandstone Samples.

Figure 3.19: Sandstone Samples for Uniaxial Compression Test.
Figure 3.20: Typical Uniaxial Compression Loading Curve for Sandstone Samples.

Table 3.12: Uniaxial Compressive Strength Results for Sandstone Cores

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>SS-1 UC</th>
<th>SS-2 UC</th>
<th>SS-3 UC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>1.499 inch</td>
<td>1.496 inch</td>
<td>1.496 inch</td>
</tr>
<tr>
<td>Thickness</td>
<td>3.012 inch</td>
<td>2.990 inch</td>
<td>3.030 inch</td>
</tr>
<tr>
<td>Maximum Loading Force</td>
<td>10,592 lb</td>
<td>13,657 lb</td>
<td>7,493 lb</td>
</tr>
<tr>
<td>Uniaxial Compressive Strength</td>
<td>41.410 MPa (6006 psi)</td>
<td>53.569 MPa (7770 psi)</td>
<td>29.391 Mpa (4263 psi)</td>
</tr>
</tbody>
</table>
### 3.3.6 Specific Heat

The specific heat of sandstone samples ranged from $782 \text{J/(kg·K)}$ to $922 \text{J/(kg·K)}$, with an average of $857 \text{J/(kg·K)}$ (Table 3.13).

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Specific Heat</th>
<th>Average Specific Heat</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS-1</td>
<td>$782 \text{J/(kg·K)}$</td>
<td></td>
</tr>
<tr>
<td>SS-2</td>
<td>$922 \text{J/(kg·K)}$</td>
<td></td>
</tr>
<tr>
<td>SS-3</td>
<td>$868 \text{J/(kg·K)}$</td>
<td></td>
</tr>
<tr>
<td>SS-4</td>
<td>$857 \text{J/(kg·K)}$</td>
<td></td>
</tr>
</tbody>
</table>

### 3.4 Properties of Shale

The shale samples were gathered from a shallow buried layer of Niobrara formation, which is about 10 feet to 30 feet under the surface, north of Boulder, CO. This section covers the measurements for shale samples.

#### 3.4.1 Specimen Preparation

The cores were dry drilled from big chunks, also from which several 8 by 8 by 8 blocks are cut for tri-axial tests. After coring, all shale cores are dry cut into the desired length with their cross-sections ground with great care.

#### 3.4.2 Acoustic Measurement

The results from acoustic measurements are presented in Table 3.14. The shale samples have the highest moduli in all three rock types.

#### 3.4.3 Permeability and Porosity

Figure 3.21 shows the core samples used for permeability and porosity measurements. The results of permeability and porosity measurements for shale cores are listed in Table 3.15. The average porosity of the shale samples is 6.65% and the average permeability is around 1 microdarcy ($1.06 \times 10^{-3}$ md). Porosity and permeability of shale samples are the lowest
Table 3.14: Acoustic Measurement Results for Shale Samples

<table>
<thead>
<tr>
<th>Property Name</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressional Velocity, $V_p$</td>
<td>4920 to 5001 m/s</td>
</tr>
<tr>
<td>Shear Velocity, $V_s$</td>
<td>2767 to 2816 m/s</td>
</tr>
<tr>
<td>Constraint Modulus, $M$</td>
<td>61.2 GPa ($8.88 \times 10^6$ psi)</td>
</tr>
<tr>
<td>Shear Modulus, $G$</td>
<td>19.5 GPa ($2.83 \times 10^6$ psi)</td>
</tr>
<tr>
<td>Bulk Modulus, $K$</td>
<td>35.5 GPa ($5.15 \times 10^6$ psi)</td>
</tr>
<tr>
<td>Young’s Modulus, $E$</td>
<td>49.3 GPa ($7.15 \times 10^6$ psi)</td>
</tr>
<tr>
<td>Poisson’s Ratio,</td>
<td>0.268</td>
</tr>
</tbody>
</table>

of all rock types. The permeability of shale samples is about 100 times smaller than the sandstone samples.

![Shale Core Samples](image)

Figure 3.21: Shale Core Samples Used for Permeability and Porosity Measurement.

3.4.4 Brazilian Test

Figure 3.22 shows shale samples for Brazilian tests. Figure 3.23 shows a typical load-and-deformation curve for shale samples during Brazilian test. Shale samples, as showing in Figure 3.24, tend to be very ductile during tests, which results in several loading peaks in the loading curve. The results of tensile strength measurements for shale cores are in Table 3.16. The average tensile strength of shale samples is 8.455 MPa (1226 psi). The significant difference between two shale samples may be due to the difference in coring direction relative to the sedimentary bedding plane or pre-existing fractures within samples.
Table 3.15: Permeability and Porosity Results from CMS-300 for Shale Samples

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>SH-1</th>
<th>SH-2</th>
<th>SH-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>1.004 inch</td>
<td>1.004 inch</td>
<td>1.003 inch</td>
</tr>
<tr>
<td>Length</td>
<td>0.984 inch</td>
<td>1.031 inch</td>
<td>0.930 inch</td>
</tr>
<tr>
<td>Weight</td>
<td>30.20 g</td>
<td>32.05 g</td>
<td>28.96 g</td>
</tr>
<tr>
<td>Bulk Volume</td>
<td>12.766 cc</td>
<td>13.376 cc</td>
<td>12.041 cc</td>
</tr>
<tr>
<td>Bulk Density</td>
<td>2.366 g/cc</td>
<td>2.396 g/cc</td>
<td>2.405 g/cc</td>
</tr>
<tr>
<td>Pore Volume</td>
<td>0.873 cc</td>
<td>0.890 cc</td>
<td>0.776 cc</td>
</tr>
<tr>
<td>Porosity</td>
<td>6.84%</td>
<td>6.65%</td>
<td>6.44%</td>
</tr>
<tr>
<td>Permeability</td>
<td>$1.05 \times 10^{-3}md$</td>
<td>$1.05 \times 10^{-3}md$</td>
<td>$1.07 \times 10^{-3}md$</td>
</tr>
</tbody>
</table>

Figure 3.22: Shale Samples for Brazilian Test.

Table 3.16: Tensile Strength Results for Shale Cores

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>SH-2 Br</th>
<th>SH-3 Br</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>1.501 inch</td>
<td>1.494 inch</td>
</tr>
<tr>
<td>Thickness</td>
<td>0.743 inch</td>
<td>0.699 inch</td>
</tr>
<tr>
<td>Maximum Loading Force</td>
<td>12,624 N</td>
<td>6074 N</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>11.170 MPa</td>
<td>5.740 MPa</td>
</tr>
<tr>
<td></td>
<td>(1620 psi)</td>
<td>(832 psi)</td>
</tr>
</tbody>
</table>
Figure 3.23: Typical Brazilian Test Deformation Curve for Shale Samples.

Figure 3.24: Shale Sample SH-2 Br after Brazilian Test.
3.4.5 Uniaxial Compression Test

Figure 3.25 shows shale samples for uniaxial compression tests. The typical loading curve for shale samples is shown in Figure 3.26. The shale samples also appear to be ductile as in the Brazilian tests. The results of uniaxial compression tests are listed in Table 3.17. The average uniaxial compressive strength of shale samples is 54.585 MPa (7917 psi). There is significant variation among samples may be due to bedding plane or pre-existing fractures.

![Shale Samples for Uniaxial Compression Test.](image)

Table 3.17: Uniaxial Compressive Strength Results for Shale Cores

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Sample Diameter</th>
<th>Sample Thickness</th>
<th>Maximum Loading Force</th>
<th>Uniaxial Compressive Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>SH-1 UC</td>
<td>1.004 inch</td>
<td>2.057 inch</td>
<td>31,553 N</td>
<td>61.775 MPa (8960 psi)</td>
</tr>
<tr>
<td>SH-2 UC</td>
<td>0.755 inch</td>
<td>1.483 inch</td>
<td>10,804 N</td>
<td>37.404 MPa (5425 psi)</td>
</tr>
<tr>
<td>SH-3 UC</td>
<td>0.757 inch</td>
<td>1.486 inch</td>
<td>18,750 N</td>
<td>64.576 MPa (9366 psi)</td>
</tr>
</tbody>
</table>

3.4.6 Specific Heat

The specific heat for shale samples ranges from $916 J/(kg \cdot K)$ to $1067 J/(kg \cdot K)$, with an average of $990 J/(kg \cdot K)$. The results are listed in Table 3.18.
Table 3.18: Specific Heat of Shale Samples

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Specific Heat</th>
<th>Average Specific Heat</th>
</tr>
</thead>
<tbody>
<tr>
<td>SH-1</td>
<td>$988, J/(kg\cdot K)$</td>
<td>$990, J/(kg\cdot K)$</td>
</tr>
<tr>
<td>SH-2</td>
<td>$1067, J/(kg\cdot K)$</td>
<td></td>
</tr>
<tr>
<td>SH-3</td>
<td>$916, J/(kg\cdot K)$</td>
<td></td>
</tr>
</tbody>
</table>

Figure 3.26: Typical Uniaxial Compression Loading Curve for Shale Samples.
CHAPTER 4
LABORATORY EXPERIMENT

This chapter describes the equipment used in cryogenic fracturing tests, experiment procedures to conduct the treatment, post-treatment, and results for cryogenic fracturing on concrete and rock samples.

4.1 Laboratory Equipment

During the course of this study, a complete tri-axial loading system with a cryogen injection system and real-time temperature and pressure monitoring capability is built. This section describes this system in detail, including the tri-axial loading sub-system, cryogen injection sub-system, temperature sensors and pressure transducers. Figure 4.1 shows a complete view of the entire equipment. The experiment setup for temperature sensors and pressure transducers are adapted from Cha et al. (2014).

4.1.1 Tri-axial Loading Sub-System

The tri-axial loading sub-system is the main component of the equipment, which is custom-built for this research project to provide true three-dimensional confining stresses to simulate in-situ stress condition. The loading system can provide up to 4500 psi (31 MPa) in horizontal directions and 6000 psi (41 MPa) in the vertical direction on an 8 inch cubed sample. The stress range is sufficient for simulating most stress conditions in typical petroleum reservoirs.

There are three major components in the tri-axial loading sub-system: pneumatic hydraulic pumps, pistons and the loading frame. The pneumatic hydraulic pumps are powered by a high-pressure air source and controlled manually to pump hydraulic fluids into connected pistons to provide stress on sample surfaces. Figure 4.2 shows three pneumatic hydraulic pumps with monitoring gauges. Two smaller pumps are used to power the pistons in the
horizontal directions (x- and y-axis) and the larger one is used to power the piston in the vertical direction (z-axis). The pistons are the actual device that exerts stresses on samples. The two horizontal pistons are removable while the piston in the vertical direction is fixed on rolling frame. The loading frame contains a fixed bed with a containment ring, which is made by carbon steel to provide support for horizontal pistons as shown in Figure 4.3, and the rolling frame. The rolling frame can be moved to the end of the fixed bed when not in use, which provides easier access to arrange the layout of the loading inside the containment ring. The roll frame also provides support for the vertical piston, which is shown in Figure 4.4.

4.1.2 Cryogen Injection Sub-System

The cryogen injection sub-system is capable of injecting a cryogenic fluid and a high-pressure gas to the borehole in the rock sample. It contains a high-pressure gas source, a liquid nitrogen (or other cryogenic fluid) source, a high-pressure gas accumulator, a cryogenic
Figure 4.2: Three Pneumatic Hydraulic Pumps.

Figure 4.3: Pistons in Horizontal Directions within Containment Ring.
Figure 4.4: Vertical Piston on Rolling Frame and Containment Ring.
fluid vessel, several valves and tubings connected to the samples. A schematic drawing of the cryogen injection sub-system for liquid nitrogen injection is shown in Figure 4.5. For low-pressure cryogenic fluid injection or circulation, the cryogenic fluid can be directly drawn from the dewar, which is automatically pressurized by vaporization of the cryogenic fluid. With the cryogenic fluid vessel, which is made of stainless steel and shown in Figure 4.6, cryogen can be first drawn into the vessel and then pressurized by the high-pressure gas pre-stored in the gas accumulator up to 500 psi for pressurized injection. The total volume of the vessel is 0.95 L.

4.1.3 Temperature Sensors

Temperature sensors used in experiment are Type T thermocouples, which are made of copper and constantan and suited for temperature measurement in the range of $-200$ to $350^\circ C$ ($-380$ to $392^\circ F$). There are up to seven thermocouples used in different experiments. Thermocouple No.1 (TC1) is attached to the borehole wall inside wellbore in samples. TC2 is suspended inside wellbore. TC3 and TC4 are attached to the surface of samples. TC5, TC6 and TC7 are used for additional temperature data collection at flexible positions.

4.1.4 Pressure Transducers

Pressure transducers are used to monitor pressure inside wellbore of samples, and injection gas pressure in gas accumulator. The pressure transducers used are rated up to 3000 psi and connected to the data acquisition system, which can provide real time reading or monitoring while testing. The pressure transducer connected to wellbore is placed about 5 inch higher than the top surface of sample, as shown in Figure 4.5, to avoid liquid nitrogen getting in touch with it and thus resulting in inaccurate reading.

4.2 Experiment Procedures

This section introduces the actual testing procedure for conducting experiment. There are two different tests with different purposes. Pressure decay test is aiming for evaluation
Figure 4.5: Schematic for Cryogen Injection Sub-System.

Figure 4.6: Cryogenic Fluid Vessel (before insulation).
of permeability of the sample as a whole. Confined cryogenic fracturing test is to conduct cryogen treatment on samples under simulated stress condition.

4.2.1 Pressure Decay Test

The pressure decay test is performed to evaluate the permeability of samples. The wellbore of a concrete or rock sample is first pressurized by gas nitrogen to 175 psi (1.2 MPa) with the venting needle valve closed. Then, the inlet of wellbore is shut by closing the needle valve on the inflow tubing. The gas inside wellbore and in the connected tubing space is forced by pressure to flow through the sample to the ambient environment, causing the pressure to decay with time. The pressure decay curve can be used to characterize the average permeability of the rock sample. By comparing pressure decay curve before and after each cryogenic fluid treatment, the effectiveness of the treatment can be revealed. In addition, by comparing the experimental decay curve to that obtained from simulations, the average permeability of the sample before and after treatment can be back calculated.

4.2.2 Confined Cryogenic Fracturing Test

Confined cryogenic fracturing test is the most important and the major test of this research project. The purpose of this test is to create fractures around the wellbore of samples by injecting cryogenic fluid at different pressures (in most cases, injection pressure is slightly higher than the ambient pressure) under three dimensional confining stresses.

These tests are conducted on different types of samples, such as concrete, sandstone and shale, with dimension of 8 inch by 8 inch by 8 inch. Samples are pre-drilled for a 6-inch deep borehole with a steel casing attached by epoxy. A wellhead type of connection is attached to the stainless steel casing creating a tubing space and an annulus space for better injection control. Once tubings and fittings are properly installed and fastened, the sample are then confined by three hydraulic pumps. The stresses on the samples are increased by controlled to follow the ratio of stresses in x-, y- and z-directions in order to minimize premature failure caused by unbalanced stress loading. Pressure decay tests are then performed to characterize
the permeability of the sample. Next, liquid nitrogen is flowed into the borehole to treat the sample. The typical treating time for circulating liquid nitrogen is 20 to 40 minutes. For higher pressure injection, the cryogenic fluid vessel is used to pressurize liquid nitrogen with higher pressure gas nitrogen. Since the volume of the cryogenic fluid vessel is very limited and one high-pressure treatment may not generate sufficient thermal-shock, multiple high-pressure treatments may be conducted in series by refilling the vessel.
CHAPTER 5
NUMERICAL MODELING

In this chapter, the theory and work flow of the simulation tool developed for this research is introduced. The objective for developing the simulation tool is to evaluate the distribution and the effect of thermally induced fractures during cryogenic fracturing treatments. This simulation tool is modified from TOUGH2-EGS (Enhanced Geothermal System), which is a coupled geomechanical and reactive geochemical simulator for fluid and heat flows in an enhanced geothermal system. With the ability of TOUGH2-EGS and modification on fracture initiation and propagation, this simulation tool can simulate cryogenic fracturing processes and predict the distribution of fractures.

5.1 Theoretical Analysis

Cryogenic fracturing is a very complex process that involves hydraulics, thermodynamics, and rock mechanics. In order to analyze this process, several assumptions have been made to simplify the physics. In addition, heat transfer, thermal stresses and failure criteria are addressed in this section.

5.1.1 Assumptions

In order to simplify the development of the simulation tool, several assumptions have been made:

- For heat transfer, only heat conduction is considered, which means that both advection and radiation are neglected. In porous media, the contacting area between fluid and rock surfaces is very large per unit volume of fluid. This indicates that heat conduction plays a much more important role than advection and radiation during a short period of cryogenic treatment. In each grid block, the temperature of the rock matrix is always the same as that of the fluid in the pore volume.
• For fracturing processes, the stress change in the rock matrix includes thermal expansion or contraction due to the change in temperature, fluid pressure in pores, and external stress condition, as imposed by the hydraulic press and pistons in the experiment. The principal stress directions follow the loading direction in the tri-axial experiments.

• The rock matrix is assumed to be homogeneous within each grid block. The heterogeneity of the sample is achieved by assigning different rock properties to different grid blocks.

• For natural fractures, since they are very difficult to characterize, pre-existing natural fractures are neglected. Only the fractures generated by the cryogenic treatment are considered and tracked.

5.1.2 Heat Transfer and Fluid Flow

The heat transfer and fluid flow model in the simulation tool is adapted from the TOUGH2-EGS simulator. The governing equation for mass and heat balance can be written in the form according to Fakcharoenphol et al. (2013):

\[
\frac{d}{dt} \int_{V_n} Q^\kappa dV_n = \int_{\Gamma_n} F^\kappa \cdot \vec{n} d\Gamma_n + \int_{V_n} q^\kappa dV_n \quad (5.1)
\]

where:

\( \kappa = 1, ..., NK \) (total number of components);

\( n = 1, ..., NEL \) (total number of grid blocks);

\( V_n \) is an arbitrary subdomain of the system under study;

\( \Gamma_n \) is the closed surface by which the subdomain is bounded by;

\( Q \) is the quantity represents mass or energy per volume;

\( F \) is mass or heat flux;
\( q \) is sinks and sources;

\( \vec{n} \) is a normal vector on surface element \( d\Gamma_n \) pointing inward into \( V_n \).

### 5.1.3 Thermal Stress

Thermal stress is the stress change caused by temperature change within a solid material. It is the most important parameter when simulating the cryogenic fracturing process. The thermally induced stress can be integrated into the generalized stress-strain relation in a rock volume, as shown below:

\[
\sigma_{kk} - B_i \times p_p - \frac{E}{(1 - 2\nu)} \left[ \beta (T - T_0) \right] = \frac{E}{(1 + \nu)} \varepsilon_{kk} + \frac{E\nu}{(1 + \nu)(1 - 2\nu)} (\varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz}) \tag{5.2}
\]

where:

- \( \sigma \) is the normal stress;
- \( \varepsilon \) is the strain;
- Subscript \( kk \) is direction, which can be \( xx \), \( yy \) and \( zz \);
- \( B_i \) is the Biot number of the rock;
- \( \beta \) is the linear thermal expansion of the rock;
- \( E \) is the Young’s modulus;
- \( \nu \) is the Poisson’s ratio;
- \( p_p \) is the pore pressure;
- \( T \) is the current temperature;
- \( T_0 \) is the reference or original temperature.
5.1.4 Failure Criteria

A failure criterion is used to judge the condition of rock fracturing. It gives the maximum strength of rock under certain stress conditions. Once the stress exceeds the maximum strength given by the failure criterion, the rock will break, in other words, be fractured. The current failure model used in this simulating tool is the Mogi-Coulomb Failure Criterion, which is first introduced by Al-Ajmi and Zimmerman (2006) and widely used in rock mechanics. The Mogi-Coulomb Failure Criterion has the following form:

\[ \tau_{oct} = k + m\sigma_{oct} \]  

where:

- \( \tau_{oct} \) is the octahedral shear stress;
- \( \sigma_{oct} \) is the octahedral normal stress;
- \( k \) is the Mogi-Coulomb intercept;
- \( m \) is the Mogi-Coulomb slope.

The octahedral shear and normal stresses are defined as:

\[ \tau_{oct} = \frac{1}{3} \sqrt{(\sigma_v - \sigma_{Hmax})^2 + (\sigma_v - \sigma_{hmin})^2 + (\sigma_{Hmax} - \sigma_{hmin})^2} \]  

\[ \sigma_{oct} = \frac{1}{3} (\sigma_v + \sigma_{Hmax} + \sigma_{hmin}) \]

where:

- \( \sigma_v \) is the vertical stress;
- \( \sigma_{hmin} \) is the minimum horizontal stress;
- \( \sigma_{Hmax} \) is the maximum horizontal stress.

In the above equations, \( k \) and \( m \) are constant that are usually obtained from fitting actual data. The failure envelope from Mogi-Coulomb Failure Criterion is as shown in Figure 5.1.
Normally the stress conditions of rock make the calculated $\tau_{oct}$ and $\sigma_{oct}$ fall into the blue area under the failure envelope. When the stress condition changes, i.e. due to cryogenic treatment, the calculated $\tau_{oct}$ and $\sigma_{oct}$ may fall onto a point outside of the failure envelope, the rock would then be fractured. Mogi-Coulomb Failure Criterion is simple and easy to adapt in simulation and has similar accuracy with other failure criteria when assuming that the physical properties of rock remain the same with temperature change.

![Failure Envelope of Mogi-Coulomb Criterion by Aadnoy and Looyeh (2011).](image)

Figure 5.1: Failure Envelope of Mogi-Coulomb Criterion by Aadnoy and Looyeh (2011).

### 5.2 Problem Setup

The simulation tool simulates the cryogenic fracturing process using a control volume finite difference method. The basic geometry of the simulated well is the same as that in the experiment: the cryogenic fluid flows into a borehole and cools its surface. Then, the fluid will permeate through the porous medium through the inner surface of the borehole. The domain dimensions are set as 8 inch by 8 inch by 8 inch, identical to the dimension of the sample in the actual experiment.
5.2.1 Geometry

The details of this case are as follows. The domain is a rock sample cube with outer dimensions 20.32 cm x 20.32 cm x 20.32 cm (8 inch x 8 inch x 8 inch). A 2.54 cm (1 inch) diameter borehole is centrally located on top surface extending 15.24 cm (6 inch) into the block. The upper 5.08 cm (2 inch) section of borehole will be cased, which means no fluid flow through this section into samples. Figure 5.2 shows the schematic of geometry for modeling.

![Figure 5.2: Schematic Drawing for Modeling Geometry.](image)

5.2.2 Outer Boundary

The outer boundary, which consists of the six surfaces of the sandstone block, is exposed to ambient pressure and temperature in the laboratory, which are 11.8 psia (81.4 KPa) and
66°F (19°C). The sample is initially set at the ambient temperature, which is 66°F (19°C).

The stress condition is set the same as that in the experiment such that simulation and experiments results can be matched and compared.

5.2.3 Mesh Design

The meshing procedure is adapted from TOUGH2-EGS, which is relatively simple. The samples are meshed into cubic cells with equal lengths in x-, y- and z-direction. Typically, a finer meshing with smaller cell size yields better resolution of the geometric features and more accurate results.

There are three types of grid blocks used in this simulation tool. The normal grid, which can be considered as the intact rock material, has the same properties with the measured rock properties. The fractured grid has larger permeability due to fractures induced by thermal shock in cryogenic fluid treatment. The other properties remain the same with the normal grid. However, since fluid flow increases with higher permeability in this kind of grid, fractured grid blocks generally appear to be more thermally conductive. The third type of grid is the wellbore grid, which is set to have the same property with void space. If the center of a grid block falls within the borehole space, it is set as wellbore grid.

5.3 Flow Chart

The basic work flow of the simulation tool follows the original work flow of TOUGH2-EGS with modification on fractured grids judgement according to the Mogi-Coulomb Failure Criterion. The exact work flow is presented in Figure 5.3. After input of pre-prepared input file, the simulation tool reads the input file and initialize fluid, heat and stress variables. Then it build Jacobian matrix for residual equations of fluid, heat and stress and then compute through an iteration process with the original thermal, hydraulic and mechanical module. Once converged, the primary and secondary variables such as pressure, temperature, mass fraction and stress are updated with new values at current time step. Then the modified mechanical module solves for the octahedral stresses for each grid with the stress condition
data. Next, these data are plugged into the Mogi-Coulomb Criterion. If the octahedral stresses of a grid indicates it is fractured, this grid will be set as the fractured grid with higher permeability. After the judgement, the grid domain will be updated and the program continues to next time step until the maximum time step has been reached.
Figure 5.3: Work Flow Chart of the Simulation Tool.
CHAPTER 6
RESULTS OF EXPERIMENT AND SIMULATION

In this chapter, results of confined tests and simulations for four Niobrara shale samples through different treatment processes are presented and compared.

6.1 Experiment Conditions

The four shale samples were treated with different cryogen injection pressure, treating time or stages, and confining stress condition. These conditions were varied to investigate the effect of different parameters on the final treatment results. The detailed experiment conditions and procedures are listed in Table 6.1.

Table 6.1: Experiment Conditions for Shale Samples

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Stress Condition (x-y-z)</th>
<th>Test Procedure</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shale-1</td>
<td>1000-1500-2000 (psi)</td>
<td>Flowing LN2 under low pressure (40 min)</td>
<td>CT scanned before and after the LN2 test.</td>
</tr>
<tr>
<td></td>
<td>6.89-10.34-13.79 (MPa)</td>
<td>Flowing LN2 under high pressure</td>
<td>3 LN2 treatment cycles.</td>
</tr>
<tr>
<td>Shale-2</td>
<td>1000-3000-4000 (psi)</td>
<td>Flowing LN2 under high pressure (1st round)</td>
<td>3 LN2 treatment cycles for each round.</td>
</tr>
<tr>
<td></td>
<td>6.89-20.68-27.58(MPa)</td>
<td>Flowing LN2 under high pressure (2nd round)</td>
<td></td>
</tr>
<tr>
<td>Shale-3</td>
<td>1000-1500-2000 (psi)</td>
<td>Flowing LN2 under low pressure (40 min)</td>
<td>The sample failed during decay test.</td>
</tr>
<tr>
<td></td>
<td>6.89-10.34-13.79 (MPa)</td>
<td>Flowing LN2 under high pressure</td>
<td></td>
</tr>
<tr>
<td>Shale-4</td>
<td>1000-1500-2000 (psi)</td>
<td>Fracking by GN2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6.89-10.34-13.79 (MPa)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

6.2 Simulation Configurations

For modeling, the thermal diffusivity of shale sample is set according to Robertson (1988) as $8.00 \times 10^{-7} m^2/s$. The constants from Mogi-Coulomb criterion are set as $k = 230$ psi (1.59 MPa) and $m = 0.58$, which are fitted by experiment data from these four shale samples. The permeability of fractured grids is set as 200 md. All basic input parameters of the simulation can be found in Table 6.2.
### Table 6.2: Input Parameters for Simulation

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient Pressure</td>
<td>11.8 psi (81.4 KPa)</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>66°F (19°C)</td>
</tr>
<tr>
<td>Rock Density</td>
<td>2.38 g/cc</td>
</tr>
<tr>
<td>Permeability</td>
<td>$1.05 \times 10^{-3}$ md</td>
</tr>
<tr>
<td>Permeability of Fractured Grid</td>
<td>200 md</td>
</tr>
<tr>
<td>Porosity</td>
<td>8%</td>
</tr>
<tr>
<td>Rock Compressibility</td>
<td>$2 \times 10^{-3} \text{psi}^{-1}$ (2.9 $\times 10^{-7}$ Pa)</td>
</tr>
<tr>
<td>Thermal Diffusivity</td>
<td>$8 \times 10^{-7} \text{m}^2/\text{s}$</td>
</tr>
<tr>
<td>Thermal Expansion Coefficient</td>
<td>$2.7 \times 10^{-5}$ $\degree C^{-1}$</td>
</tr>
<tr>
<td>Specific Heat</td>
<td>990 J/(kg $\cdot$ K)</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>$7.15 \times 10^6$ psi (4.93 $\times 10^4$ MPa)</td>
</tr>
<tr>
<td>Poisson’s Ratio</td>
<td>0.268</td>
</tr>
<tr>
<td>Mogi-Coulomb Constant, k</td>
<td>230 psi (1.59 MPa)</td>
</tr>
<tr>
<td>Mogi-Coulomb Slope, m</td>
<td>0.58</td>
</tr>
</tbody>
</table>

### 6.3 Results

In this section, experimental results for four shale samples are presented. The results from simulations for each sample are also described, matched and compared with the experiment to evaluate the effect of different test procedures and conditions.

#### 6.3.1 Shale Sample No. 1

Shale sample number 1 (Shale-1) was treated two rounds with liquid nitrogen under the room temperature. The confining stress profile used for this sample is 1000 psi (6.89 MPa) in x direction, 1500 psi (10.34 MPa) in y direction and 2000 psi (13.79 MPa) in z direction. The first liquid nitrogen treatment is a low-pressure (about 15 psi or 0.1 MPa) circulation and lasts for 40 minutes. The second treatment includes three cycles of high-pressure liquid nitrogen injection with outlet partially open to provide opportunities for circulation while maintaining a back pressure.

The results of pressure decay tests before and after each treatment is shown in Figure 6.1. The average permeability matched from simulation for Shale-1 before the first cryogen treatment is $1.30 \times 10^{-3}$ md, after the first treatment is $1.65 \times 10^{-3}$ md, and after the second...
treatment is $3.45 \times 10^{-3}$ md. The first round of low-pressure liquid nitrogen circulation increases the average permeability of Shale-1 to 1.26 times to its original value. The second round of high-pressure liquid nitrogen treatment increases the permeability to 2.64 times of its original value.

![Figure 6.1: Pressure Decay Tests for Shale-1.](image)

The simulation cases for Shale-1 include two situations. The first one is a low-pressure liquid nitrogen circulating through the borehole of the sample. The pressure used for injection is 15 psi (0.1 MPa) and the time for injection is 40 minutes (2400 seconds). The temperature distribution of Shale-1 after the first low-pressure liquid nitrogen treatment is shown in Figure 6.2. The fractured grid distribution is shown in Figure 6.3. The results show that although heat conduction is not affected by fractures, the increased fluid or gas flow in fractures results in directional temperature distribution inside Shale-1. The fracture half-length after first treatment is about 0.6 inch (1.52 cm) around wellbore in y direction. The average permeability matched from simulation is $1.30 \times 10^{-3}$ md, which is 1.3 times of its
original value \((1.00 \times 10^{-3} \text{ md})\).

The second test is a high-pressure liquid nitrogen injection. The injection pressure is set as 450 psi (3.10 MPa) and time of injection for each cycle is 15 seconds, which is the approximate time for high-pressure treatment in the experiment. After the injection, there is 10-minute relaxation for the sample to warm up. The pressure distribution in Shale-1 after the second high-pressure liquid nitrogen treatment is shown in Figure 6.4. The temperature distribution is shown in Figure 6.5 and the fractured grid distribution is shown in Figure 6.6. The results show that the high pressure treatment extends the existing fracture grids to the
Figure 6.3: Fractured Grids Distribution in Shale-1 after the First Treatment.
direction perpendicular to the minimum horizontal stress direction. The fracture half-length after the second treatment increased to about 0.9 inch (2.29 cm) around the wellbore in y direction. The average permeability matched from simulation is $2.25 \times 10^{-3}$ md, which is 2.25 times of its original value.

Finally, the results of experiment and simulation for Shale-1 are compared side-by-side in Table 6.3. Clearly, the average permeability improvements from simulation are in reasonable agreement with those in the experiment.

Figure 6.4: Pressure Distribution in Shale-1 after the Second Treatment.
Figure 6.5: Temperature Distribution in Shale-1 after the Second Treatment.

Table 6.3: Experiment and Simulation Results for Shale-1

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Experiment</th>
<th>Simulation</th>
<th>Fracture Half-length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before treatment</td>
<td>$1.30 \times 10^{-3}$ md</td>
<td>$1.00 \times 10^{-3}$ md</td>
<td>0</td>
</tr>
<tr>
<td>After low pressure circulation for 40 mins</td>
<td>$1.65 \times 10^{-3}$ md</td>
<td>$1.30 \times 10^{-3}$ md</td>
<td>0.6 inch</td>
</tr>
<tr>
<td>After 3 cycles of high pressure injection</td>
<td>$3.45 \times 10^{-3}$ md</td>
<td>$2.25 \times 10^{-3}$ md</td>
<td>0.9 inch</td>
</tr>
</tbody>
</table>
Figure 6.6: Fractured Grid Distribution in Shale-1 after the Second Treatment.
6.3.2 Shale Sample No. 2

Shale sample number 2 (Shale-2) is treated twice with high-pressure liquid nitrogen under the room temperature. The confining stress profile used for this sample is 1000 psi (6.89 MPa) in x direction, 3000 psi (20.68 MPa) in y direction and 4000 psi (27.58 MPa) in z direction. Both treatment used a pressure of about 450 psi (3.10 MPa) and contained three cycles of liquid nitrogen injection.

The results of pressure decay tests before and after each treatment is shown in Figure 6.7. The average permeability matched from simulation for Shale before the first cryogen treatment is $2.90 \times 10^{-4}$ md, after the first treatment is $1.25 \times 10^{-3}$ md, and after the second treatment is $2.90 \times 10^{-3}$ md. The first round of high-pressure liquid nitrogen treatment increases the average permeability of Shale-2 to 4.32 times to its original value. The second round of high pressure liquid nitrogen treatment increases the permeability to 10 times of its original value.

After the cryogen treatment, Shale-2 is fractured by high-pressure gas nitrogen. The section area of fracture plane shows a clear profile for fracture induced by thermal shock, as shown in Figure 6.8. The thermal fracture profile shows a slight deviation in direction with pressure induced fracture. This observation may indicate that thermal shock may cause local stress re-orientation. The induced fracture half-length is 1.1 inch (2.79 cm) from borehole wall.

The simulation cases for Shale-2 include two rounds. Both of them are high-pressure liquid nitrogen injections. For each round, the treatment procedures are the same with the second case for Shale-1 with higher contrast in the confining stress anisotropy. High-pressure liquid nitrogen is injected at 450 psi (3.10 MPa) for three cycles with 10 minutes relaxation between each cycle. In addition, there is a sufficient long period of time between the two rounds to simulate the warm up period for Shale-2. After the first round, distributions of pressure, temperature and fractured grids are shown in Figure 6.9, Figure 6.10, and Figure 6.11, respectively. With a higher contrast in the confining stress, fractured grids should
Figure 6.7: Pressure Decay Tests for Shale-2.

Figure 6.8: Fracture Plane of Shale-2.
be more distributed along the plane perpendicular to the minimum horizontal stress. However, the results show that although the number of fractured grids is larger, the distribution of fractured grids is more circular. This may be related to the simple stress calculation function adapted in the simulation tool and the mesh used. Also, with a high contrast in the confining stress, grids tend to be fractured more easily under lower pressure and smaller temperature change. Thus, there are more fractured grids outside the fracture plane. The fracture half-length after the first treatment is about 0.8 inch (2.03 cm) in y direction and 0.4 inch (1.02 cm) in x direction. The average permeability from matching with simulation is $3.80 \times 10^{-3}$ md, which is 3.80 times of its original value.

Figure 6.9: Pressure Distribution in Shale-2 after the First Treatment.
Figure 6.10: Temperature Distribution in Shale-2 after the First Treatment.
Figure 6.11: Fractured Grids Distribution in Shale-2 after the First Treatment.
For the second round, distributions of pressure, temperature and fractured grids are shown in Figure 6.12, Figure 6.13, and Figure 6.14, respectively. The fracture half-length after the first treatment is about 0.7 inch (1.24 cm) in y direction and 0.4 inch (1.02 cm) in x direction. The average permeability from simulation is $6.10 \times 10^{-3}$ md, which is 6.1 times of its original value. All results of experiment and simulation for Shale-2 are shown in Table 6.4.

Figure 6.12: Pressure Distribution in Shale-2 after the Second Treatment.
Figure 6.13: Temperature Distribution in Shale-2 after the Second Treatment.

Table 6.4: Experiment and Simulation Results for Shale-2

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Experiment</th>
<th>Simulation</th>
<th>Fracture Half-length</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average Permeability</td>
<td>Improvement</td>
<td>Average Permeability</td>
</tr>
<tr>
<td>Before treatment</td>
<td>2.90 × 10⁻³ md</td>
<td>1.00</td>
<td>1.00 × 10⁻³ md</td>
</tr>
<tr>
<td>After 1st round of high pressure</td>
<td>1.25 × 10⁻³ md</td>
<td>4.32</td>
<td>3.80 × 10⁻³ md</td>
</tr>
<tr>
<td>injection</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>After 2nd round of high pressure</td>
<td>2.90 × 10⁻³ md</td>
<td>10</td>
<td>6.10 × 10⁻³ md</td>
</tr>
<tr>
<td>injection</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 6.14: Fractured Grids Distribution in Shale-2 after the Second Treatment.
6.3.3 \textbf{Shale Sample No. 3}

Shale sample number 3 (Shale-3) was planned to be treated twice with liquid nitrogen under the room temperature following exact the same procedures with Shale-1. The confining stress profile used for this sample is 1000 psi (6.89 MPa) in x direction, 1500 psi (10.34 MPa) in y direction and 2000 psi (13.79 MPa) in z direction. However, after the second cycle of low-pressure liquid nitrogen treatment, Shale-3 was fractured during the pressure build-up process of pressure decay test. The possible reasons for Shale-3 fractured at an early stage of treatment include pre-existing natural fractures, weak sedimentary layers, and induced defects during drilling of the wellbore.

The results of the pressure decay tests before and after the first treatment are shown in Figure 6.15. The average permeability from simulation for Shale-3 before the first cryogen treatment is $1.32 \times 10^{-3}$ md, and after the first treatment is $2.52 \times 10^{-3}$ md. The first round of low pressure liquid nitrogen circulation increases the average permeability of Shale-3 to 1.91 times of its original value.

Since testing condition of Shale-3 is identical to that of Shale-1, no simulation is performed for Shale-3.

6.3.4 \textbf{Shale Sample No. 4}

Shale sample number 4 (Shale-4) is directly fractured by high-pressure gas nitrogen to establish a reference for evaluation of cryogenic fracturing efficacy. The confining stress profile used for this sample is 1000 psi (6.89 MPa) in x direction, 1500 psi (10.34 MPa) in y direction and 2000 psi (13.79 MPa) in z direction. The break down pressure for Shale-4 is about 2460 psi (18.2 MPa), which is shown in Figure 6.16. Figure 6.17 shows pictures of all surfaces of Shale-4 after fractured by high-pressure gas. An observation from the surfaces of Shale-4 is that the fracture is basically in yz plane (surface 2, 4, top and bottom in Figure 6.17), which is perpendicular to direction of minimum horizontal stress.
Figure 6.15: Pressure Decay Test for Shale-3.
Figure 6.16: Breakdown Pressure of Shale-4.

Figure 6.17: Sample Shale-4 after Fractured by High Pressure Gas.
A pressure-only (i.e. no cryogen) simulation was run for Shale-4, which provides a fracture distribution as shown in Figure 6.18. Fractured grids for this case are mostly distributed in the yz plane, which agrees with the fracture description for Shale-4 mentioned above. Fractures induced by high-pressure nitrogen gas have reached the surfaces on z directions (top and bottom), causing most of gas flow leaks out to ambient environment through these grids. The breakdown pressure matched by Mogi-Coulomb criterion using parameters in Table 6.2 for this case is 2472 psi (17.1 MPa), which is very close to the actual breakdown pressure shown in Figure 6.16.
CHAPTER 7
CONCLUSIONS AND FUTURE WORK

This research proves the feasibility of using cryogenic fluid as a fracturing fluid for stimulation treatment on concrete and shale samples in the laboratory. In addition, a simulation tool for modeling cryogenic fracturing processes incorporating heat and mass transfer and fracture initiation and propagation was developed. This work has clearly demonstrated how different cryogenic treatment conditions affect the initiation and growth of the fractures using both experimental and modeling approaches.

7.1 Conclusions

In this research, we have reached the following conclusions:

- The results of multiple cryogenic fracturing experiments have clearly shown the potential of cryogenic fracturing. The improvements on the average permeability of rock samples confirm that using solely cryogenic fluid as fracturing fluid is capable of generating fractures and enhancing productivity of an unconventional reservoir.

- With a low-pressure liquid nitrogen circulation, shale samples obtained from Niobrara formation show a general 50% incremental increase of the average permeability for 8 inch cubic blocks. This also implies that circulation of cryogenic fluid in wellbore at low pressure can be applied as a near-wellbore formation damage remediation technique with very low cost and without any environmental concerns.

- Effectiveness of injection pressure is demonstrated by comparing different shale samples undergoing various treatment procedures along with corresponding modeling results. High injection pressure tends to enhance cryogenic fracturing process in both speed and fracture conductivity. Shale samples treated with liquid nitrogen at high pressure
for a very short time have at least two times larger average permeability on 8 inch cubic blocks.

- Higher contrast in the confining stress anisotropy also aids the cryogenic fracturing process. The average permeability improvement for 8 inch cubic block under a high contrast confining stress condition is significantly higher than those under lower contrast confining stress conditions.

- A simulation tool modified from TOUGH2-EGS is developed for cryogenic fracturing experiments. It can provide predictions that are in good agreement with experiment.

- The current simulation tool can predict fracture distribution for cryogenic fracturing experiments under different stress conditions. After calibration that is specific to rock type, this simulation tool can provide reasonable fracture profile along with pressure and temperature distributions.

7.2 Future Work

Possible future directions and recommendations motivated by results of this research include:

- Laboratory results have shown some unexpected phenomena during testing, such as stress re-orientation caused by thermal shock. The theories behind these phenomena need to be addressed in future analysis and models.

- The consistency of experimental results is highly dependant on parameters such as the quality of sample preparation, heterogeneity of samples, and pre-existing fractures. A more rigorous quality control procedure needs to be established for more consistent laboratory results.

- The techniques to detect the fractures that leads to permeability improvement of samples have to be improved. Currently the only methods to evaluate cryogenic fracturing
treatment are pressure decay tests and simulation analyses, which converge on the average permeability of samples. Direct proof of initiated fractures is still lacking.

- The mechanical module in the simulation tool needs to be improved. The simulation tool is based on TOUGH2-EGS, which is a grid based reservoir simulator. Although TOUGH2-EGS solves thermal, hydraulic and mechanical processes in a fully coupled manner, the mechanical module is still over simplified for three-dimensional stress conditions with complex geometry. Thus, the stress field change around the wellbore is probably not adequately represented in this simulation tool.
REFERENCES CITED


