LABORATORY STUDY OF CRYOGENIC FRACTURING OF CONCRETE SAMPLES

by

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A thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Masters of Science (Petroleum Engineering)

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ABSTRACT

While hydraulic fracturing has revolutionized hydrocarbon production from unconventional resources, waterless, or reduced-water technologies, are actively being sought due to concerns arising from its heavy dependence on water and clay swelling in water sensitive formations. Although today it is possible to fracture with liquefied petroleum gas or CO₂, there has been very limited research on fracturing using cold liquefied gas. Some simple immersion tests to determine the effectiveness of liquid nitrogen as a fracturing fluid have been conducted with promising results (Grundmann 1998); however, there are no known comprehensive laboratory studies that exhaust the use of liquid nitrogen to determine its effectiveness. This study investigates the feasibility of wellbore fracturing by using pressurized cryogenic fluids to create a thermal gradient generating local tensile stresses on the borehole surface. The thermal tensile stress adds to the stresses incurred by pressurization, which, in many cases, leads to lowered breakdown pressure.
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ACKNOWLEDGMENTS

I would like to thank my advisor, Dr. Xiaolong Yin, for all of his support and guidance along the way to help me publish this thesis. I would also like to think my committee members, Dr. Yu-Shu Wu and Dr. Azra Tutuncu, for their supervision along the way. The majority of this thesis is centered on laboratory work and experimentation. This work could not have been completed without the help of Minsu Cha, Naif Alqahtani, and Bowen Yao. They all spent long restless hours in the lab with me to obtain the results presented in this thesis. I would also like to thank the petroleum department’s laboratory coordinator Joe Chen, as he was vital in the logistics of setting up the experiment and helping me obtain additional necessary equipment.
CHAPTER 1 INTRODUCTION

Traditional hydraulic fracturing technology, commonly known as fracking, uses a pressurized engineered water mixture at certain flowing conditions to fracture a formation. The fractures in the formation increase the drainage surface area, thus increasing oil and gas production. Although effective, there are many shortcomings associated with hydraulic fracturing that a waterless fracturing system can alleviate. Cryogenic fracturing is a concept that looks to expand and improve on traditional hydraulic fracturing technology. Cryogenic fracturing rests on the idea that a frigid liquid can induce fractures when brought into contact with a much warmer rock under downhole conditions. The cold liquid that effectuates such a fracture is known as the cryogenic fluid, or cryogen. When liquid nitrogen is injected into a formation whose temperature is drastically higher, the heat from the formation will quickly transfer to the liquid nitrogen. This rapid reduction of surface temperature causes the surface of the rock to contract and fail in tension, thus inducing fractures orthogonal to the contact plane of the cryogen and rock. The liquid nitrogen will also evaporate, which increases the pressure and helps to further develop the newly induced fractures. Liquid nitrogen has a liquid to gas expansion ratio of 1:694 at ambient conditions (Houghton, 2008). Though in the reservoir, the expansion will not be as significant, it still helps to create a high pressure gradient that propagates the fractures.

1.1 Background

Traditional hydraulic fracturing in low permeable formations uses a highly pressurized fracturing fluid to create a complex network of fractures. Then, proppants are mixed into the fracturing fluid to prop open the fractures and maintain their conductivity. These conductive fractures increase the contact area and allow more reservoir fluids to flow into the wellbore and be extracted. Hydraulic fracturing and the advancements associated with this technology have drastically changed the United States’ oil and gas producing abilities. Without a doubt hydraulic fracturing has revolutionized the exploitation of hydrocarbons in the United States and has helped sparked an energy boom.

Hydraulic fracturing technology relies on water-based fluids due to the general availability and low cost of water; however, the dependence upon water presents several major shortcomings. First, water can cause significant formation damage, which occurs as clay swells when contacted
with water and as the relative permeability of the formation is reduced by capillary retention of the fracturing fluid (Yost, et al., 1993). Formation damage mechanisms inhibit hydrocarbon flow and thus impair production rates and recovery efficiency. Stimulating a well with a waterless fracturing fluid may therefore provide higher production rates and ultimate recovery over fracturing with water. Second, water usage in large quantities may place significant stresses upon the local environments where fracturing activities occur. 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. The stress is even greater in drought stricken regions such as West Texas. Finally, the downhole injection of chemicals required in water-based fracturing programs, including slickwater and gel-based fracturing treatments, can lead to a contentious political climate. In recent times the topic of hydraulic fracturing has been so controversial that it is being outright banned in certain municipalities and states. In contrast to hydraulic fracturing, cryogenic fracturing offers potentially greater fracturing capabilities without the issues associated with water based fracturing fluids while mitigating potential reservoir damage from swelling clays and water retention. In addition to the advantages with using a waterless fracturing technology there is also less risk for ground water to be contaminated with pollutants. Since there is no water used in the process there would be no flowback associated with the stimulation treatment. Flowback from a hydraulic fracturing stimulation generally consists of salty water with a mixture of friction reducers and hydrocarbons. The costs and issues associated with properly treating and disposing of flowback would be eliminated if water-less fracturing was used.

Cryogenic fracturing, as a water-less fracturing technology, uses cold temperature in conjunction with pressure to initiate and propagate the fractures. Although little research on cryogenic fracturing has been conducted, some early work suggests promising results. King (1983) examined the use of gelled liquid carbon dioxide, instead of water, to stimulate tight gas sand formations. His primary motivation for finding an alternative to water as the fracturing fluid was to prevent formation damage. After performing the cryogenic fracturing treatment, the carbon dioxide would evaporate and not cause swelling near the wellbore in water sensitive formations. He also lists other benefits of liquid carbon dioxide use, including that carbon dioxide’s recovery rate does not depend on reservoir pressure, thus cleanup proceeds at a faster pace and carbon
dioxide’s high solubility in oil serves to lower oil viscosity and enhance oil production. Since the
gelled carbon dioxide that King used was capable of carrying proppant due to its higher viscosity
than liquid nitrogen, the fractures were able to stay open. Accordingly, all the wells for which he
published results experienced increased production rates (King 1983). Unfortunately, post
fracturing production data over an extended period of time were not available so the data are
inconclusive in determining the long-term production characteristics.

Even though successful results were produced in King’s research, his research only
included theory and fieldwork; no controlled laboratory experiments were performed. Without a
control group to compare the results to it is impossible to determine if the increased well production
data is from thermal stresses creating fractures or from fluid pressure creating the fractures.

In a separate cryogenic fracturing study, Grundmann (1998) treated a Devonian shale well
with cryogenic nitrogen and observed an initial production rate 8% higher than the rate in a nearby
offset well that had undergone traditional fracturing with nitrogen gas. Unfortunately, subsequent
production information was unavailable because the well had to be shut in for logistical reasons.
Although the increased initial production rate in this research suggests the efficacy of cryogenic
fracturing, there could be a number of reasons why an offset well in a shale formation might
produce differently including anisotropic stress conditions and heterogeneous reservoir conditions
over short distances. Similar to King’s research, nothing in the Grundmann study points to
cryogenic fracturing, as opposed to hydraulic fracturing, as the stimulation mechanism.

To further advance the study of cryogen fluids on hydrocarbon producing formations,
McDaniel (1997) conducted simple laboratory studies where coal samples were immersed in
cryogenic nitrogen. The coal samples experienced significant shrinkage and fracturing into
smaller cubic units, with the creation of microfractures orthogonal to the surface exposed to the
cold fluid, which is shown below in Figure 1.1.1
The researchers found that repeated exposure cycles to the cryogen caused the coal to break into smaller and smaller pieces, or become rubblized. After three cycles of exposing the coal to liquid nitrogen and allowing the coal to warm back to ambient temperatures, the coal was reduced to grain size particles. If the creation of fractures due to thermal stresses can occur in coal bed formations, it has the potential to occur in other types of rock as well. McDaniel (1997) also conducted field re-fracturing experiments with cryogenic nitrogen, and published before and after production rate data for five wells. The results were mixed: three wells showed increased production, one well showed equivalent production, and one well showed decreased production. From the three wells that showed an increase in production, two of them had long-term increases in production.

The prior research suggests some promising benefits associated with cryogenic fracturing fluid, but does not identify the specific fracturing mechanisms at work in downhole conditions. There are also many obstacles to overcome in the field such as equipment rated for cryogenic temperatures and figuring out how to transport proppant in the cryogen to prop open the newly formed fractures. Cryogenic nitrogen and carbon dioxide lack significant viscosity (Rudenko 1968; Fenghour 1998) and; therefore, may inadequately carry proppant if viscosity serves as the
primary transport mechanism. Gupta (1998) concluded that cryogenic carbon dioxide’s low viscosity could not enable adequate proppant transport; however, it is possible to create a high Reynolds number by increasing the fluid velocity. The accompanying turbulence permits good transport of the proppant, at least through the wellbore to the perforations, if not through the fracture as well (Gupta 1988). There is also some evidence that points to shale formations treated with multiple cryogenic treatments may form self-propping fractures. For instance, McDaniel’s research demonstrated coal rubblization in laboratory experiments that can be a self-propping mechanism. If rock undergoes sufficient breakage into small pieces at the fracture/rock interface, the formation’s inability to close on this rubblized rock may enable the fracture to stay open against in-situ compressive stresses after cessation of treatment pressure. Although McDaniel only performed this experiment on coal, it can be deduced that if the same rubblization process occurs in shale formations, the need for proppants to prop open fractures may be significantly reduced.

If neither traditional proppants nor a self-propping mechanism can effectively keep the created fracture open, ultra-light weight proppants (ULWPs) may fill the gap. ULWPs are manufactured proppants that consists of a chemically hardened walnut hull core with multiple layers of epoxy resin coating acting as the outer shell (Kendrick 1995). Kendrick’s research observed improved post-stimulation production in Devonian Shale wells treated with hydraulic fracturing methods using nitrogen foam fluid and ULWPs. The research shows that the majority of the wells with the ULWPs performed as good if not better than wells with traditional proppant. If the low viscosity nitrogen foam could successfully transport ULWPs, cryogenic fluid may be able to do so as well. Although these proppants will be expensive compared to traditional sand proppant, it may prove economic if the cryogenic stimulation is more effective than hydraulic fracturing in stimulating the initial production.

Like any new technology, there will be technical hurdles that must be overcome before progress can be made. Injecting pressurized liquid nitrogen downhole will call for a re-design of many current fracturing equipment components. All of the material that comes into contact with the cryogen must be able to withstand temperatures of nearly -200°C. Dealing with massive amounts of pressure at extremely cold conditions will also bring about safety issues. If this concept is implemented into fracturing wells, a systematic approach will need to be implemented to protect the workers from the dangers of pressurized cryogenic fluids.
1.2 Objectives

The primary objective of this study is to build a laboratory cryogenic fracturing system and study the fracturing capabilities of liquid nitrogen. As previously stated, some research has been conducted on using liquefied gases as a mechanism to fracture reservoir rocks, but those studies were not comprehensive and had no comparable standards. This study will take a methodical approach to analyze the efficacy of cryogenic fracturing by monitoring pressure and temperature while injecting liquid nitrogen into unconfined concrete samples. The purpose of this study is to better understand the characteristics of cryogenic fracturing before implementing a field study.
CHAPTER 2 CONCRETE DESIGN

In this study, it is vital that all samples tested are similar to each other. To ensure this, a specific type of concrete was used for all liquid nitrogen injection tests. The mechanical properties of concrete vary greatly based on numerous variables such as material ratios, curing time, and the curing environment. This chapter will review the design and implementation of the concrete making procedure by giving verbatim procedures as to how the concrete was made from raw materials to a finished product.

2.1 Design and Making of the Specimen

First and foremost is the design of the actual testing material that was used in each of the ensuing experiments. Ideally, this experiment should be performed on in-situ shale samples or tight sandstone samples. However, due to availability, costs, and the general anisotropic nature of formations, all liquid nitrogen injection experiments were performed on concrete samples. The concrete samples were made using 8” x 8” x 8” plastic molds. The molds guarantee that each sample is precisely the same size. Each sample was made with 12 kg of dried sand, 4.8 kg of commercial grade Portland Cement Quikrete Type I/II #1124 and 2.64 kg of tap water. Table 2.1.1 outlines the sieve analysis for the sand used to construct the concrete blocks. The sand has a pH of 7.87.

Table 2.1.1 Results of sieve analysis for sand used in making concrete blocks

<table>
<thead>
<tr>
<th>Sand Mesh Size</th>
<th>Percent Passing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve</td>
<td></td>
</tr>
<tr>
<td>No. 4</td>
<td>100.0%</td>
</tr>
<tr>
<td>No. 8</td>
<td>99.4%</td>
</tr>
<tr>
<td>No. 16</td>
<td>86.8%</td>
</tr>
<tr>
<td>No. 30</td>
<td>58.4%</td>
</tr>
<tr>
<td>No. 50</td>
<td>27.6%</td>
</tr>
<tr>
<td>No. 100</td>
<td>6.9%</td>
</tr>
<tr>
<td>No. 200</td>
<td>2.4%</td>
</tr>
</tbody>
</table>

The sand, water, and cement were then mixed for 10 minutes until a homogenous mixture was achieved. The mixture was then carefully placed into the plastic mold in a way that minimize air bubbles from forming. Once the mold was completely full of concrete, the sample was enclosed
in a plastic bag for 24 hours to allow the concrete to cure and to prevent water from evaporating out. After the initial 24 hours, the concrete sample was removed from the mold and placed underwater for 28 days to allow for full curing. The samples were cured for 28 days because this is the standard for working stress in concrete design; however, additional curing time does increase the strength of the concrete. Because of this additional strength when curing for more than 28 days, it is ideal for all samples to be tested at the same number of curing days for consistency; unfortunately, not all samples had the same number of curing days. The curing conditions and mixture ratios were all acquired from Walter Price’s study of Factors Influencing Concrete Strength (Price 1951). Based on Price’s work, the conditions chosen maximize the strength of the concrete. Since the purpose of this study is to translate what is learned from fracturing concrete with liquid nitrogen into field studies of actual reservoir rock, Table 2.1.2 displays the different nominal mechanical properties for typical concrete, sandstone, and shale samples.

Table 2.1.2 Typical mechanical properties for experimental specimens (Cha 2014)

<table>
<thead>
<tr>
<th>Rock Type Properties</th>
<th>Concrete</th>
<th>Sandstone</th>
<th>Shale</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (gm/cc)</td>
<td>2.24 – 2.4</td>
<td>2.2 – 2.8</td>
<td>2.4 – 2.8</td>
</tr>
<tr>
<td>Compressive strength (MPa)</td>
<td>20 – 40</td>
<td>20 – 170</td>
<td>5 – 100</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>2 – 5</td>
<td>4 – 25</td>
<td>2 – 10</td>
</tr>
<tr>
<td>Young’s Modulus (GPa)</td>
<td>14 – 41</td>
<td>1 – 20</td>
<td>1 – 70</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.20 – 0.21</td>
<td>0.21 – 0.38</td>
<td>0.20 – 0.40</td>
</tr>
<tr>
<td>Shear strength (MPa)</td>
<td>6 – 17</td>
<td>8 – 40</td>
<td>3 – 30</td>
</tr>
<tr>
<td>Specific heat capacity (kJ/kg K)</td>
<td>0.75</td>
<td>0.92</td>
<td>0.88 – 1.09</td>
</tr>
</tbody>
</table>

In each of these samples a borehole must be made to allow for injection stimulation treatments. In the first few samples the borehole was made by inserting a 1.05” diameter plastic...
tube that allowed for the concrete to cure with a pre-made borehole. After a few successes with this design the plastic mold began to become stuck in the concrete. Once this occurred all concrete samples were made without a borehole and a 1.05” diameter borehole was later drilled into the sample using a diamond imbedded coring device under wet cutting conditions after the sample had cured for 28 days. The borehole was drilled to a depth of approximately 6” into the specimen. Once cored, two thermocouples were inserted into the borehole, one freely-hanging in the wellbore and the other attached to the side of the borehole, to measure the temperature of the specimen once the experiment were to take place. The thermocouples used were made by Omega Engineering with part number TT-T-20-SLE. This part number means the thermocouples are dual insulated made with copper-constantan metals with a temperature range from -270°C - 400°C and an error of 0.5°C. As mentioned, there are two thermocouples inserted into the borehole, one freely hanging and one adhered to the surface by superglue. The purpose of this placement is twofold: if one thermocouple were to fail there would be an additional one to measure the temperature; also, the attached thermocouple reads the actual temperature of the wellbore wall whereas the hanging thermocouple measures the temperature of the fluid in the wellbore. The purpose of having an attached thermocouple is to differentiate the wall temperature from the fluid temperature, as the Leidenfrost Effect, which takes place in the film boiling regime, usually makes the wall temperature significantly different from the liquid nitrogen temperature. Such measurement tells whether the liquid nitrogen is adequately cooling the borehole wall to create the desired thermal gradient in the concrete sample.

Next, the casing must be inserted and attached to the borehole. The borehole and surface around the borehole were thoroughly scrubbed with a wire brush and cleaned with alcohol and compressed air. This removed any debris that might hinder a strong bonding between the casing and the specimen. Then, a 4.25” piece of stainless steel casing with and an OD of 1” and an ID of 0.8000” was inserted 2” into the wellbore and epoxied using industrial strength JB Weld part number 8280. After a few initial trials with this design it was noticed that in some instances the thermal shock from the liquid nitrogen would cause the epoxy to break from the stainless steel and the concrete because of the difference in the thermal expansion coefficients of the materials. The design is shown below in Figure 2.1.1.
This breakage was suppressed by drilling a 2” diameter hole with a depth of .25” around the borehole (the shaded volume in Figure 2.1.1). This volume allowed for additional epoxy to set and fix the tubing to the concrete. After this change in the design was made, there were no more issues with epoxy bonding.
2.2 Testing Parameters

To gather a better understanding of the performance of injecting liquid nitrogen in creating fractures, various testing conditions were used to gather a plethora of data that represents different reservoir environments. The “saturated” samples, specifically, simulate the situation that the formation to be fractured may be totally or partly saturated with water that may expands upon freezing. The “high temperature” tests, on the other hand, study the effect of sample temperature. Table 2.2.1 shows the various testing conditions that were assessed in this study.

Table 2.2.1 The various sampling conditions for concrete specimens

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid N₂</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

For each entry in the testing matrix, the samples were prepared following identical procedures. For the tests involving oven dried samples, the samples were placed in a Quincy Lab Oven (part # 21-350) and set to 65°C to dry. After 10 days of drying, the changes in weights from day to day measured by an Adams Scale (Part # CPWplus-75) with a readability of 0.05 pounds were insignificant; therefore, all of the oven dried samples were placed in the oven for 10 days before they were performed. The samples were then taken out of the oven and allowed to cool to a room temperature of 20°C before performing injections tests.

The saturated samples were prepared by completely submerging oven dried samples in a bin filled with tap water at the 20°C room temperature. They were submerged for three days to allow water to completely permeate the samples. After three days passed, the samples were taken out and tested. On average, the samples weighed approximately 1kg more saturated than when dry. Once the samples were fully saturated they were taken out of the water bin, sponged of any excess water on the surface, and tested immediately.

The dry high-temperature samples were prepared similar to the oven dried samples, except they were not allowed to cool back down to the room temperature. These samples, with a temperature of 65°C, were taken out of the oven and a pressure leak-off test was performed on...
them. Since the pressure leak-off tests takes time, the samples would cool down significantly. They were then placed in the oven overnight to allow the temperature of the sample to increase back to 65°C and tested the next day.

The final entry in the testing matrix is a combination of high temperature and water saturation. For these experiments the sample was prepared by placing it in a large plastic bin filled with water. The bin was then covered, to reduce evaporation, and placed in the oven at 65°C. It was left in the oven for 3 days to allow for full saturation and to allow adequate time for the sample temperature to reach the desired temperature.

Due to the nature of experimental work, the samples made following the same set of procedures are not strictly identical. Table 2.2.2 shows the measured values for porosity, permeability, and the tensile stress for some concrete specimens. The permeability was measured using a CMS-300, which measures the permeability with helium under an isotropic confining pressure of 1000 psi. The tensile strength was measured using a Brazilian test. Shown below in Table 2.2.3 are the various specifications for each concrete specimen that was tested. Overall, the sample variations, measured by weights, are relatively consistent.

By performing these controlled tests and repeating them on similar samples, the results can be compared across the board to reveal the effect of sample condition on their frackability. By revealing what conditions are ideal for using cryogenic nitrogen as a stimulation fluid, we expect that these observations are useful to guide future field studies.

<table>
<thead>
<tr>
<th>Porosity (%)</th>
<th>Permeability (µD)</th>
<th>Tensile Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.15 - 0.17</td>
<td>7 - 70</td>
<td>2.18 – 2.21</td>
</tr>
</tbody>
</table>
Table 2.2.3 Concrete sample specifications

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Experiment Type</th>
<th>Saturated Weight (kg)</th>
<th>Dry Weight (kg)</th>
<th>Borehole Creation</th>
<th>Curing Time (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Oven Dried Gas (_{N_2})</td>
<td>17.62</td>
<td>17.02</td>
<td>Mold</td>
<td>143</td>
</tr>
<tr>
<td>2</td>
<td>Oven Dried Gas (_{N_2})</td>
<td>17.68</td>
<td>17.10</td>
<td>Mold</td>
<td>67</td>
</tr>
<tr>
<td>3</td>
<td>Oven Dried Gas (_{N_2})</td>
<td>17.58</td>
<td>16.98</td>
<td>Mold</td>
<td>96</td>
</tr>
<tr>
<td>4</td>
<td>Oven Dried LN(_2)</td>
<td>17.88</td>
<td>17.16</td>
<td>Mold</td>
<td>148</td>
</tr>
<tr>
<td>5</td>
<td>Oven Dried LN(_2)</td>
<td>17.64</td>
<td>16.78</td>
<td>Mold</td>
<td>75</td>
</tr>
<tr>
<td>6</td>
<td>Oven Dried LN(_2)</td>
<td>17.86</td>
<td>16.96</td>
<td>Mold</td>
<td>96</td>
</tr>
<tr>
<td>7</td>
<td>Water Saturated LN(_2)</td>
<td>17.92</td>
<td></td>
<td>Drilled</td>
<td>52</td>
</tr>
<tr>
<td>8</td>
<td>Water Saturated LN(_2)</td>
<td>17.84</td>
<td></td>
<td>Drilled</td>
<td>45</td>
</tr>
<tr>
<td>9</td>
<td>Water Saturated LN(_2)</td>
<td>17.86</td>
<td></td>
<td>Drilled</td>
<td>45</td>
</tr>
<tr>
<td>10</td>
<td>Dried High Temp LN(_2)</td>
<td>17.78</td>
<td>17.04</td>
<td>Drilled</td>
<td>55</td>
</tr>
<tr>
<td>11</td>
<td>Dried High Temp LN(_2)</td>
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<td>17.10</td>
<td>Drilled</td>
<td>115</td>
</tr>
<tr>
<td>12</td>
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<td>16.98</td>
<td>Drilled</td>
<td>118</td>
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<tr>
<td>13</td>
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<td>17.92</td>
<td></td>
<td>Drilled</td>
<td>104</td>
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<tr>
<td>14</td>
<td>Saturated &amp; High Temp</td>
<td>17.84</td>
<td></td>
<td>Drilled</td>
<td>66</td>
</tr>
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<td>15</td>
<td>Saturated &amp; High Temp</td>
<td>17.80</td>
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<td>Drilled</td>
<td>67</td>
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</tbody>
</table>
CHAPTER 3 EQUIPMENT AND OPERATION PROCEDURES

In this experiment, there are multiple types of fracturing mechanisms to study. The first type is fracturing due to thermal shock. This first mechanism explores the idea of using liquid nitrogen to generate thermal stress. If strong enough, this thermal stress will create fractures on the surface of the borehole orthogonal to the contact plane. This was previously illustrated in Figure 1.1.1. The second fracturing mechanism explored in this experiment is using the pressure differential built up inside the borehole from both the evaporation of liquid nitrogen and the pressurization of nitrogen gas. An expansion ratio is the amount of volume of a given substance in liquid form compared of that to its gaseous counterpart under atmospheric pressure and assumes adiabatic conditions. Liquid nitrogen has an expansion ratio of nearly 1:700 (Houghton 2008). This large expansion ratio is realized and taken advantage of to help pressurize the borehole of the specimen.

Combining pressurization with thermal shock is beneficial for a number of reasons. First, pressurization helps to reduce the effects of a phenomena known as the Leidenfrost effect. The Leidenfrost effect occurs when a fluid comes into contact with a surface whose temperature is much greater than that of its boiling point. When this occurs, a vapor cushion is formed between the liquid and the surface practically eliminating direct contact between the liquid and the surface. This vapor cushion acts as an insulating layer that greatly hinders the heat transfer from the liquid nitrogen to the specimen, thus reducing the stresses induced from the thermal shock. By increasing the pressure within the borehole the Leidenfrost phenomena can be greatly reduced if not entirely eliminated. This is advantageous in creating a thermal fracture because the greater the temperature difference between the surface of the specimen and its interior the greater the induced stresses will be. If this temperature gradient occurs at a near instantaneous time then the stress generated will be maximized, thus increasing the likelihood of tensile failure and fracture generation.

Another advantage of pressurizing the borehole is to force the liquid nitrogen to penetrate further into the microfractures that are formed by the initial thermal shock. The pressurization will effectively act as a driving force that will further disseminate the liquid nitrogen throughout the specimen. By forcing the cryogen through the microfractures generated in the specimen more surface area will be exposed creating a more extensive fracture network. The pressurization will also add to the stress acting on the specimen. The stress from the pressurization works in
conjunction with the thermal stress incurred on the specimen, enhancing the effectiveness of cryogenic fracturing.

To adequately study the process of liquid nitrogen fracturing, there must be a methodical setup so each experiment can be compared to the others. In this study, much work was spent on iterating the design of the setup for continuous improvements. Here, we only present the “final” design used to perform the tests listed in Table 2.2.1.

3.1 Pressure and Temperature Data Acquisition

In this experiment, we employed both temperature and pressure signals to study the fracturing process. Shown below in Figure 3.1.1 is the pressure and temperature data acquisition equipment used in this experiment. The machine on top in Figure 3.1.1 is a Keithley 2701 data acquisition system. The thermocouples and pressure transducers send voltages to this device that reads and translates the signal into temperatures and pressures at an adjustable frequency. The pressure transducers are made by Omega, have a range from 0-3000 psi, and have an accuracy within 0.25% (Part # PX309-3KG5V). For this study, the sampling frequency was four Hertz. This device transmits the data collected to a computer via an Ethernet port. Then, using an add-in called Excel Link, these data are displayed in real-time in excel. The system shown at the bottom of Figure 3.1.1 is an Agilent 0-30 volt 3 amp DC power source (part # U8001A) used to power pressure transducers and flowmeters that are installed along the flow lines. Before any experiment took place, both the data acquisition system and the power supply were turned on and configured according to the requirements for each experiment. Figure 3.1.2 shows the electrical system schematic used for this experiment. This figure is to be used in conjunction with Figures 3.2.1 and 3.3.1 for to fully comprehend the experimental setup. Also it should be noted that the electrical schematic in Figure 3.1.2 shows the relative location for each of the components. This schematic changed slightly between experiments but generally speaking it is a representative of the electrical components.
Figure 3.1.1 Data logger (top) and dc power supply (bottom).
3.2 Design of Specimen Wellhead

An important design feature in this experiment is the specimen wellhead through which the cryogen is injected into the borehole. It must be properly designed and vented so when the liquid nitrogen is vaporized, the gas nitrogen does not get trapped as a gas “cushion” at the bottom of the borehole. With the gas “cushion”, the borehole surface will be insulated from the liquid cryogen and the thermal shock will not be effective to generate sufficient stress to fracture the specimen. Shown below in Figure 3.2.1 is the design of the wellhead injection point. By injecting the liquid nitrogen through the tubing and allowing the gas to escape through the area between the
borehole and the tubing, an insulating nitrogen gas bubble will not impede the thermal shock created by the liquid nitrogen.

Figure 3.2.1 Schematic of wellhead for unconfined liquid nitrogen injection.

3.3 Unconfined High Pressure Cryogenic Injection

Shown below in Figure 3.3.1 is an expanded schematic of the wellhead that shows how the liquid nitrogen is pressurized and injected into the wellbore. This section will detail the experimental setup that directs the flow of liquid nitrogen and controls pressurization and injection into the sample. As to the operation procedures, we will also make reference to Figure 3.3.1 when explaining the process. It should also be noted that all tubing lines that flows liquid nitrogen are thoroughly insulated with rubber, foam, or fiberglass.

The next part of this design is explaining how the cryogenic fluid was pressurized and injected into the specimen. To allow for the pressurization of liquid nitrogen without a cryogenic pump, a stainless steel cryogenic accumulator was used to store liquid nitrogen. The accumulator was made by using 2” Swagelok stainless steel tubing with a series of reducers on both ends to allow for the accumulator to attach to the rest of the system. The accumulator has a pressure rating
of 1000 psi at liquid nitrogen levels, but as a factor of safety, the pressure was limited to 500 psi. The accumulator, along with any lines or tubing that flow liquid nitrogen, was heavily insulated with rubber and Styrofoam to minimize evaporation. The cryogen fills the accumulator by the pressure differential built by the vaporization of liquid nitrogen and expansion inside the liquid nitrogen vessel. Once an adequate amount of cryogen is stored in the accumulator, the valve that connects the accumulator to the storage tank is shut off. From this point, the upper relief valve located at the top of the accumulator is closed and a regulated nitrogen gas source, which creates a pressure buildup inside the accumulator, is opened. This gas nitrogen source pressurizes the cryogen accumulator to 450 psi. It is important to note that all pressure safety relief valves were set at 500 psi so the system does not get over pressured. Once the cryogen is pressurized, the cryogenic valve that connects the specimen wellbore to the accumulator is opened and the specimen is exposed to pressurized cryogenic fluid. An important aspect of this design is the orifice opening of the exhaust valve, labeled as Outlet LN\textsubscript{2} Valve in Figure 3.2.1 and Figure 3.3.1. The liquid nitrogen must be actively flowing through the lines and the wellbore to allow the borehole surface to research liquid nitrogen temperatures thus the valve must be kept open. However, if the valve is opened too much the liquid nitrogen will leak out too quickly and will not build up pressure or effectively chill the wellbore. If it is closed too much the nitrogen gas will form a nearly stationary vapor cushion that will prevent the liquid nitrogen from making contact with the borehole. This aspect will be further elaborated on in the results and discussion section. Since this experiment deals with highly pressured liquid nitrogen, it is very dangerous and the appropriate PPE (Personal Protection Equipment) must be worn at all times during the experiment. This includes wearing a long sleeve shirt, long pants, steel-toed boots, goggles, and cryogenic gloves when opening and closing the cryogenic valves. In addition, polycarbonate sheets were set up between the experiment and the operator (me). This offers an additional protection for the operator from any shrapnel that may occur from the specimen fracturing.

The detailed operation procedures are as follows. First the valves were situated where cryogen would only flow into the liquid nitrogen accumulator by opening cryogenic valve 1, closing cryogenic valve 2, and opening the LN\textsubscript{2} accumulator pressure relief valve. It is very important that the pressure relief valve is opened for safety reasons. If not the liquid nitrogen accumulating in the vessel would vaporize to its gas phase, expand, and create a confined pressure
that is very dangerous; in this study, the pressure relief safety valve was set to 500 psi which is well within the LN$_2$ accumulator’s pressure tolerance. If the pressure were to accidentally build above 500 psi the pressure relief safety valve would open and regulate the pressure to 500 psi. Once the valves were set, the liquid nitrogen source valve was opened allowing the liquid nitrogen to flow into the accumulator. When performing the first cryogenic run of the day, the 1-liter LN$_2$ accumulator must be cooled down so the liquid nitrogen does not immediately boil off.

It takes approximately 25 minutes to fill the empty LN$_2$ accumulator with liquid nitrogen starting from the room temperature; the subsequent fillings after the lines have cooled take approximately 4 minutes. While the accumulator was being filled with liquid nitrogen the nitrogen gas accumulator was prepared to be pressurized by closing both the gas N$_2$ valve connected to the LN$_2$ accumulator and the pressure relief valve that relieves the pressure into the atmosphere. Once the valves were in the correct positions, and while the LN$_2$ accumulator was being filled, the nitrogen gas accumulator was pressurized to 450 psi. Once the nitrogen gas accumulator reached 450 psi, the source was shut off until the liquid nitrogen was ready to be injected. While the cryogenic nitrogen was filling the accumulator, the liquid nitrogen outlet from the sample is adjusted to where it is only open between 5%-20%. This outlet will ultimately determine the pressure and flowrate of the liquid nitrogen; this concept will be elaborated on further in the results and discussion section.

Once the thermocouple located at the top of the liquid nitrogen accumulator read temperatures near cryogenic levels and the LN$_2$ accumulator pressure relief valve’s exhaust is a mixture of liquid nitrogen and gas, the accumulator was completely filled and the system was ready for a test. At this point, cryogenic valve 2 was opened, the LN$_2$ accumulator pressure relief valve was closed, and cryogenic valve 1 was closed in that order. This setup would then allow the cryogenic fluid to flow freely into the sample wellbore. Immediately after the cryogen flowed into the sample, the gas nitrogen valve 2 was opened while the gas nitrogen source was simultaneously turned on. This action allows the 450 psi nitrogen gas to flow into the cryogenic accumulator, pressurize the remaining liquid nitrogen, and force it into the specimen borehole.
Figure 3.3.1 Schematic of injecting pressurized liquid nitrogen into an unconfined specimen.
Depending on the results from each cryogenic treatment, more cryogenic treatments may be repeated on each specimen. If additional treatments were performed, they were done exactly as described above in the initial treatment.

Once the specimen was fractured, or the number of desired cryogenic treatments were performed, the gas and liquid nitrogen sources were shut off and all valves were opened to allow for the setup to warm up to room temperature and to allow the lines to clear of any water that might have condensed.

3.4 Pressure Decay Test

For each experiment in which concrete samples were not fractured during liquid nitrogen treatment, we performed a before-stimulation and an after-stimulation pressure leak off test. These tests, by providing the rate of pressure decay, are indicative of the effective air permeability. This pressure leak off test was performed by pressurizing the borehole to 175 psi, shutting in the wellbore, and allowing the pressure to slowly leak off. In some instances the specimen fractured during the liquid nitrogen stimulation. When this occurred there was no after-stimulation pressure leak off test because the broken specimen could not hold pressure.
CHAPTER 4 EXPERIMENTAL RESULTS

This section presents all the data gathered in the stimulation experiments. These data include the pressure and temperature transients as well as pictures showing the fracture characteristics of each sample. Some experiments may have more or less data than others due to either an experimental component failing during testing or an unexpected change in testing condition. We will discuss and compare cases with similar testing procedures.

4.1 Nitrogen Gas Stimulating Dry Samples

The following experiments were all performed on dry unconfined concrete samples. The injecting fluid was nitrogen gas. This was an attempt to determine a baseline to compare subsequent tests to. As seen below in Figure 4.1.1 the breakage points for the three samples were 909 psi, 566 psi, and 491 psi, respectively. Sample 1 has a much higher breakdown pressure than Samples 2 and 3 because it had a much longer curing time.

Figure 4.1.2 shows the pressure decay tests for Samples 2 and 3. The experiment on Sample 1 was performed before deciding to do pressure decay tests on all samples therefore it did not produce pressure decay data. It can be observed that, between Samples 2 and 3, that 2 has a faster decay and therefore higher permeability. These two tests were performed with an initial pressure of 175 psig at a room temperature of 20°C.

Figures 4.1.3-4.1.5 shows the fracture profile of Samples 1, 2, and 3. For each sample tested the fracture profile will be presented where each specimen face and the internal faces will be viewable. The purpose of this is to draw parallels between different specimens and fracturing conditions and see if there is a correlation by the way the sample fractures. It can be observed from these three figures that Sample 1 and 2 developed fractures that are parallel to the axis of the borehole, whereas Sample 3 developed a fracture that is almost perpendicular. For unconfined samples, direction of the fracture can be arbitrary, depending on the locations of weaknesses.
Figure 4.1.1 Breakage point for dry Samples 1-3 Using nitrogen gas.
Figure 4.1.2 Pre-fracture pressure decay test for Samples 2 and 3.
Figure 4.1.3 Fracture Profile of Sample 1.
Figure 4.1.4 Fracture Profile of Sample 2.
Figure 4.1.5 Fracture Profile of Sample 3.
4.2 Liquid Nitrogen Stimulating Dry Samples

The following experiments were all performed on oven-dried concrete specimens. The injected fluid was liquid nitrogen. Samples 4, 5 and 6 were dry concrete specimens. While Samples 5 and 6 show sound data, Sample 4 was performed while the testing procedure was still being fine-tuned; therefore, the data for Sample 4 are rather sparse, inconclusive, and different from the other two samples. The data acquisition system also failed during the testing of Sample 4, so some of the data gathered were lost. In addition, Sample 4 was treated with liquid nitrogen at 15 psi for 30 minutes, allowed to warm back up to room temperature, and then fractured with gas nitrogen; Samples 5 and 6, on the other hand, were treated with liquid nitrogen pressurized to 305 psi and 320 psi respectively. Samples 5 and 6 were also not fractured with liquid nitrogen, but gas nitrogen after the samples warmed back to the room temperature. Shown below in Figure 4.2.1 and 4.2.2 are the results from injecting liquid nitrogen into Sample 5 and 6 respectively. For Sample 5, at \( t = 2290 \) (all time units are in second) the valve from the liquid nitrogen accumulator into the borehole was opened, allowing for liquid nitrogen to flow into the wellbore. At \( t = 2550 \) the liquid nitrogen in the accumulator was depleted. As a result, the temperature in the borehole began to rise back to the room temperature. There are two distinct decreases in temperature during this experiment. The first occurred at \( t = 2290 \), while analyzing the data in real-time, it was determined that the cryogenic outlet valve was not opened enough. So the second temperature drop, at \( t = 2505 \), occurred because the outlet was opened more. There is a distinct pressure difference in the accumulator and the sample when the outlet was opened more, because the nitrogen gas has to pressurize both the nitrogen gas accumulator and the liquid nitrogen accumulator, both of which have relatively large volumes in comparison with the deliverability of the tubing that supplies the gas nitrogen. Another observation is the liquid nitrogen accumulator temperature. The liquid nitrogen supply was turned on at \( t = 285 \), leading to the decrease in temperature of the accumulator. The temperature decreased linearly until -70°C, when there was a noticeable change in the cooling rate of the accumulator. The temperature of the accumulator still decreased linearly but at a much slower rate. This trend was seen for all of the tests performed.
Figure 4.2.1 Pressure and temperature records during treatment of Sample 5.
Figure 4.2.2 Pressure and temperature records during treatment of Sample 6.

Compared to Sample 5, which was treated with liquid nitrogen only once, Sample 6 was treated twice, as indicated by the two separate major temperature drops in the borehole in Figure 4.2.2. The specific list of events in Figure 4.2.2 is as follows: liquid nitrogen began filling the accumulator at $t = 67$; the first treatment started at $t = 1917$; the first treatment completed and the accumulator started to refill at $t = 2117$; second treatment began at $t = 2528$; second treatment completed at $t = 2636$. As seen in this figure, the liquid nitrogen injecting pressure was not a constant, but varied with the temperature and pressure conditions in the borehole and the accumulator. This is a limitation of the design, as we do not have a liquid nitrogen pump, the maximum volume of liquid nitrogen that can be discharged from the accumulator per treatment is one liter, and it is difficult to have consistent pressure as the process was manually controlled by a valve.
Shown below in Figure 4.2.3 are the results of pre- and post-treatment pressure decay tests for Samples 5 and 6. It appears that Sample 5 had a significant increase in air permeability. It should also be noted that there was practically no change between pre- and post-treatment pressure decay data for Sample 6. Pressure decay tests should only be compared only between pre- and post-treatment on the same sample. It was noticed that not every sample formed a perfect seal. Depending on how much epoxy was used, air was audibly heard and physically felt leaking through the concrete around the epoxy near the borehole. At times, hissing sound indicating leak was also heard through the thermocouple lines. Table 4.2.1 summarizes the results from the dry concrete liquid nitrogen experiments.

In addition to the pre-stimulation and post-stimulation pressure decay rates, I wanted to show a phenomenon present in all of the pressure leak off tests. In the pressure decay tests I have shown in Figure 4.1.2 and 4.2.3, the temperature data were omitted and the test began exactly when the pressure in the sample is shut-in at 175 psi. Below in Figure 4.2.4 is a complete pre-fracture pressure leak off test on Sample 6 with temperature data. Notice how the temperature changed with the pressure. This is an observation of the Joule-Thomson effect which occurs when a gas expands under adiabatic conditions. As shown in this case, as pressure increases, temperature increases, and as the pressure is relieved, the temperature decreases. This was observed in all pressure decay tests. However, only this instance is shown here. It is an indication that in order to precisely match the pressure decay, we may need to use a non-isothermal reservoir simulator. Figures 4.2.5-4.2.7 shown on the following page are the fracture profiles of Samples 4, 5, and 6.

Table 4.2.1 Summary of dry concrete liquid nitrogen injection tests

<table>
<thead>
<tr>
<th>Sample</th>
<th>Breakdown Pressure (psi, Nitrogen Gas)</th>
<th>Injecting Pressure at Lowest Temperature (psi)</th>
<th>Minimum Temperature in Borehole (°C)</th>
<th>Number of Treatments</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>580</td>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>352</td>
<td>305</td>
<td>-176</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>822</td>
<td>320</td>
<td>-182</td>
<td>2</td>
</tr>
</tbody>
</table>
Figure 4.2.3 Pressure decay tests for dry concrete with liquid nitrogen treatments.
Figure 4.2.4 Observation of Joule-Thomson Cooling Effect during a pressure decay test.
Figure 4.2.5 Fracture profile of Sample 4
Figure 4.2.6 Fracture profile of Sample 5
Figure 4.2.7 Fracture profile of Sample 6
4.3 Liquid Nitrogen Stimulating Saturated Samples

The following experiments were all performed on fully saturated concrete samples. As detailed previously, the samples were all submerged in water for an ample amount of time until fully water saturated. Figures 4.3.1-4.3.3 show the liquid nitrogen injection results for Samples 7, 8, and 9.

Sample 7 Saturated Concrete LN2 Injection

![Graph showing pressure and temperature records during treatment of Sample 7.](image)

Figure 4.3.1 Pressure and temperature records during treatment of Sample 7.

Sample 7 was exposed to liquid nitrogen twice and was fractured during the second stimulation. The specific list of events in Figure 4.3.1 is as follows: liquid nitrogen began filling the accumulator at $t = 55$; the first treatment started at $t = 1679$; the first treatment completed and the accumulator started to refill at $t = 1849$; the second treatment began at $t = 2309$; second treatment completed at $t = 2321$ when the specimen fractured. It was also during this experiment that an important feature was noticed about the experiment. The orifice size of the outlet plays a crucial role in controlling the injecting pressure and flowrate of the liquid nitrogen. It was noticed
that if the orifice was opened too much, with the limited cryogenic supply used in these experiments, the liquid nitrogen would quickly escape the system and dissipate into the atmosphere without pressurizing or cooling the wellbore sufficiently. On the contrary, if the outlet orifice were too small, not enough liquid nitrogen would flow into the wellbore to adequately cool the borehole to generate thermal fractures. To measure the opening of the outlet orifice for each experiment, I considered fully closing the valve being 0% open and fully opening the valve being 100% open. When fully opened the outlet ID is 0.1785 inches. I then precisely measured the percent of the orifice being opened for each cryogenic stimulation based on how far the valve was opened. For Experiment 7, the first cryogenic stimulation was 20% open and the second stimulation was 8% open. This was not realized until this experiment, so the outlet orifice size was not measured in previous experiments.

Sample 8 Saturated Concrete LN2 Injection

![Graph of temperature and pressure data for Sample 8](image)

Figure 4.3.2 Pressure and temperature records during treatment of Sample 8.

Sample 8 experienced three thermal shocks and was fractured during the third test. The specific list of events in Figure 4.3.2 is as follows: liquid nitrogen began filling the accumulator at
t = 100; the first treatment started at t = 1679 and outlet was opened to 18%; the first treatment completed and the accumulator started to refill at t = 1783; the second treatment began at t = 2094 with the outlet opened to 18%; second treatment completed at t = 2201; the third treatment began at 2496 with the outlet opened to 8%; the third treatment ended at 2502 when the specimen broke.

Figure 4.3.3 Pressure and temperature records during treatment of Sample 9.

As shown above in Figure 4.3.3, Sample 9 was fractured with only one stimulation treatment. The specific list of events in Figure 4.3.2 is as follows: liquid nitrogen began filling the accumulator at t = 53; the first treatment started at t = 1753 and outlet was opened to 5%; the first treatment completed at t = 1807 when the sample fractured. Notice that at this opening level, the borehole was not sufficiently cooled as the temperature did not reach the cryogenic levels.

Figure 4.3.4 presents the pre-stimulation pressure decay tests for Samples 7, 8, and 9. There does not seem to be a correlation between the permeability of the sample and the number of stimulations needed to fracture them.
Figure 4.3.4 Shows the various pressure decay tests for Samples 7, 8, and 9.

Table 4.3.1 summarizes the results of stimulating saturated concrete samples with liquid nitrogen. Compared with Samples 5 and 6, liquid nitrogen directly fractured the samples. This observation, though preliminary, indicates that water saturation helps to reduce the breakdown pressure during cryogenic treatment. This may occur because as the water freezes in the sample it will expand. This expansion introduces an additional stress on the surface of the rock face. Figure 4.3.5-4.3.7 show the fracture profiles for the saturated concrete specimens fractured using liquid nitrogen.
Table 4.3.1 Summarization of results for stimulation of saturated specimen

<table>
<thead>
<tr>
<th>Sample</th>
<th>Breakdown Pressure (psi)</th>
<th>Temperature at Breakdown Point (°C)</th>
<th>Δ Temperature At Breakdown Point (°C)</th>
<th>Number of Treatments</th>
</tr>
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<tbody>
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<td>7</td>
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<td>90</td>
<td>2</td>
</tr>
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<tr>
<td>9</td>
<td>416</td>
<td>-150</td>
<td>168</td>
<td>1</td>
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</table>

Figure 4.3.5 Fracture profile of Sample 7.
Figure 4.3.6 Fracture profile of Sample 8.
Figure 4.3.7 Fracture profile of Sample 9.
4.4 Liquid Nitrogen Stimulating Dry Heated Samples

The following experiments were all performed on dry heated concrete samples. As previously stated, the samples were dried and heated to 65°C for testing. The initial thermocouple readings in these experiments are a little less than 65°C because of the time it took to take the sample out of the oven and prepare it for testing. Figures 4.4.1-4.4.3 shown below are the stimulation results for Samples 10, 11, and 12.

![Sample 10 Dry Heated Concrete LN2 Injection](image)

Figure 4.4.1 Pressure and temperature records during treatment of Sample 10.

As shown above in Figure 4.4.1, there were a total of two treatments performed on this sample. The specific list of events in Figure 4.4.1 is as follows: liquid nitrogen began filling the accumulator at \( t = 88 \); the first treatment started at \( t = 1772 \) and outlet was opened to 12%; the first treatment completed and the accumulator started to refill at \( t = 1886 \); the second treatment began at \( t = 2188 \) with the outlet opened to 8%; the second treatment completed at \( t = 2204 \) when the
specimen fractured. As the thermocouple attached to the borehole failed for this experiment, only the temperature data from the hanging thermocouple are displayed.

Figure 4.4.2 Pressure and temperature records during treatment of Sample 11.

For Sample 11, three separate stimulation treatments were conducted before the sample was fractured. The specific list of events in Figure 4.4.2 is as follows: liquid nitrogen began filling the accumulator at t = 18; the first treatment started at t = 1660 and outlet was opened to 12%; the first treatment completed and the accumulator started to refill at t = 1730; the second treatment began at t = 2056 with the outlet opened to 12%; the second treatment completed at t = 2129; the third treatment began at 2373 with the outlet opened to 8%; the third treatment ended at 2388 when the specimen fractured. The fracture profile for Samples 10, 11, and 12 are shown below in Figures 4.4.4-4.4.6. The pressure decay test is shown in Figure 4.4.7.
Figure 4.4.3 Pressure and temperature records during treatment of Sample 12

For Sample 12 shown above in Figure 4.4.3 there was only one stimulation treatment. The specific list of events in Figure 4.4.3 is as follows: liquid nitrogen began filling the accumulator at $t = 115$; the first treatment started at $t = 1748$ and outlet was opened to 8%; the first treatment completed at $t = 1788$ when the sample fractured. Table 4.4.1 displays the breakdown data for Samples 10, 11, and 12.

Table 4.4.1 Summarization of results for dry heated concrete samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Breakdown Pressure (psi)</th>
<th>Temperature at Breakdown Point (°C)</th>
<th>$\Delta$ Temperature At Breakdown Point (°C)</th>
<th>Number of Treatments</th>
</tr>
</thead>
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<tr>
<td>10</td>
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<td>56</td>
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<tr>
<td>11</td>
<td>408</td>
<td>-150</td>
<td>196</td>
<td>3</td>
</tr>
<tr>
<td>12</td>
<td>416</td>
<td>-37</td>
<td>97</td>
<td>1</td>
</tr>
</tbody>
</table>
Figure 4.4.4 Fracture profile of Sample 10.
Figure 4.4.5 Fracture profile of Sample 11.
Figure 4.4.6 Fracture profile of Sample 12.
Figure 4.4.7 Pressure decay tests for Samples 10, 11, and 12.

Until Sample 12, most pressure leak off tests lasted about 1,500 – 2,500 seconds. Sample 12, as seen above, lasted for nearly 13,000 seconds until the pressure fully leaked off. This sample was prepared following identical procedures. It is not clear why the decay for Sample 12 was so much longer than other samples, but I speculate that the seal, for whatever reason, was much better than previous samples. There also must have been no pressure leaking through the thermocouples, which was present in some of the previous samples.

4.5 Liquid Nitrogen Stimulating Saturated Heated Samples

The following experiments were all performed on saturated heated concrete samples. As previously stated, the samples were placed in a covered water bath and heated to 65°C for three days. Figure 4.5.1 - 4.5.3 shown below are the results from the cryogenic stimulation of Samples 13, 14, and 15.
For Sample 13, four separate stimulation treatments were conducted before the sample was fractured. The specific list of events in Figure 4.5.1 is as follows: liquid nitrogen began filling the accumulator at $t = 76$; the first treatment started at $t = 1774$ and outlet was opened to 5%; after noticing that the outlet was not opened enough, the outlet was adjusted to 20% at $t = 1854$ and then 100% at $t = 2190$; the first treatment completed and the accumulator started to refill at $t = 2380$; the second treatment began at $t = 2818$ with the outlet opened to 20%; the second treatment completed at $t = 2877$; the third treatment began at $t = 3195$ with the outlet opened to 15%; the third treatment ended at $t = 3276$; the fourth treatment began at $t = 3694$ with outlet opened to 8%; the fourth treatment ended at $t = 3709$ when the sample fractured. During the first treatment it appears that the water from the sample froze in the tubing and blocked the nitrogen gas from escaping. Even after opening up the outlet to 20% the line still appeared to be clogged. It was not fully unclogged until the outlet was fully open.

Figure 4.5.1 Pressure and temperature records during treatment of Sample 13.
For Sample 14, two separate stimulation treatments were conducted before the sample was fractured. And again, it appears that water froze in the tubing blocking the nitrogen gas from escaping. The specific list of events in Figure 4.5.2 is as follows: liquid nitrogen began filling the accumulator at \( t = 176 \); the first treatment started at \( t = 1951 \) and outlet was opened to 8%; the outlet seemed to be clogged so the outlet was opened to 20% at \( t = 2311 \); the first treatment completed and the accumulator started to refill at \( t = 2403 \); the second treatment began at \( t = 2931 \) with the outlet opened to 8%; the second treatment completed at \( t = 2940 \) when the specimen fractured at 313 psi. When the tubing becomes clogged with ice notice how both the pressure and temperature deviate from what is expected. This should be taken into consideration when performing field tests. This will be further elaborated on in the discussion section.
For Sample 15 only one stimulation treatment was conducted before the sample was fractured. The specific list of events in Figure 4.5.3 is as follows: liquid nitrogen began filling the accumulator at $t = 60$; the first treatment started at $t = 1887$ and outlet was opened to $8\%$; the first treatment completed at $t = 1930$ when the specimen fractured. Table 4.5.1 summarizes the results for Samples 13, 14, and 15. Figure 4.5.4 shows the pressure decay test for Samples 13, 14, and 15. There is no significant difference among the three samples in the pressure decay data. Since all of the samples fractured during the treatments, there are no post-fracture data to compare to. Figures 4.5.5-4.5.7 show the fracture profiles for Samples 13, 14, and 15.
Table 4.5.1 Summarization of results for saturated heated concrete samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Breakdown Pressure (psi)</th>
<th>Temperature at Breakdown Point (°C)</th>
<th>Δ Temperature At Breakdown Point (°C)</th>
<th>Number of Treatments</th>
</tr>
</thead>
<tbody>
<tr>
<td>13</td>
<td>400</td>
<td>-50</td>
<td>104</td>
<td>4</td>
</tr>
<tr>
<td>14</td>
<td>313</td>
<td>-84</td>
<td>134</td>
<td>2</td>
</tr>
<tr>
<td>15</td>
<td>471</td>
<td>-80</td>
<td>127</td>
<td>1</td>
</tr>
</tbody>
</table>

Figure 4.5.4 Pressure decay tests for Samples 13, 14, and 15.
Figure 4.5.5 Fracture profile of Sample 13.
Figure 4.5.6 Fracture profile of Sample 14.
Figure 4.5.7 Fracture profile of Sample 15.
CHAPTER 5 DISCUSSION

This chapter reviews the results from this study and discusses what was learned and the interesting phenomena observed.

5.1 Comparison of Results

When comparing results from experiments, there are numerous factors to look at that may have influenced the pressure at which the samples fractured. Table 5.1.1 summarizes these factors that I deem critical in influencing the pressure breakage point. These factors are the temperature at which the sample fractures, the differential temperature \( \Delta T \) between the starting temperature and that at the actual breakage point, the number of cryogenic treatments performed on the sample, and the amount of time allotted for the samples to cure. One critical factor that is not presented in this table is the size of the outlet orifice. Since this variable changes for each individual treatment it could not be summarized in a simple table; however, it will be discussed in detail later in this chapter.

Table 5.1.1 Summary of results for each experiment

<table>
<thead>
<tr>
<th>Sample</th>
<th>Breakdown Pressure (psi)</th>
<th>Temperature at Breakdown Point (°C)</th>
<th>( \Delta T ) At Breakdown Point (°C)</th>
<th>Number of Treatments</th>
<th>Curing Time (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>909</td>
<td>19</td>
<td>0</td>
<td>0</td>
<td>143</td>
</tr>
<tr>
<td>2</td>
<td>566</td>
<td>20</td>
<td>0</td>
<td>0</td>
<td>67</td>
</tr>
<tr>
<td>3</td>
<td>491</td>
<td>20</td>
<td>0</td>
<td>0</td>
<td>96</td>
</tr>
<tr>
<td>4</td>
<td>580</td>
<td></td>
<td></td>
<td>1</td>
<td>148</td>
</tr>
<tr>
<td>5</td>
<td>352</td>
<td></td>
<td></td>
<td>1</td>
<td>75</td>
</tr>
<tr>
<td>6</td>
<td>822</td>
<td></td>
<td></td>
<td>2</td>
<td>96</td>
</tr>
<tr>
<td>7</td>
<td>308</td>
<td>-73</td>
<td>90</td>
<td>2</td>
<td>52</td>
</tr>
<tr>
<td>8</td>
<td>224</td>
<td>-38</td>
<td>56</td>
<td>3</td>
<td>45</td>
</tr>
<tr>
<td>9</td>
<td>416</td>
<td>-150</td>
<td>168</td>
<td>1</td>
<td>45</td>
</tr>
<tr>
<td>10</td>
<td>484</td>
<td>-2</td>
<td>56</td>
<td>2</td>
<td>55</td>
</tr>
<tr>
<td>11</td>
<td>408</td>
<td>-150</td>
<td>196</td>
<td>3</td>
<td>115</td>
</tr>
<tr>
<td>12</td>
<td>416</td>
<td>-37</td>
<td>97</td>
<td>1</td>
<td>118</td>
</tr>
<tr>
<td>13</td>
<td>400</td>
<td>-50</td>
<td>104</td>
<td>4</td>
<td>104</td>
</tr>
<tr>
<td>14</td>
<td>313</td>
<td>-84</td>
<td>134</td>
<td>2</td>
<td>66</td>
</tr>
<tr>
<td>15</td>
<td>471</td>
<td>-80</td>
<td>127</td>
<td>1</td>
<td>67</td>
</tr>
</tbody>
</table>
As a reminder, the first 3 samples were fractured using nitrogen gas on dried concrete specimens at 20°C, samples 4-6 were dried concrete specimens treated with liquid nitrogen and fractured with nitrogen gas at 20°C, samples 7-9 were saturated concrete samples at 20°C and fractured with liquid nitrogen, samples 10-12 were dry concrete specimens heated to 65°C and fractured with liquid nitrogen, and samples 13-15 were saturated specimens heated to 65°C and fractured with liquid nitrogen. Samples 4-6 temperature data were intentionally left off of this table because, unlike the other samples, these samples were treated with liquid nitrogen but then fractured with gas nitrogen after their temperatures returned to the room temperature.

Based on the data, it can be deduced that the strength of the concrete increases as the curing time increases. This is evident in the positive correlation between the curing time and the breakage pressure, as shown in Figure 5.1.1. Despite this correlation, there are other factors that may have influenced each specimen’s testing condition; these factors are presented in Table 5.1.1.

The breakage pressure and the number of treatments each sample was subjected to also seems to have a correlation. Figure 5.1.2 shows the breakage pressure as a function of treatments. Although not strongly correlated, Figure 5.1.2 suggests that more treatments weaken the specimen, subsequently resulting in a lower fracturing pressure. This increase of the likelihood of failure with more treatments can be explained by thermal shock. As the surface of the borehole rapidly cools, the thermal expansion coefficient of the specimen dictates how much the surface of the specimen will shrink. This shrinkage subjects the surface of the specimen to a tensile stress. If the thermal gradient between the cooled surface and the relatively warm interior is great enough, the rock specimen will fail in tension. An increase in the number of cryogenic treatments increases the likelihood that the specimen would fail due to small fractures that have formed during each treatment, which, when added up, significantly weakened the specimen.

Because the samples were tested at unconfined conditions, the fracturing profiles are somewhat random. Even so, we listed descriptions of the generated fractures in Table 5.1.2, as an attempt to characterize and compare the fracture profiles.
Figure 5.1.1 Breakage pressure of specimens as a function of curing time.
Figure 5.1.2 Breakage pressure as a function of treatments.
Table 5.1.2 Characterization of fracture profiles

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fracturing Pressure (psig)</th>
<th>Fracture Description</th>
<th>Fracture Complexity (1-5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>909</td>
<td>Two Vertical Fractures</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>566</td>
<td>Three Vertical Fractures</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>491</td>
<td>Horizontal Fractures, Bottom Broken Off</td>
<td>2</td>
</tr>
<tr>
<td>4</td>
<td>580</td>
<td>Vertical and Horizontal Fractures</td>
<td>3</td>
</tr>
<tr>
<td>5</td>
<td>352</td>
<td>Three Vertical Fractures</td>
<td>2</td>
</tr>
<tr>
<td>6</td>
<td>822</td>
<td>Four Vertical and Horizontal Fractures</td>
<td>5</td>
</tr>
<tr>
<td>7</td>
<td>308</td>
<td>Two Vertical Fractures</td>
<td>1</td>
</tr>
<tr>
<td>8</td>
<td>224</td>
<td>Three Vertical Fractures</td>
<td>2</td>
</tr>
<tr>
<td>9</td>
<td>416</td>
<td>Three Vertical Fractures</td>
<td>4</td>
</tr>
<tr>
<td>10</td>
<td>484</td>
<td>Horizontal Fractures, Bottom Broken Off</td>
<td>2</td>
</tr>
<tr>
<td>11</td>
<td>408</td>
<td>Horizontal Fractures, Bottom Broken Off</td>
<td>2</td>
</tr>
<tr>
<td>12</td>
<td>416</td>
<td>Vertical Fracture</td>
<td>1</td>
</tr>
<tr>
<td>13</td>
<td>400</td>
<td>Three Vertical Fractures</td>
<td>3</td>
</tr>
<tr>
<td>14</td>
<td>313</td>
<td>Numerous Vertical and Horizontal Fractures</td>
<td>5</td>
</tr>
<tr>
<td>15</td>
<td>471</td>
<td>Three Vertical Fractures</td>
<td>2</td>
</tr>
</tbody>
</table>

The fracture complexity is an index that I came up with to help categorize the fractures. It places the fractures formed in a category from 1-5, with 1 being the simplest type of fracture and 5 being the most complex. The goal was to see if more or less complex fractures would form at
certain conditions. Based on the table, the fracture complexity has no correlation with the type of experiment being performed or the pressure at which the sample broke. The only similarities that I found between similar treatments are that dry samples, at room temperature or heated, tend to form horizontal fractures that broke off the bottom of the specimen and saturated samples tend to form three vertical fractures spaced approximately 120° apart.

5.2 Applications of Results

The purpose of this study is to examine cryogenic fracturing in a laboratory setting and make observations to learn the process so that one can apply the process to field testing. This section reviews what can be taken from these experiments.

Based on the experimental results it seems that cryogenic treatment of water saturated samples resulted in the lowest breakdown pressures. Intuitively, this is logical. In addition to the stresses generated from the pressurization of the borehole and the thermal stress generated from the liquid nitrogen, the water frozen inside the formation is expanding, offering an additional stress to fracture the specimen. This information can be useful, because many unconventional reservoirs have significant connate water saturations.

An important aspect of this experiment that is hard to capture in tables and figures is the outlet orifice opening percentage. Unfortunately, this parameter was not realized until mid-testing of Sample 7. It was experimentally found that if the orifice was “too open” the pressure inside the borehole was too low for fracturing and the liquid nitrogen was not making adequate contact with the borehole for enough heat transfer to occur. If we had a larger supply of pressurized liquid nitrogen, I believe this would greatly reduce the heat transfer problem. Similarly, if the orifice was “too closed”, vapor would not escape the borehole easily; instead, it accumulates in the bore hole and insulates the borehole surface from liquid nitrogen, again preventing effective heat transfer. Based on the experimental data, it seems that when the orifice was set to 5% open or less it was “too closed”, but 10% open or higher was “too open”. An 8% orifice opening seems to be the ideal situation for fracturing for this experimental setup. This concept is important for upscaling and transferring this study into field testing. The bottom line is that the liquid nitrogen must adequately flow through the wellbore in order to maximize the temperature differential that creates the necessary thermal stresses. When designing the field study, this must be taken into consideration.
when planning the injection strategy. The liquid nitrogen must be actively flowing against the formation so a vapor cushion does not form and insulate the formation from the cryogenic temperatures. Ultimately, in this experiment, the orifice outlet opening is part of a function that determines the pressure of the sample, the flowrate of the liquid nitrogen, and the temperature of the borehole.

By considering other important factors from this study, we can better conduct field tests. Although only two pressure decay tests in this study had pre-treatment and post-treatment data to analyze, this data was important in determining if the liquid nitrogen would increase permeability. Based on samples 5 and 6, it can be determined that in sample 5 there is clear indication that the liquid nitrogen treatment increased the permeability of the sample as the pressure decay test took significant less time than before treatment. The pressure decay tests also help verify that the Joule-Thomson cooling effect occurred during the test. This will be important for future work as it suggests that in order to better model the experiment, a non-isothermal reservoir simulator may be needed.

In some fracture tests, it was observed that the sample pressure would initially go above that at which the sample ultimately broke. The sample would not fracture at this pressure because the liquid nitrogen had not sufficiently cooled the sample allowing the thermal stresses to occur. In sample 12, for example, the fracture occurred at 416 psi; however, before fracturing, while the pressure was still varying, the sample pressure once reached as high as 449 psi. At this pressure, the temperature of the borehole was 30°C and just started to drop quickly. The sample eventually broke when the temperature of the borehole reached -37°C at a pressure of 416 psi. The sample’s initial temperature was 63°C. This experiment proves that the thermal stress generated in the borehole by the liquid nitrogen is a significant addition to the bore hole pressure when it comes to fracture initiation.

While performing a saturated heated concrete cryogenic treatment on Sample 13, it was observed that the water from the sample frozen by the liquid nitrogen clogged the outlet line. During the experiment, sputtering was initially heard from the outlet valve and then it was almost completely clogged as only a small amount of gas was flowing through the outlet valve even though the outlet was fully open. This observation should be taken into consideration when performing field tests.
CHAPTER 6 CONCLUSION

Just as hydraulic fracturing changed the resource development landscape, cryogenic fracturing offers much promise as a new technology in 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 while decreasing the cost of fracturing resulting in more formations becoming economically recoverable. Cryogenic fracturing has the potential to drastically change the way tight oil and gas resources are developed.

The ultimate goal in stimulation technology is to develop a water-less fracturing technology that will drastically reduce environmental issues associated with the fracturing industry. I believe this study will pave the way for innovation in the quest of the ultimate stimulation technology. Although this study was performed at the laboratory scale on reservoir-like specimens, it lays the foundation for future work to build off of what is presented in this study. If we, as an industry, can develop an effective waterless fracturing technology, the benefits from such an ability would be limitless. Along with increasing potential reserves, this technology would be seen as an acceptable and desirable form of stimulation that may change the way the public view the oil industry. Fracturing would no longer bring with it as much political and environmental tension as waste water generation would be practically eliminated.

To further develop this water-less fracturing technology, future research must go past the unconfined testing that is presented in this study. Confined testing and testing involving reservoir rocks such as shale and tight sandstones must be performed. Once enough information is obtained from laboratory experiments the testing should be moved to field studies in an actual reservoir. Based on the results from these experiments, in the laboratory, cryogenic fracturing is indeed a viable stimulation option that is able to initiate fractures effectively. Although cryogenic fracturing brings with it substantial technical challenges, through this study, much progress has been made in understanding its physical mechanisms. Preliminary results suggest potentially huge rewards in that cryogenic fracturing has the possibility of unlocking new oil and gas reserves. Further research is required to better understand what cryogenic fracturing has to offer by upscaling it to field tests and learning how to integrate it into our current fracturing technology.
REFERENCES CITED


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APPENDIX A

Before this thesis was completed, my colleagues and I published an article in the Journal of Petroleum Science and Engineering that is also based on the work done for this thesis. The following figures show the permissions for me to re-use any data that was previously published from both the publisher and any co-authors of the article.

Figure A-1 Permission from publisher to reuse information in thesis.
Dear all,

I am emailing you all because we are all co-authors to a paper recently published in the Journal of Petroleum Science and Engineering (Cryogenic fracturing for reservoir stimulation). I recently have written and successfully defended my thesis on the laboratory study of cryogenic fracturing of concrete samples; however, part of the introduction and abstract that was used in my thesis was also used in our published paper. In order for me to be able to use this in my thesis I need permission from each of you saying that you grant me permission to use the material. A simple one-line email will do.

Thanks for your time and please email me back if you have any questions,

Thanks!

---

Timothy Kneafsey <tj.kneafsey@lbl.gov>  Mar 25  ⭐

to me, Minsu, Naif, Jennifer, Brent

I grant permission for you to use the referenced material for the stated purpose.
Tim Kneafsey

---

Minsu Cha <mcha@mines.edu>  Mar 25  ⭐

to me

Taylor, you got my permission.

---

From: Taylor Patterson [mailto:twp1124@gmail.com]
Sent: Wednesday, March 25, 2015 2:41 PM
To: Minsu Cha; Timothy Kneafsey; Naif Bandar Alqahtani; Jennifer Miskimins; bjohanso@mymail.mines.edu
Subject: Permission to Publish

---

Naif Bandar Alqahtani <nalaqhta@mymail.mines.edu>  Mar 26  ⭐

to me

Taylor,

I grant permission for you to use the material.

Good luck!
Naif

---

Figure A-2 Permission from co-authors Timothy Kneafsey, Minsu Cha, and Naif Alqahtani to reuse information in thesis.
Taylor-

You have my permission, although I’m confused why the text was used in the first place. Shouldn’t they both be standalone documents?

Dr. Miskimins

From: Taylor Patterson [mailto:twp1124@gmail.com]
Sent: Monday, March 30, 2015 10:05 AM
To: Jennifer Miskimins
Subject: Permission to Publish

Dr. Miskimins,

I recently sent out a group email to you and other co-authors for the journal article "Cryogenic Fracturing for reservoir stimulation - Laboratory Studies" that is published in The Journal of Petroleum Science and Engineering. I am also a co-author for this paper. My thesis, which I am turning in this week, is largely related to this article. I am emailing you because in order to use the material in my thesis from this journal article I need permission from each co-author allowing me to use the material. When you get a chance, could you please reply back giving me permission to use the material in my thesis.

Thank you,

Taylor Patterson

---
Taylor W. Patterson, E.I.T.
M.S. Petroleum Engineering Student CSM
B.S. Environmental Engineering UGA
Golden, CO 80401
twp1124@gmail.com

Figure A-3 Permission from co-author Jennifer Miskimins to reuse information in thesis.
On 3/27/15, 6:34 AM, Brent Johanson wrote:

Hi Taylor,

Good to hear from you, and I hope you're doing well. Congrats on defending your thesis - certainly no small feat!

Yes it's fine to publish - I grant you my permission to do that.

I'd love to see a copy of your thesis, just out of curiosity about your research interests. Forward me a copy?

Take care,
Brent

Figure A-4 Permission from co-author Brent Johanson to reuse information in thesis.