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Development of Non-Contaminating Cryogenic Fracturing Technology for Shale and Tight Gas Reservoirs Project Number: 10122-20

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ABSTRACT

This report presents the continual efforts and progress made and summarizes the results of our research conducted in the second year of the project. We have developed a numerical tool and applied it for preliminary modeling studies of cryogenic fracturing processes. We also have designed and built a lab experimental setup for cryogenic fracturing study under tri-axial loading conditions, which allows to perform proper experiments to capture cryogenic fracturing process in laboratory, such as various loading conditions and different cryogenic fracturing schemes – with and without borehole gas pressure, and pressurized and unpressurized liquid nitrogen flow along borehole. Importantly, we have completed a series of cryogenic fracturing experimental tests and their preliminary analyses, carried out on concretes, sandstones, mancos shale as well as transparent glass samples, using submersion, borehole tests of unconfined specimen, and tri-axial tests with and without borehole pressurization. Data gathered from these experimental tests provided better understanding in cryogenic stimulation mechanisms and fracture processes. In addition, we discuss the challenges, issues, solutions, and work plan for the next phase of the project. To demonstrate the significant progress made, we have submitted two peer-reviewed journal papers with one accepted during the year, presented one poster last year, and scheduled to make two more presentation later this year.

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1. Introduction

The objective of the project is to study, test and develop a technology for cryogenic fracturing method applicable to shale gas and tight gas reservoirs. The research is focused on developing a cryogenic fracturing technology for significant reduction of flow resistance near wells and increase mobile gas volume in unconventional gas reservoirs.

This approach, once proven and completed, will make significant contributions in an environmentally sound manner without using water or other fracturing fluids to improve recovery from shale and tight gas reservoirs. Although cryogenic fracturing brings with it technical challenges, we have made significant progress in understanding its physical mechanisms. Further research is required to better understand and optimize cryogenic fracturing process and how we can integrate it into our current fracturing technology.

In Year One, we conducted preliminary cryogenic tests to understand the cryogen and material behavior. Then, we developed experimental setups and procedures that are designed to conduct cryogenic fracturing tests without confining stress, with integrated cryogen transport, measurements, and fracture characterization. The cryogenic stimulations conducted were able to create cracks in the experimental blocks. Fractures were created along a line of the strongest thermal gradient in a concrete block half-submerged in LN₂. From cryogenic test on different rock block, it is found that the generation of fractures is dependent on material properties. Using transparent specimens, we studied cryogenic fracture process and morphology, which provided insight regarding the expected fracture propagation when sufficient thermal contraction/stress is achieved. It allows for key observations in crack developments at the borehole geometry, such as exclusion distance, crack morphologies driven by both longitudinal and circumferential thermal contraction. It is found that water in the formation plays a competing role during cryogenic cooling with contraction of the rock, thus a complicating factor.

In Year Two, we developed a laboratory system for cryogenic fracturing study under triaxial loading conditions. It allows to perform proper experiments to characterize cryogenic fracturing processes in the laboratory, such as various loading conditions and different cryogenic fracturing schemes – with and without borehole gas pressure, and pressurized and unpressurized liquid nitrogen flow along borehole. Initial data gathered from the new system provided understanding in mechanisms. High gas pressurization following thermal shock create undesirable rapid temperature increase due to discontinued LN_2 supply and adiabatic heating. Liquid nitrogen flowing through borehole under higher pressure decreases temperature more quickly than lower-pressure LN_2 flow due to more suppressed surface vapor cushion under pressure. A reasonable flow rate of cryogen is required to cool the borehole, by either a circulation path to the outside of the borehole or by leakoff through existing or newly created fractures. The decrease of leakoff rate was observed in no-fracture cases probably due to moisture gathered at the borehole surface or by cryogen-induced shrinkage and the resulted permeability reduction.

In addition, we made further progresses in studying fracturing processes and morphology using transparent samples. Cryogenic fracturing in glass blocks was observed under several tri-axially stressed conditions. Fractures occurred perpendicular to the borehole propagating radially from the borehole, as well as parallel to the borehole. These tests indicate that the stress state is very important in determining the extent of the cryogenic fractures. Under a low uniaxial stress, annular fractures extended nearly to the end of the planar fracture parallel to the borehole that formed in the direction of the maximum principal stress. Increasing stress reduced the extent of the fracturing. Also the simulation tool is being developed, and preliminary results are presented.

We are in the early stages of study using the new cryogenic fracturing system equipped with TX system, and will incorporate the observations made into future work. The effect of stress level and anisotropy on fracturing characteristics will be investigated. We will do parametric studies on gas and liquid nitrogen pressure applied to a borehole. For example, we will apply LN_2 flow under varying pressure and varying duration by obtaining a larger vacuum-jacketed LN_2 vessel that can store more than 20 liters and resist 1000 psi. The effect of some of specimen properties, including strain accumulated due to loading and bound water in specimens, on cryogenic fracturing needs to be investigated. The stress-strain behavior of specimens, especially those from target formations – shale and/or sandstones samples – at cryogenic temperature will be studied.

We will continue to improve the models in the simulator and add more components to complete the model. The predictions on the fracture distribution will be compared to the measurements made in the tri-axial loading/fracturing equipment. Continued testing on tri-axially stressed transparent brittle glass, and more ductile plastic cubes is planned to better understand fundamental principles of cryogenic fracturing, including the effect of initial block temperature. Cryogenic fracturing tests on tri-axially stressed shale blocks, and sandstone blocks with varying water saturation will be performed to better understand cryogenic fracturing in shales, and the effect of water freezing in the rock. Better methods of fracture detection will be developed to quantify fracture changes in the tested specimen.

2. Numerical Simulation

2.1 Introduction

Our objective using numerical simulation is to develop a numerical simulation tool based on analytical or numerical approaches to model the influence of cryogenic fracturing and the resulting artificial fractures distribution of the stimulated rock. The fracturing mechanisms will be more clearly understood by developing and applying this tool. The results of the simulation can also be used to guide future field test design.

The simulating tool being developed will be able to handle multiple types of rocks based on different rock properties, including Young's Modulus, Poisson's Ratio, tensile strength, Biot number, thermal conductivity, etc. Furthermore, the simulating tool could be coupled into other simulators to allow simulation of multi-phase flow through a medium to evaluate the result of the cryogenic fracturing treatment. The simulation tool will be used to evaluate the general effect and the distribution of the artificial fractures after the cryogenic fracturing procedures instead of the dimension and actual distribution of the artificial fractures.

2.2 Assumptions

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

- For heat transfer, only conduction is considered, which means that both advection and radiation are neglected. For each grid, the temperature of the rock matrix is always the same as that of the fluid in the pore volume.
- For the fracturing process, the stress change in the rock matrix includes thermal expansion or contraction from temperature change, fluid pressure change in pores, and external stress condition as imposed by the hydraulic press and pistons in the experiment.
- For the rock matrix, the rock is assumed to be homogeneous within each grid. The heterogeneity of the sample is achieved by assigning different properties to different grids.
- For natural fractures, since it is very difficult to characterize, pre-existing natural fractures will be neglected. Only the artificial fractures generated by the cryogenic treatment will be taken into consideration.

2.3 Theoretical Analysis

The simulation tool simulates the cryogenic fracturing process using a grid-based control volume finite difference method. The basic geometry of the simulated well is the same as

that in the experiment: the cryogenic fluid will flow into a borehole and cool 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" by 8" by 8", identical to the dimension of the sample in the actual experiment.

Heat Transfer

The heat transfer or temperature decrement by cryogenic fluid is simulated by the classic heat conduction model. The governing equation is as shown below.

 $(Q_k + \dot{q}V)dt = \rho cVdT$

where Q_k is the heat conduction over all surface of a unit volume (a finite difference grid), \dot{q} is the heat generation for the unit volume, V is the volume of the grid, ρ is the density, c is the specific heat, dt is the time period considered, and dT is the temperature change during the time period.

The continuous form of this equation is as shown below

$$\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} + \frac{\dot{q}}{k_T} = \frac{1}{\alpha} \frac{\partial T}{\partial t}$$

where ∂x , ∂y , and ∂z are the derivatives along the coordinate directions, k_T is the thermal conductivity, and α is the thermal diffusivity.

Thermal Stress

Thermal stress is the stress change due to 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 for a rock, which is as shown below.

$$\sigma_{h} - Bi \times p_{pore} = \frac{\upsilon}{1 - \upsilon} \left(\sigma_{v} - Bi \times p_{pore} \right) + \frac{E}{1 - \upsilon} \left[\alpha_{L} \left(T - T_{0} \right) \right]$$

Here, σ_h and σ_v are the horizontal and vertical stresses, respectively, Bi is the Biot number of the rock, α is linear thermal expansion of the rock, E is the Young's modulus, v is the Poisson's ratio, and p_{pore} is the pore pressure.

Failure Criteria

The failure criteria is used to judge the condition of the rock. It gives the maximum strength of rock under certain stress conditions. Once the stress excesses the maximum strength given by the failure criteria, 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 widely used in rock mechanics. The Mogi – Coulomb Failure Criterion has the following form.

$$\tau_{oct} = k + m\sigma_{oct}$$

where τ_{oct} and σ_{oct} are octahedral shear and normal stresses, defined as:

$$\tau_{oct} = \frac{1}{3} \sqrt{\left(\sigma_v - \sigma_H\right)^2 + \left(\sigma_v - \sigma_h\right)^2 + \left(\sigma_H - \sigma_h\right)^2}$$
$$\sigma_{oct} = \frac{1}{3} \left(\sigma_v + \sigma_H + \sigma_h\right)$$

In the above equations, k and m are constants that are usually obtained from fitting actual data and σ_H is the maximum horizontal stress. The failure envelope from Mogi – Coulomb Failure Criterion is as shown in Figure 1. Normally the stress conditions of rock make the calculated τ_{oct} and σ_{oct} fall into the blue area under the failure envelope. When the stress condition changes, i.e. due to cryogenic treatment, the calculated τ_{oct} and σ_{oct} may fall onto a point outside of the failure envelope, the rock would then be fractured.



Figure 1: Failure Envelope of Mogi - Coulomb Criterion.

2.4 Preliminary Results

The simulation tool is undergoing continued development, therefore here we present some preliminary results. In the following sections, we will describe the problem set up and discuss the results and their implications when compared to a previous test without tri-axial loading.

Problem Setup

The details of this case are as follows. Our domain is a sandstone cube with outer dimensions 20.32 cm x 20.32 cm x 20.32 cm (8 in x 8 in x 8 in). A 2.54 cm (1 in.) diameter borehole is centrally located on one face extending 15.24 cm (6 in) into the block. Liquid nitrogen (LN_2) is injected into the borehole with pressure slightly higher than ambient pressure at a constant rate. The injection starts at time 0 and stops at 4200 seconds. For comparison, the injection in the real test was stopped about 4220 seconds after initiation of injection.

The outer boundary, which consists of the six surfaces of the sandstone block, is exposed to ambient pressure and temperature, which are 11.8 psia (81.4 KPa) and 66 °F (19 °C or 292 °K). The sample is initially set at the ambient temperature, which is 66 °F. The thermal diffusivity of the sandstone sample is currently assumed to be the same as that of quartz, which is 3.33×10^{-6} m²/s. The permeability of the sandstone sample is set as 30 mD. The constants from Mogi – Coulomb criterion are set as k = 12 MPa and m = 0.67. And the permeability of fractured grids is set as 200 mD.

Results

The simulation was run on 64 by 64 by 64 grids for the 8" by 8" by 8" sandstone sample. Figure 2 and Figure 3 show the temperature distributions inside the sandstone sample at the end of liquid nitrogen injection for different cross sections.



Figure 2: Temperature distribution on an XY cross-section in the middle of the sample at the end of injection (unit in °K).



Figure 3: Temperature distribution on an XZ cross-section in the middle of the sample at the end of injection (unit in °K).

As shown in Figure 2, the temperature inside the sandstone sample increases gradually starting from the borehole forming a circular shape around the borehole. Figure 3 shows that on the XZ plane, the temperature distribution is influenced by the block and borehole geometry. The maximum temperature drop takes place at ½ of the borehole depth.

Figure 4 portrays the temperature reduction profile of the same XZ cross section shown in Figure 3. As shown in Figure 4, most of the temperature drop happens very close to the borehole surface. The borehole is cooled to the boiling point of liquid nitrogen, which is - 197.6 °C or 75.5 °K (at ambient pressure in lab). The surface of the sandstone sample remains at the ambient temperature.



Figure 4: Temperature reduction on the XZ plane in the middle of the sample at the end of injection.

Figure 5 shows measured and simulated borehole temperature changes with time for the sandstone sample. As shown in Figure 5, the dotted lines (simulated temperatures) agree very well with the solid lines (measured temperatures) in the test.



Figure 5: Borehole temperature and surface temperature comparison.

Figure 6 shows the simulated fracture distribution inside the sandstone sample after the cryogenic treatment. The green grids in Figure 6 are the fractured grids, where the stress condition exceeds the failure envelope from the Mogi – Coulomb Failure Criterion, which is mentioned in Section 2.3. The red grids stand for the borehole and the blue grids

are the intact grids. The fracture distribution coincides with the temperature distribution because the fracture model relates fractures to large temperature drop.



Figure 6: Predicted fracture distribution inside the sandstone sample after cryogenic treatment.

The original permeability of the sample was set as 30 mD. After the cryogenic fracturing treatment, the effective permeability of the sandstone reached 53.3 mD, a 77.6% increase over the original permeability. This effective permeability is calculated by plugging the fracture profile into a flow model to achieve the same flow capacity with a homogeneous block. However, this result is highly dependent on the validity of the assumptions taken in the model and especially the choice of the permeability of the fractured grids (taken to be 200 mD in this calculation). Based on this model, the large increase in the permeability is due to the small relative size of the sample block relative to the borehole size and the long treatment time. This result still needs to be calibrated with the actual tests.

2.5 Discussion

Since the simulation tool is being developed, the results presented are only preliminary. We will continue to improve the models in the simulator and add more components to complete the model. The predictions on the fracture distribution will be compared to the measurements made in the tri-axial loading / fracturing equipment.

3. Experiment Designs

We consider two cryogenic stimulation plans: thermal shock and thermal shock combined with borehole pressurization. Fracturing by thermal shock uses thermally induced tensile stress to initiate fractures. After application of the initial thermal shock, borehole pressurization can help to propagate the fractures. We have designed and built three experimental setups: submersion, borehole stimulation without confining stress conditions, and borehole stimulation with confining stress conditions that uses a tri-axial loading system. These setups have already been presented in the previous annual report. Here, we briefly recap the descriptions and diagrams of the setups.

3.1 Submersion Test

Submersion tests consist of a concrete block placed in an insulating container. The container is filled with liquid nitrogen until the block is fully submerged. The test observations run for about 50 minutes and then the sample is left for several hours for the liquid nitrogen to finish evaporating and the sample to re-equilibrate with room temperature. Figure 7 shows the setup.



Figure 7 Submersion test setup

3.2 Borehole Thermal Shock without Pressurization

In this test setup, we cool the borehole as rapidly as possible by flowing LN_2 continuously through the borehole. The basic scheme is illustrated in Figure 8. LN_2 is pumped from the dewar (an insulating tank for LN_2 storage) by pressure difference using a liquid nitrogen withdrawal device. Liquid nitrogen is transported by a vacuum-jacketed

hose to the specimen, and injected into the borehole and then directed to an outlet. A pressure transducer is attached to monitor the borehole pressure. In this thermal shock setup, pressure inside the borehole is approximately the same as the pressure inside the dewar. The experimental equipment employs cryogenic-rated transport, control, and measurement systems. We have set up real-time monitoring and logging of various parameters including pressure inside the borehole, LN_2 consumption, temperature, and acoustic signals. A structure that confines a packer in place is built to prevent leakage through the packer.

3.3 Borehole Thermal Shock with Pressurization

This test plan is to enhance the fractures created by thermal shock by applying a level of pressure to the borehole during and/or after the thermal shock. One scheme of pressurization can be performed by letting existing LN_2 in the borehole evaporate, while shutting off all of the inlet and outlet valves. Another scheme is by increasing pressure in the borehole by applying nitrogen gas (Figure 9). High borehole pressurization is possible for confined specimens loaded by the triaxial loading equipment. However, unconfined specimens cannot sustain much borehole pressure (rock splitting observed in weak concrete due to pressurization at pressure lower than ~100psi).

3.4 Borehole Test with Tri-Axial Stress Conditions

A tri-axial loading system has been built to simulate reservoir confining stress conditions. The system operates two hydraulic cylinders and a press to load up to 6500 psi vertically and 4500 psi horizontally in $8"\times8"\times8"$ blocks, and is able to keep quasi-constant force using its hydraulic control system (Figure 10).

Most other commercially available tri-axial loading equipment use membrane packs that are connected to the hydraulic power system, and are closed systems where the confining structure and loading pad, or piston, encloses the specimen. Our equipment is a straightforward open system where all three loading drivers and the specimen blocks are exposed and assembled inside the containment ring. This open system is versatile because it is easier to observe internal processes during the experiment and we can act immediately upon an accidental internal problem (e.g. cryogen spill). This also makes the system much less expensive than hydraulic power systems contained by flexible membrane bladder. The simple, yet flexible design can also easily be converted for hydraulic fracturing experiments.

The only expected disadvantage of this system is that it is not ideal for specimens with uneven surfaces or tilted surfaces, although the pistons can accommodate small tilt. Uneven surfaces or significantly tilted surfaces will create uneven stress distribution. At high stress, this may fracture the rock. While we are trying to prepare even and untilted specimens as much as possible, the effect of uneven and/or tilted surfaces can be alleviated by inserting flexible rubber pads to evenly distribute stresses.



Figure 8 Schematic drawing for cryogenic thermal shock experiments without borehole pressurization.



Figure 9 Schematic drawing for cryogenic stimulation experiments with borehole pressurization.



Figure 10 Tri-axial loading system designed for cryogenic fracturing experiment.

4. Cryogenic Stimulation Experiments

Several tests will be conducted on three different rock types. These three rock types are concrete, sandstone, and shale. The concrete samples are made in the laboratory while the sandstone and shale samples are acquired. Table 1 shows generic values of the mechanical and thermal properties of the samples.

These samples will be tested under different stresses and temperature scenarios including unconfined and confined tests. More test details are presented later in this section.

Rock Type Properties	Concrete	Sandstone	Shale
Density (gm/cc)	2.24 - 2.4	2.2 - 2.8	2.4 - 2.8
Compressive strength (psi)	2900 - 5800	2900 - 25000	720 – 14000
Tensile strength (psi)	290 - 720	580 - 3600	290 - 1400
Young's Modulus (10 ⁶ psi)	2-6	0.15 – 3	0.15 – 10
Poisson's ratio	0.20 - 0.21	0.21 - 0.38	0.20 - 0.40
Shear strength (psi)	870 - 2500	1200 - 5800	440 - 4400
Specific heat capacity (kJ/kg K)	0.75	0.92	0.88 - 1.09

 Table 1: Mechanical and Thermal Properties for Subjected Samples

Moreover, we continue to carry out experiments on transparent glass samples to study fracture morphology and its relation to stress conditions. These experiments are also reported in this section.

4.1 Submersion Tests

Submersion tests were conducted using the device shown in Section 3.1 (Figure 7). The concrete block tested showed significant shrinkage while cold and several fractures were identified. The main fractures formed polygonal shapes around the exterior of the block when inserted dry. This behavior indicates that the block was fractured due to the application of the thermal gradient. Figure 11 shows the dimensions of the concrete block.



Figure 11 Illustration shows the dimensions of the concrete sample that has been used in submersion test. Side (3) is the opposite face of side (1), and side (4) is the opposite face of side (2).

Figure 12 shows the images of the top side of the concrete block test before and after the submersion test. The image marked as "after" was taken immediately after the cryogenic test because the fracture was clear and easy to be observed by the naked eye. Fractures formed a polygonal network. Since there are no stresses applied during this test, the block was fractured purely due to the application of the thermal shock. All faces of the block had newly created fractures and/or extensions of existing fractures. Figure 13 shows a new fracture on face 1. Next to it is a pre-existing fracture that was extended vertically. The scale of this picture is 1 mm. After the sample relaxed back to the room temperature, some fractures were observed to close. This test will also be conducted on sandstone and shale gas rocks to compare with cement.



(a) before (b) after Figure 12 Before and after images of the concrete block top side after the full submersion test. The after (b) figure shows fractures in the block resulting from cryogenic fracturing.



(a) before

(b) after

Figure 13 Before and after images of the concrete block Side 1 after the full submersion test showing the creation of large, new fractures resulting from cryogenic fracturing. Red arrows show the locations of the fractures on the surface of the concrete sample.

4.2 Borehole Test of Unconfined Specimen

Compared to the submersion test, borehole test simulates cryogenic stimulation with a more realistic geometry. It also allows multiple measurements be conducted. The measurements that taken during the tests include:

- **Pressure:** Pressure transducers are used to measure the pressure inside the wellbore. In cryogenic tests the transducers will be placed far from the LN₂ and we will use hot water tube or thermostated electric heater to warm up the tube with LN₂ before it reaches the pressure transducer equipment since the transducers are generally not capable of withstanding very cold temperature environments.
- **Temperature:** Seven thermocouples (type T) are placed in different locations on the system. Figure 14 shows where these thermocouples placed. Type T (copper constantan) thermocouples are suited for measurements in the –200 to 350 °C range.



Figure 14 The locations of the thermocouple sensors in block tests. The unconfined system that does not use borehole pressure is shown here as an example.

• Acoustic measurement: The P and S wave signatures before and after the cryogenic test are compared. This method shows the differences in the acoustic signatures before and after the cryogenic tests. Figure 15 shows the locations for the acoustic measurements.



Figure 15 Acoustic ultrasonic transducers locations.

- LN_2 consumption: The amount of LN_2 used will be measured by monitoring the weight of the dewar using a scale.
- X-ray computed tomography (CT scan): CT scans are used to generate a threedimensional image of the inside of an object from a large series of two-dimensional images taken around a single axis of rotation. CT scanning is carried out before and after the cryogenic test to detect large and visible fractures resulted from the stimulation test.

In this section, we present thermal shock experiments on multiple concrete and sandstone specimens under relatively low borehole pressurization. The sandstone samples were taken from a nearby surface quarry and the cement samples were made in-house and water-cured for at least 6 weeks. The submerged curing of the cement block greatly enhances its compressive and tensile strength, resulting in a specimen whose mechanical properties are more similar to those of reservoir rocks.

Why Unconfined Tests

While a reservoir rock under unconfined conditions does not replicate the environment seen downhole in the actual reservoir, there are numerous benefits to stimulating these samples at surface conditions. Performing these relatively inexpensive stimulations allow us to gauge if our experimental setup is effective in fracturing the specimen before taking it full scale and running it under reservoir stress conditions. We also perform these unconfined stimulations tests relatively quickly for very low cost to gather the data needed to optimize our future testing under reservoir stress conditions. By having the ability to test our samples under unconfined conditions we are able to test the effectiveness of the stimulation technology without having to apply reservoir conditions to the sample thus saving time and money.

Previous Work

Last year, much preliminary experimental work was done that led us into our current state of design for the unconfined packer system. As a refresher, we will quickly cover the highlights from last year's work that leads us into what we have accomplished thus far and what we plan to accomplish in the near future. In our previous experiments, we explored the idea of generating a fracture network by creating a thermal shock to a reservoir. To achieve this, we treated the rock specimen by injecting the cryogen fluid into the wellbore by using pressure generated from the dewar container at 10 psi. Figure 16 shows the initial setup we used to stimulate and monitor the fracturing procedure.



Figure 16 Initial experimental setup

Although our initial experiment was somewhat crude, it was also successful as we obtained valuable data. We learned of a number of hurdles to overcome if we wanted accurate data that represented effective stimulation of our reservoir specimen.

In the simplest terms we discovered that in order to determine the effectiveness of our stimulation treatment we need better rock samples, to overcome the Leidenforst effect, and a way to accurately measure and interpret the effects of our treatment on the reservoir specimen. By incorporating these needs into our next design the team has engineered the following experimental setup.

Unconfined Tests without Borehole Pressurization

We attempted to create fractures in boreholes drilled into different types of samples (concrete, sandstone, and shale) with dimensions 8 in x 8 in x 8 in at atmospheric pressure. In early tests, the initial temperature will be set at the room temperature and in later tests set at a reasonable reservoir temperature. Since we did not pressurize the borehole, LN_2 were naturally flowed into the borehole by gravity. The experimental equipment employs cryogenic-rated transport, control, and measurement systems. In addition to the experimental setup illustrated in Figure 8 which we used last year, we set up and used the simpler system as shown in Figure 17. There, epoxy is used to bond the casing to the formation and to set a wellhead on the top of it. No packer is used in this setup. A funnel is connected to the casing to allow LN_2 be poured in. Measurements for this setup include temperature, acoustic velocities, LN_2 consumption, and CT scanning. For all cases, specimen are photographed before and after the stimulation.



Figure 17 Schematic for cryogenic stimulation experiments for unconfined tests using a funnel.

In the experiment, LN_2 was poured into the wellbore through the funnel (Figure 17). The LN_2 volume poured each time was about 350 in³. Figure 18 and Figure 19 show the results of two consecutive cryogenic tests applied to the same sandstone sample. Along the blue line showing the temperature at the top of the block specimen, the red arrows mark the times when the LN_2 was poured. The temperature of the formation in the wellbore (#1) did not reach the boiling point of the liquid nitrogen (#2), indicating that the Leidenfrost effect prevented the liquid to be in contact with formation. Thus, a ventilation setup should be added to this setup to remove the vapor cushion in the Leidenfrost effect, or, a pressure may be applied on the LN_2 to reduce the Leidenfrost barrier for heat transfer. We are currently modifying the setup shown in Figure 9 to make this test possible. An updated diagram is shown in Figure 20.

It is worth noting that this test showed some enhancement in the permeability by comparing the pressure decay test before and after the cryogenic test. Before the cryogenic test, the pressure inside the wellbore used 47 minutes to decay; after the cryogenic test, the decay time was 39 minutes. This change clearly indicates permeability enhancements that can be due to fracturing.

Even though we did not use a packer, the epoxy used to attach the casing to the sample withstood the thermal shock during our first two tests. However, on the third test, the epoxy was damaged (Figure 21).



Figure 18 Temperature values during the 1st cryogenic test. The numbers (1-7) are the locations of the thermocouples sensors in a borehole (Figure 14). Red arrows in the figure mark the times when LN_2 was poured.



Figure 19 Temperature values during the 2nd cryogenic test. The numbers (1-7) are the locations of the thermocouples sensors in a borehole (Figure 14). Red arrows in the figure mark the times when LN_2 was poured.



Figure 20 Schematic for cryogenic stimulation experiments for unconfined tests using pressurized N2.



Figure 21 Epoxy damaged by repeated exposures to the cryogenic temperature.

Plan to Conduct Leak-Off Tests to Quantify the Effect of Cryogenic Stimulation

To measure the effectiveness of the stimulation treatment, pre-stimulation and poststimulation gas leak-off tests will be performed. This is done by pressurizing the specimen with nitrogen at a constant pressure (~100 psi) and flow rate (~600 mL/min) and shutting it in, which is shown schematically in Figure 22. We then measure the time it takes for the pressure of the sample to reach the baseline pressure which was set before the experiment took place. The sample is then stimulated with the cryogenic treatment previously outlined. After the stimulation is completed and the specimen is back to STP we will perform another gas leak-off test. This experimental setup is shown in Figure 23. By measuring whether there is any significant difference in the leak-off rate, we are able to quantify whether cryogenic treatment has increased the permeability of the specimen. If the gas leaks off at a faster rate than it initially did, we have successfully increased the permeability of the specimen. An interesting phenomenon occurred while monitoring the temperature while shutting in the wellbore. As shown in Figure 24, the temperature of the hanging thermocouple (#2 in Figure 14) increased when we began pressurizing the borehole of the specimen and decreased as we shut in the reservoir specimen. We believe this is due to a gas being compressed under adiabatic-like conditions. As a constant volume is pressurized, the temperature should increase; likewise, as a constant volume is relieved of pressure, the temperature should decrease. The attached thermocouple remained unchanged as the wellbore wall that it is attached to has a much greater thermal

capacity, making the temperature variation there insensitive to the temperature change due to pressure change inside of the borehole.

Once the leak-off test system and the models for interpretation are in place, we will be able to quantify the effect of the treatment. This will allow us to compare different treatments and treatment conditions and determine the best practice.



Figure 22 Constant pressure and flow rate are reached before being shut-in for the subsequent leak-off test.



Figure 23: Pressure Decay Rate and Mass Flow Equilibrium Equipment.

Table 2: Identification of Parts in Figure 23.		
ID Number	Item	
1	Pressure Transducer	
2	Mass Flow Meter	
3	Nitrogen Gas In	
4	Pressure Relief Valve	
5	Sandstone Specimen	
6	Data Reader	
7	Power Source	


Figure 24 Observation of the ideal gas law.

Going forward, we will produce more thorough results for the unconfined stimulation experiments. To do this we have re-designed the unconfined experiment to allow for the pressurized injection of cryogenic fluid into the wellbore. We believe that this will eliminate, or greatly reduce, the Leidenfrost effect that is currently limiting the generated thermal gradient. Figure 20 shows our updated design over what we planned last year (Figure 9). As shown in the figure, an accumulator will be filled with liquid nitrogen using the passive pressure that is generated inside the dewar storage tank (~10 psi). Once the accumulator has accumulated enough cryogen fluid, the valve leading into the dewar holding tank will be turned off. We will then pressure the liquid nitrogen using nitrogen gas to ~150 psi. By opening the ball valve that leads to the specimen we will be able to effectively treat the specimen with pressurized liquid nitrogen. As you can see in Figure 25 the specimen wellhead is designed to allow for gas to escape the wellbore allowing the treatment to come into direct contact with the surface of the wellbore, thus reducing or eliminating the Leidenfrost effect.

The plans are in place to begin experimenting with multiple unconfined pressurized cryogenic treatments over the next several months. Sandstone, cement, and Niobrara shale samples have been gathered and are ready for testing. Once these tests have been performed we will evaluate the efficacy of the new design and move forward from there.



Figure 25 Schematic of Wellhead Injection Design

4.3 Tri-Axial Tests with Borehole Pressure

Devices

We have developed a laboratory system for cryogenic fracturing study under triaxial loading conditions. It is mainly consist of a triaxial loading system and a liquid nitrogen delivery and other control/measurement system (Figure 26 and Figure 27).

Triaxial Loading Device. The triaxial loading (TX) system can load up to 4500 psi in x and y axes, and 6000 psi in z axis, and can independently control loadings in the three axes. The two hydraulic pistons (x and y axes) and the hydraulic press (z axis) are powered by three pneumatic hydraulic pumps. Three heavy-duty ratchet tie-down straps (5000 lbs. each) surround the containment for extra safety. One practical advantage of our system is the vertical loading frame can be easily removed by rolling it away after unlocking it from the bed. This ability provides a user with space to work on specimens and inside the containment.



(a) TX loader mounted in the press bed frame (b) X&Y axes within the containment Figure 26. Triaxial loading system.

Cryogenic fracturing system under TX condition. The cryogen fracturing system under TX condition has liquid nitrogen injection system, GN_2/LN_2 pressurization, and measurement/data acquisition system. Compressed N₂ gas source is used to either directly pressurize boreholes or to push liquid nitrogen into borehole at high pressure.



Figure 27. Laboratory system for cryogenic fracturing study with triaxial loading.

A liquid nitrogen vessel is specially built to temporarily store liquid nitrogen before it is pushed into the specimen borehole. The vessel is made of annealed stainless steel tubing with 2"OD, 1.62"ID, and 2.3ft length (Figure 28). The 2"OD tubing is reduced to ¼"OD tubing using multiple tube fittings. The vessel tubing is rated for 1000 psi, but 500 psi is maximum recommended pressure at cryogenic temperature from the vendor. The vessel is heavily insulated to minimize heat transfer, and its internal storage volume is 0.95 liter.



Figure 28. Liquid nitrogen vessel made of 2"OD annealed stainless steel tubing and fittings to store liquid nitrogen (Photo taken before applying insulation).

Gaskets were used to seal between packers and the surface around boreholes. The gaskets that we used are made of PTFE, which resists temperatures down to -350 °F. To provide good contacts between gasket and specimen surface, epoxy is applied to a packer/gasket seat to fill any uneven surfaces created during concrete making to provide tight seal between gasket and specimen surface (Figure 29).



(a) Specimen #9

(b) Specimen #16

Figure 29. Locations (dents) where gaskets and packers are placed. Uneven surfaces are filled with epoxy shown as darker areas in the pictures.

Thermocouples (one on the borehole wall, and the other hanging in the borehole) are placed first before applying gaskets and packers. Then, packers are loaded by top load platen to create tight seal (Figure 30).



(a) Silicone pad, packer, and gasket (left to right) (b) Thermocouple attached to borehole wall

Figure 30. Placing thermocouples, gaskets, and packers.

Triaxial Loading Tests

Forces exerted by the hydraulic pistons are calculated by multiplying hydraulic pressure in the hydraulic lines measured using pressure transducers by effective area of hydraulic pistons. Then the specimen stresses are obtained by dividing the piston forces by the specimen surface areas. Figure 31 shows loading axes and location of specimen sides.



Figure 31. Loading tests with loading axes and specimen side numbers shown.

Constant forces can be maintained by either manual control or in quasi-automatic manner using pressure relief valves attached to the hydraulic lines. Natural decay of pressure in hydraulic lines are unavoidable due to hydraulic leak; therefore, hydraulic pressure needs to be kept by regular updates. Our hydraulic system make use of pressure relief valves to quasi-automatically constant pressure in the hydraulic lines. The load test in Figure 32 shows some drift, therefore it does require occasional check – so we call it "quasi-automatic", but needs less attention than the manual control.



Figure 32. Loading tests - Quasi-automatic control using pressure relief valves (2000 psi).

In the manual control, we manually pump small amount of pressure when certain amount of natural decay occurs. The manual control can be more accurate than quasi-automatic control, but require more attention to changes in pressure. Note that the Figure 33b is manually controlled within $\pm 2\%$ of errors, which is possible by reasonable attention. In our cryogenic stimulation tests, we control stresses manually for better accuracy.



(a) Loading to 1000 psi specimen stress



Figure 33. Loading tests – manual control of constant stress (specimen stress 1000 psi, 2000 psi, and 4000 psi).

It appears that the silicone pad that we used functioned well in uniformly distributing the load on the uneven surfaces of the sample, as we did not observe any specimen damages/cracks after the loading tests up to 4000 psi. Especially, the top surfaces are quite rough, but the silicone pads did excellent jobs in distributing load over the entire area of the surfaces. However, it appears that the pad seems too soft as we observed that the pad extruded out the contacts at high loading (Figure 34b&c). The silicon pads resist temperature down to -60°F. In our short-term tests (less than 1hr), this rating was sufficient as the block surface remained close to the ambient temperature.



(a) No loading (b) At 2000psi loading (c) At 4000psi loading Figure 34. Extrusion of the silicone pads under loadings.

After the concrete specimens are cured and produced, the top surfaces were quite rough and undulating. Therefore, the top surface is ground by chisel as much as possible, but still there are some uneven areas. The silicon pad did decent job in distributing load along the surface, but is also damaged after several repeated uses as it extruded out of the surface against rough and uneven surfaces under high pressure. As the pad materials extruded out of the contact, some pad materials are smeared into the surfaces as shown in Figure 35. The amount of smeared materials increased with increasing pressure (Figure 35).



(a) After 1000 psi loading(b) After 2000 psi loading(c) After 4000 psi loadingFigure 35. Silicone material smeared into the concrete block (Specimen #9)

One notable observation we made after loading tests is that some natural dimples on the specimen surface that existed before the tests were enlarged. There were also new dents created. This is because the materials of silicon pads intruded the open pores at high pressure due to its softness and flexibility. Results are pores become widened, and closed pores located much closed to the surface collapsed. However, these behavior did not affect integrity of specimen.



(a) Before loading (b) After loading (c) Silicone intrusion into pores Figure 36. Enlarged holes and newly surfaced holes after loading up to 4000psi (Specimen #7).

For next tests, we will also try PTFE sheets, which is much stiffer than the silicon pads, but expected to be soft and elastic enough at high pressure to accommodate any local, undulating surfaces, and have exceptional temperature rating down to -350 °F.

In some of early loading tests that we performed, unloading processes were abrupt and not perfectly synchronized in all three axes, which created acute anisotropic stress conditions in the specimen blocks. For example, Specimen #9 were subjected to anisotropic condition where x and y axes are loaded to ~ 2000 psi and z axis is loaded to ~ 0 psi due to unsynchronized rapid unloading for a couple of times (each time anisotropic condition lasted about 0.5 sec).



Figure 37. Prominent fracture along the centers at the sides created due to repeated, acute anisotropic conditions (Specimen # 9).

After this observation, we devised to make use of pressure relief valves to slowly and simultaneously unload the three axes as shown in Figure 38.



Figure 38. An example of gradual, synchronized unloading that minimized the abrupt anisotropic conditions, using pressure relief valves in the hydraulic system.

Cryogenic Tests under Triaxial Loading Condition

Using our cryogenic fracturing apparatus under triaxial loading condition, we performed two different cryogenic stimulation schemes: *thermal shock followed by pressurization* and *liquid nitrogen flow under pressure*. We applied an isotropic specimen stress 2000 psi in all the axes to the specimens.

In *thermal shock followed by pressurization*, the thermal shock to boreholes were performed by first flowing liquid nitrogen directly from Dewar with low pressure 10~15 psig. Then, when the borehole was sufficiently cooled down for a 10~30 minutes, we closed liquid nitrogen injection, and pressurized the borehole with high nitrogen gas pressure (in our experiments, we applied a nitrogen gas pressure of about 450 psig to the borehole).

In *liquid nitrogen flow under pressure*, the special vessel (Figure 28) was filled with liquid nitrogen from Dewar temporarily. After the vessel was fully filled, we injected the liquid nitrogen in the vessel to the borehole at high pressure by using a high gas nitrogen pressure which is connected to the LN_2 vessel (in our experiments, we applied a nitrogen gas pressure of about 450 psig to the vessel).

			1	61	
		Specimen	Stress	Measurements	Permeability
			condition		indicator test
Thermal shock		# 16	2000 psi	P- and S-waves, temperature	Permeability
followed by			isotropic	(borehole, borehole wall, &	test before and
pressurization				specimen surfaces), fluid	after stimulation
				pressure, & specimen	test
				stresses	
LN ₂	1 st	# 16	2000 psi	Same as above	Same as above
flow	try		isotropic		
under	2 nd	# 16	2000 psi	Same as above	Same as above
pressure	try		isotropic		

Table 3. Cryogenic stimulation experiments under triaxial loading performed

Thermal shock followed by pressurization

In this scheme, the plan is that we use a low pressure LN_2 injection to initialize fractures at the borehole by thermal shock. It is followed by a high gas nitrogen pressure in an attempt to open up cracks generated by the thermal shock. If cracks are not made from the initial thermal shock, the following pressurization would still help generate fractures in a weakened borehole wall that is under thermal tensile stresses/contraction. As shown in Figure 39b, liquid nitrogen was injected until borehole and borehole wall was fully cooled down to the nitrogen boiling point. As soon as the liquid nitrogen injection was stopped, we opened gas nitrogen gas to the borehole, pressurizing it at about 420 psi. During the pressurization period (about 20 seconds), rapid temperature increase is observed probably following the adiabatic compression of gas. The light blue arrow in the Figure 39b may indicates fluid transition to complete liquid phase in the borehole. However, the boiling at the borehole wall surface continued for more than 20 minutes after that, as we see that the borehole wall did not reach nitrogen boiling point for an extended time.



Figure 39. Thermal shock & pressurization under 2000 psi isotropic confining stress.

Contraction and expansion of specimens were observed from pressure responses of the hydraulic system. When the specimen was cooled down, the specimen shrank and the piston in contact with the specimen lost pressure at a faster rate than at the steady-state condition. When the temperature of the specimen was increased, the specimen expanded and the piston started to pick up pressure from the expanding specimen, making the hydraulic pressure increase.



Figure 40. Indication of shrinkage and expansion of the specimen due to temperature changes from hydraulic pressure responses – during *thermal shock followed by pressurization* (Specimen #16)

LN₂ flow through borehole under pressure

By performing high-pressure liquid nitrogen flow through borehole, we expect faster borehole wall cooling and simultaneous, combined thermal shock/pressurization effect.

In order to have LN_2 flow under pressure, liquid nitrogen needs to be temporarily stored in a special container outside of the Dewar, as the Dewar cannot accommodate any high pressure. Therefore, the vessel was filled with liquid nitrogen from the Dewar. Figure 41 shows the temperature history during LN_2 filling recorded by a thermocouple attached at the top of the LN_2 vessel. At time 46 min, the temperature became plateaued, which indicates that the equilibrium temperature was reached soon after the vessel was filled with LN_2 . It took 28 minutes to fully fill the vessel with LN_2 .



Figure 41. LN_2 vessel filling estimates from temperature changes at the top of the LN_2 vessel (Photo was taken before applying insulation).

It took a long time to fill the vessel with LN_2 because the initial nitrogen leaving the Dewar is mostly gas. Another reason is that the vessel was much warmer than LN_2 , and much evaporation must take place before the vessel can be cooled down to accommodate liquid nitrogen. LN_2 filling took significantly less time in the 2nd filling to the vessel because of the much colder vessel and agitated Dewar from the 1st filling (Figure 42).



Figure 42. Time taken to fill up the LN₂ vessel.

The same concrete specimen (#16) used for *thermal shock* & *pressurization* was also used for LN_2 flow through borehole under pressure. After filling up the LN_2 vessel, we applied ~420psi gas nitrogen pressure to the LN_2 vessel from the top of the vessel and opened the vessel outlet located at the bottom of the vessel to the specimen borehole to inject liquid nitrogen at high pressure and high flow rate. However, in the 1^{st} attempt, we did not open the borehole outlet valve. This resulted in a temperature in the borehole that did not decrease (Figure 43) because there did not appear to be enough permeation through the specimen (under high confining stresses) to vacate the gas in the borehole to let in the liquid nitrogen. There must existed a long, warm nitrogen gas cushion between the LN₂ vessel and the borehole when the borehole outlet was closed.

After this observation, we opened the borehole outlet valve at time 58.3 min as shown in Figure 43 to let in the liquid nitrogen. Upon the valve opening, the nitrogen (initially gas, and later on gas and liquid mixture) came out of the borehole outlet at a very high flow rate. Temperature decreases fairly rapidly in the borehole, at a much faster rate than the thermal shock under ambient pressure (10~15psi). This rapid decrease of temperature may be because of more suppressed surface boiling/ vapor cushion (Leidenfrost effect) due to borehole pressure and faster liquid nitrogen circulation.



Figure 43. LN_2 flow under pressure -1^{st} attempt (LN_2 pressurization while initially borehole outlet is closed).

After the 1st attempt, while keeping the current rig, we attempted another LN_2 flow under pressure experiment. In the 2nd attempt, the borehole outlet was open at first to let pressurized LN₂ enter the borehole. As observed before, the liquid nitrogen rapidly discharged through the borehole to the outlet. The slope of temperature drop is more rapid than the 1st attempt's discharge because, in the 1st attempt, nitrogen may have warmed up a bit when it was not moving for a few minutes before it was discharged. LN₂ depleted from the vessel before the borehole temperature reaches the lowest possible temperature (-196°C). Therefore, it is concluded that the volume of the LN₂ vessel (0.95 liter) is too small to sufficiently cool the borehole and at the same time maintaining a high pressure.



Figure 44. LN_2 flow under pressure – 2^{nd} attempt (LN pressurization while borehole outlet is open).

Fracture assessment

Several methods are applied here to check whether any fracture has been created by the two cryogenic stimulations. They include permeation test, pressure decay, acoustic waves, and visual inspection. The permeation test measures a flow rate at a constant

borehole pressure. The permeation test was performed under the same triaxial loading that was used for the cryogenic tests. The boundary permeation except at the edges may be inhibited due to the silicone pads, but since the conditions were the same for all permeation tests, we can compare the results. Table 4 shows flow rates at several different borehole pressure before and after the cryogenic stimulations. The tests were performed right after the cryogenic test at which the specimen was still cold and 12 hours after the cryogenic test when the specimen was much closer to the ambient temperature. From Table 4, there is a consistent trend that flow rate decreased after cryogenic stimulations. These are unexpected results as we thought the flow rate would increase due to the generation of fractures or microcracks during the cryogenic stimulations. It may be possible that borehole and specimen surfaces attracted frost or moist while the specimen were cold, which blocked some pores near the surface, reducing permeation. It is also possible that under the isotropic stress condition, shrinkage of the sample volume due to the low temperature reduced the permeability of the sample and the reduced permeability did not fully recover even after the sample returned to the ambient temperature.

		Borehole pressure	Flow rate
Before the thermal		44 psi	400 ml/min
shock & pressurization			
After the thermal shock	Right after	63 psi	505 ml/min
& pressurization	After 12 hrs	47 psi	270 ml/min
Before the LN_2 flow		44 psi	260 ml/min
under pressure		80 psi	660 ml/min
		100 psi	900 ml/min
After the <i>LN</i> ₂ <i>flow under</i>	Right after	100 psi	780 ml/min
pressure	After 12 hrs	50 psi	250 ml/min
		100 psi	740 ml/min

Table 4. Flow rates measured at several constant borehole pressure before and after the cryogenic stimulations.

Figure 45 illustrates photos of six specimen surfaces before and after the two cryogenic stimulations. Visual examination of specimen surfaces shows no major fracture or microcracks generated due the cryogenic stimulations. Similarly, boreholes pictures at Figure 46 indicate there is no major fracture created. Note that the microcracks cannot be inspected using photos taken from outside of the borehole.



(a) Before thermal shock followed by pressurization



(b) After *LN*₂ *flow under pressure*

Figure 45. Specimen surfaces before and after the cryogenic stimulations (Specimen #16)



(a) Before thermal shock followed by pressurization



(b) After *LN*₂ *flow under pressure*

Figure 46. Borehole before and after the cryogenic stimulations (Specimen #16)

Elastic wave data have been collected and they are to be processed soon to obtain more quantitative parameters on changes in specimen stiffness and quality. Pressure decay data are also to be analyzed. Although we have not completed the waveform comparison and pressure decay analysis, it is quite clear that the fractures were not created from the two cryogenic stimulations. There can be reasons for this in terms of specimen characteristics and in terms of cryogenic stimulation parameters as we will discuss in the next chapter. We have performed experiments for isotropic stress condition, which is the most difficult condition to fracture a specimen, and for future experiment we will vary stress parameters to observe the difference. Although we have not observed fractures created from the cryogenic stimulations, we made important observations needed to improve our experiments.

4.4 Tri-axially Stressed Transparent Cubes

Since March 2014, five cryogenic fracturing tests have been performed on transparent glass cubes. For these tests, glass cubes were prepared by carefully coring out a borehole that penetrates the cube about ³/₄ of the side length. The cube is installed in the load frame (Figure 47) and stresses applied in the ratio of 1:1.5:2 (horizontal along the borehole: horizontal perpendicular to the borehole: vertical). Two sizes of blocks have been used - 2 inch and 4 inch cubes.



Figure 47. Triaxial stress frame and preliminary liquid nitrogen delivery system.

Test G1 – 2 inch glass cube, uniaxial stress

In test G1, only a small vertical stress [190 psi vertical (z) direction] was applied. A video camera was set up, and video monitoring was performed along the borehole axis through a hole in the x-axis resistance plate. Fractures initiated at the bottom and end of the horizontal borehole, with annular fractures growing with time outward from the borehole. As the test progressed, a vertical fracture extended in the direction of maximum principal stress (vertical), finally surpassing the extent of the annular fractures. During the test, it appeared that liquid nitrogen momentarily imbibed into fractures, or that the aperture of the fractures widened and narrowed over time. The duration of this test was less than 2 minutes, and the resulting fracture pattern is shown in Figure 48 and shows that the vertical fracture parallel to the borehole propagated in the direction of maximum

principal stress, and several annular fractures with somewhat uniform spacing resulted from tension along the borehole.



Figure 48. Test G1 with stress applied in the z direction.

Test G2, 4 inch glass block – tri-axial stress

Test G2 was performed on a 4 inch glass cube with x:y:z stresses of 700:1050:1400 psi (Figure 49). The same general behavior was observed with fractures starting at the bottom of the horizontal borehole from the liquid nitrogen present there. In this case, instead of a vertical propagating fracture beneath the borehole, two fractures propagated in sub-horizontal directions. In this test, glass fracture surface temperatures were also monitored. These temperatures are directly affected by the temperature of 1) the glass block surface, and 2) the temperatures of the supporting platens. The test was run for about 20 minutes.



Figure 49. 4-inch block with annular (red arrows), vertical (white arrow), and subhorizontal (yellow arrows) fractures. Left – side view, Center – view along the borehole, Right – top view.

Tests G3 and G5-2 inch glass cubes, tri-axial stress, 1x and 4x

Test G3 was performed with the same stress distribution as Test G2. Test G5 was performed with the stresses increased by a factor of 4. The images show a clear impact of the effect of stress. The fractures in the unaixial case (left in Figure 50 and Figure 51) are clearly more extensive than those in the tri-axially-stressed cube (center in Figure 50 and Figure 51), while the fractures in the 4x tri-axial case (right in Figure 50 D and Figure 51) are far less extensive. This is because to fracture a stressed cubed, the tension generated from the application of the cryogen must first overcome the compressive stresses, then overcome the tensile strength of the glass and the increased stresses provide a larger barrier to fracturing.



Figure 50. Views along the boreholes of cryogenically fractured 2-inch glass blocks. Left – uniaxial vertical stress, Center – tri-axial 700:1050, 1400 psi, Right – tri-axial 2800:4200:5600 psi



Figure 51. View perpendicular to the borehole of cryogenically fractured 2-inch glass blocks. Left – uniaxial vertical stress, Center – Tri-axial 700:1050:1400 psi, Right – tri-axial 2800:4200:5600 psi.

Two things should be noted about the high stress case. The first is that the fractures observed in Figure 50 and Figure 51 do not show the extent of fracturing due to fracture closing. For reference, see the final image collected in Test G5 (Figure 52).



Figure 52. Final image from Test G5 after 12 minutes.

The other thing to note is the progression of fracture opening upon removal of the stress. Fifty minutes after the completion of cryogen addition, stress was removed from the block in a stepwise manner. Fractures that had appeared to close while the block warmed under stress opened again when stress was removed (Figure 53). Some of these fractures partially closed as the block fully returned to room temperature and that is the condition shown in Figure 50 and Figure 51.



Figure 53. Images collected of visibly observed fractures upon stress removal. Stress removal occurred 50 minutes after the application of cryogen was completed. From left to right, top to bottom - fully stressed, three-quarters stressed, one-half stressed, one-quarter stressed, and unstressed.

Test G4 – 2 inch glass cube under tri-axial stress, vertical borehole

In this test the maximum principal stress was oriented parallel to the borehole whereas in the other tests, where the maximum principal stress was oriented perpendicular to the borehole. Because of the orientation of the stresses in this test, annular fractures were suppressed as was expected, and vertical fractures propagated outward from the borehole (Figure 54). As in Test G2, three major fractures propagated, with the orientation of the major fractures either propagating in the direction of the maximum principal stress, or sub-perpendicular to it. Further analysis is required to understand this phenomenon.



Figure 54. Fractures in vertical borehole with annular fractures suppressed.

4.5 Tri-axially Stressed Mancos Shale

Three cubes of Mancos shale (4 inches on a side) were procured from Kocurek Industries (Caldwell, TX). Initial CT scanning showed numerous natural fractures in the blocks, and that the blocks had an epoxy-like material at the outer reaches of some of the larger fractures (Figure 55). As with the glass blocks, one-quarter inch boreholes were placed about three-quarters of the way through the block, using tap water as a cutting fluid. Two of the blocks were then painted with epoxy and epoxy was forced into the borehole. After the epoxy set, the borehole was drilled out. This was done to contain the liquid nitrogen to be injected in the fracturing test in the borehole. The blocks were CT scanned again in a cylindrical holder surrounded by sand to decrease artifacts, and a fiberscope (Figure 56) was run through the blocks were placed in the tri-axial load frame and tri-axial stress was applied (first two tests 700 psi horizontal parallel to the borehole, 1050 psi horizontal perpendicular to the borehole, and 1400 psi vertical), and tests similar to the glass block tests were performed. Temperatures of the block sides were recorded over time. Unlike the glass blocks, there was no direct indication of fracturing during the tests.



Figure 55. Initial scans of one of the Mancos shale blocks (typical). Left – image adjusted to show fractures. Right – Image adjusted to show epoxy (see arrows). Note "X"-like artifacts resulting from block geometry.

Post-test analysis included CT scanning (Figure 57) and borehole inspection using the fiberscope. Techniques such as pre- and post-test flow resistance (indicative of permeability changes) and pre- and post-test acoustics (indicative of changes in the mechanical structure of the blocks) were not performed because of the presence of large fractures at the start of the test. These fractures in the block intersecting the borehole would already have high permeability, and would strongly affect acoustic signals. Post-test CT and borehole imaging did not show a significant change in the nature of the blocks. This neither confirms nor denies the occurrence of cryogenically induced fractures however. Additional methods are being devised to allow conclusive determination of the presence and effects of the cryogenically formed fractures.



Figure 56. Shale sample borehole inspection and video recording using a fiberscope.



Figure 57. Post-test CT scan cross-section. The borehole is black. The cross-cutting fracture (wavy black feature) was also present at the start of the test.

5. Challenges and Issues

Quantifying the effects of cryogenic fracturing in opaque, heterogeneous, anisotropic media has been challenging. Many of the fractures formed in the glass blocks are below the resolution of the medical CT scanners, so it is expected that fractures in shale might be below the resolution of the CT scanner as well. A number of techniques are available to be attempted. Conventionally, a dense enhancing agent can be introduced, typically dissolved in fluid. Our cryogen limits our choices for injection during the test. A possible solution is to place a dense powder, such as barium chloride in the borehole, as it may be dragged into fractures as liquid nitrogen may be imbibed. CT scanning would show where the dense powder was placed by the flowing cryogen. A second technique would be to inject dye "A" into the borehole at low pressure some time before the test, and dye "B" following the test, using destructive testing to look for new regions stained by dye "B", guided by fracture patterns observed in glass blocks. Other density contrasting materials are also available and they are under consideration.

One of the differences in properties between artificial specimens and in-situ rocks is a stress history. Concrete specimens have never been pressurized before. Therefore, elastic strain will be created when compressed and the specimen will expand upon pressure release. In-situ rock blocks, however, are under compression over geologic times, and they would not expand immediately. Our specimens were loaded to 2000 psi, which generate significant volumetric strain depending on the materials' bulk modulus. Thermal tensile strain generated by the cryogenic stimulation must overcome this compressional strain to reach the threshold for a tensile failure. In other words, the thermal strain must be bigger than the strain generated by the volumetric compression due to loadings plus tensile strain at materials' tensile strength to achieve any fracture. Calculation for volumetric stress and strain can be done and compared with thermal stress and strain created due to the cryogenic stimulation. Note that stress-strain relations of concrete/rock may change at lower temperature, which is another research that should be performed to more precisely perform the calculation.

Our current LN_2 vessel's storing volume 0.95 liter is too small for reliable pressurized LN_2 flow, and too inefficient as it takes a significant time to fill; during filling, much nitrogen was lost as gas before the vessel was filled. In order to make the pressurized LN_2 stimulation scheme feasible, a larger capacity vessel preferably more than 20 liters may be needed, and a vacuum-jacketed wall can be a good insulator for the efficient filling of the LN_2 vessel. While high pressure cryogenic vessel is not common, pressure rating for over 1000 psi may be sought for our application.

From the concrete drying test, at least 14% of weight was lost due to heated drying test for 100% cement block (Figure 58a). Little rebound observed after putting the specimen

#2 out of oven. For concrete block made of cement and sand, it lost 5% of its weight after 16 days and but did not plateau (Figure 58b). The effect of these water in the concrete on the specimen freezing has not been systematically studied before. We will investigate the effect of concrete bound water on cryogenic fracturing to reliably interpret laboratory study using concrete specimens.



(a) Non-convection drying at 188°F - Specimen #2 (mix design: W/C=0.4, no sand; cured in atmosphere for 5 months)



(b) Convection drying at 150°F - Specimen #10 (mix design: W/C=0.55, S/C=2.5; cured underwater for 1 year; fully air dried before the drying test)

Figure 58. Concrete drying over time

6. Conclusions and Future Work

Conclusions

Since the simulation tool is being developed, the results presented are only preliminary. So far the simulation tool has the ability to couple the flow process, phase behavior and geomechanics by using the in-house thermal-hydrological-mechanical simulator TOUGH2. The modified modules for this simulation tool include phase behavior of nitrogen at low temperature, the failure criterion of the matrix, and the properties of failed matrix grids. The current failure criterion, which is Mogi - Coulomb Criterion, was derived based on mechanical tests. It may not be able to reflect the entire nature of cryogenic fracturing process. The parameters, like thermal diffusivity and coefficients for Mogi - Coulomb Criterion, are also need to be validated.

Cryogenic fracturing in glass blocks was visually observed under several tri-axially stressed conditions. Fractures occurred perpendicular to the borehole propagating radially from the borehole, as well as parallel to the borehole. These tests indicate that the stress state is very important in determining the extent of the cryogenic fractures. Under low uniaxial stress, annular fractures extended nearly to the end of the planar fracture parallel to the borehole that formed in the direction of the maximum principal stress. Increasing stress reduced the extent of the fracturing, and at the highest stress tested, fracturing was hindered with only small annular fractures forming. Fractures in the glass blocks are very easy to detect and follow. Those in shale blocks are difficult to detect. Our attempts at CT scanning the blocks without the use of a contrast enhancing agent have not detected any new fractures. This indicates that either there are no fractures, or that the method requires improvement.

We built a laboratory system for cryogenic fracturing study under tri-axial loading conditions, which mainly consist of a large tri-axial loading system and liquid nitrogen delivery and other control / measurement system. It allows cryogenic fracturing studies under various tri-axial loading conditions, and different cryogenic fracturing schemes – with and without borehole gas pressure, and pressurized and unpressurized liquid nitrogen flow along borehole. Our large tri-axial loading (TX) system can load up to 4500 psi in x and y axes, and 6000 psi in z axis on $8"\times8"\times8"$ block, independently control loadings in the three axes, and keep constant pressure to specimens. The removable vertical loading frame provides a user with space to work on specimens and activities inside the containment. The cryogenic fracturing system under TX condition has liquid nitrogen injection, GN_2 / LN_2 pressurization, and measurement/data acquisition systems.

Initial data were gathered from the newly developed system under an isotropic stress condition 2000 psi specimen stress. In *thermal shock followed by pressurization*, high pressurization following thermal shock create undesirable rapid temperature increment

due to discontinued LN_2 supply and adiabatic heating. In LN_2 flowing under pressure, liquid nitrogen flow through borehole under higher pressure decreases temperature more quickly than lower-pressure LN_2 flow due to more suppressed surface vapor cushion under pressure, and also probably expansion of some gas phase in liquid phase near the borehole outlet. A reasonable flow rate of cryogen is required to cool the borehole, by flowing either by force circulation back to the surface or by fracturing. Data indicate fracture did not initiated in this high isotropic stress, which is the most difficult stress condition The decrease of flow rate is observed probably due to moist/moisture gathered at the surface or by cryogen-induced shrinkage and resulted lowered permeability.

Future Work

The simulation tool will continue to be improved with refined physical model and parameters. The process of fracturing is still not very completed and need more correction for the failure criterion and validation by comparing to experiments. The heat transfer model also need to be characterized with more realistic parameters measured from the samples at multiple temperatures. The predictions on the fracture distribution will be compared to the permeability measurements made in the tri-axial loading / fracturing equipment.

Continued testing on triaxially stressed transparent brittle glass, and more ductile plastic cubes is planned to better understand fundamental principles of cryogenic fracturing. These tests will include the effect of changing the initial block temperature. Cryogenic fracturing tests on triaxially stressed shale blocks, and sandstone blocks with varying water saturation will be performed to better understand cryogenic fracturing in shales, and the effect of water freezing in the rock. Better methods of fracture detection will be developed to quantify fracture changes in the tested specimen.

We are in the early stages of using the new cryogenic fracturing system equipped with TX system, and will incorporate the observations made in our preliminary experiments into future work. We will vary level of confining stress and anisotropic stress condition. We will also do parametric studies on gas and liquid nitrogen pressure. The gas nitrogen pressure 450 psi that we applied right after thermal shock may be too small; we will apply higher pressure (up to 1000psi~2000psi depending on confining stresses) to see how it behave in *thermal shock and pressurization*. In *LN*₂ *flow under pressure*, we will apply LN₂ flow under varying pressure and varying duration. In order to do this, a larger vacuum jacketed liquid nitrogen vessel that can store more than 20 liters and resist more than 1000 psi will be needed. Also as discussed in discussed in Challenges and Issues, the effect of some of specimen properties, especially strain accumulated due to loading and bound water in specimens on cryogenic fracturing will be studied. The stress-strain

behavior of specimens, especially those from target formations – shale and / or sandstones samples – at cryogenic temperature will be investigated.

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APPENDIX A: Presentations / Posters / Publications

In the last year, we have prepared the following poster, presentations, and publications

- Patterson, T.W. The Effects of Cryogen Fluid on Reservoir Rocks. Presented at the Conference on Earth & Energy Research. February 28, 2014. Golden, Colorado.
- Cha, M., Yin, X., Kneafsey, T., Johanson, B., Alqahtani, N., and Miskimins, J., Patterson, T., and Wu, Y.S. (2014) "Cryogenic fracturing for reservoir stimulation – Laboratory studies" Journal of Petroleum Science and Engineering. DOI: 10.1016/j.petrol.2014.09.003.
- Cha, M., Yin, X., Patterson, T., Alqahtani, N., Kneafsey, T., Wu, Y.S., and Yao, B. (2014) "Studying Cryogenic Fracturing Process and Fracture Morphology using Transparent Specimens" Journal of Petroleum Science and Engineering (Under Review)
- Cha, M., Yin, X., Kneafsey, T., Wu, Y.S., Alqahtani, N., Patterson, T., Yao, B., and Miskimins, J. Studying Cryogenic Fracturing Process and Fracture Morphology using Transparent Specimens, to be presented at the 2014 AGU Fall Meeting, San Francisco
- Kneafsey, T.J., S. Nakagawa, Y. Wu, S. Mukhopadhyay, Laboratory Visualization Experiments of Temperature-induced Fractures Around a Borehole (Cryogenic Fracturing) in Shale and Analogue Rock Samples, to be presented at the 2014 AGU Fall Meeting, San Francisco

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- Seiji Nakagawa
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APPENDIX C: Cha et al., JPSE, 2014.

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Cryogenic fracturing for reservoir stimulation – Laboratory studies

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ABSTRACT

While hydraulic fracturing has revolutionized hydrocarbon production from unconventional resources, waterless or reduced-water fracturing technologies have been actively sought due to concerns arising from the heavy use of water. This study investigates the feasibility of fracture stimulation by using cryogenic fluids to create a strong thermal gradient generating local tensile stress in the rocks surrounding a borehole. Cracks form when the tensile stress exceeds the material's tensile strength. This mechanism has not been exploited in the context of stimulation and may be used to fracture reservoir rocks to reduce or eliminate water usage. This paper reports initial results from a laboratory study of cryogenic fracturing. In particular, we have developed experimental setups and procedures to conduct cryogenic fracturing tests with and without confining stress, with integrated cryogen transport, measurements, and fracture characterization. Borehole pressure, liquid nitrogen, and temperature can be monitored continuously. Acoustic signals are used to characterize fractures before and after the experiments. Cryogenic tests conducted in the absence of the confining stress were able to create cracks in the experimental blocks and alter rock properties. Fractures were created by generating a strong thermal gradient in a concrete block semi-submerged in liquid nitrogen. Increasing the number of cryogenic stimulations enhanced fracturing by both creating new cracks as well as widening the existing cracks. By comparing the cryogenic fracturing results from unstressed weak concrete and sandstone, we found that the generation of fractures is dependent on the material properties. Water in the formation expands as it freezes and plays a competing role during cryogenic cooling with rock contraction, thus is an unfavorable factor. A rapid cooling rate is desired to achieve high thermal gradient.

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1. Introduction

Cryogenic fracturing is a concept that looks to expand and improve on traditional hydraulic fracturing technology. The concept of cryogenic fracturing rests on the idea that a large thermal gradient, perhaps caused by the evaporation of a liquified gas can induce fractures when brought into contact with a much warmer rock under downhole conditions. When liquid nitrogen (LN_2) is injected into a rock at warm reservoir temperature, heat from the rock will quickly transfer to the liquid nitrogen resulting in rapid cooling. This rapid cooling will cause the surface of the rock to contract and may fail when tension is sufficiently built up, thus creating fractures orthogonal to the contact surface of the cryogen and the rock. These newly induced fractures can be further developed using higher pressure gas generated by evaporating

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http://dx.doi.org/10.1016/j.petrol.2014.09.003 0920-4105/© 2014 Elsevier B.V. All rights reserved. liquid nitrogen. Note that nitrogen has a liquid to gas expansion ratio of 1:694 at 20 $^\circ C$ (68 $^\circ F)$ and atmospheric pressure.

The modern hydraulic fracturing industry relies on water-based fluids due to the general availability and low cost of water; however, this dependence upon water presents several major shortcomings. First, water can cause significant formation damage, such as clay swelling and relative permeability effects stemming from capillary fluid retention (Mazza, 1997). Secondly, water usage in large quantities may place significant stresses upon the local water supply and environments where fracturing activities occur. Finally, the downhole injection of chemicals needed in waterbased fracturing programs, including slickwater and gel-based fracturing treatments has led to a contentious political climate. In contrast to hydraulic fracturing, cryogenic fracturing offers potentially greater fracturing capabilities without the issues associated with water based fracturing fluids.

King (1983) examined the use of gelled liquid carbon dioxide, instead of water, to stimulate tight gas sand formations. After performing the cryogenic fracturing, the carbon dioxide would
evaporate and not cause swelling near the wellbore in water sensitive formations. Since the gelled carbon dioxide that King (1983) used was capable of carrying proppant due to its higher viscosity than pure CO_2 , the fractures were able to stay open. Accordingly, all the wells for which he published results experienced increased production rates (King, 1983).

Grundmann et al. (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. 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.

McDaniel et al. (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. The researchers found that repeated exposure cycles to the cryogen caused the coal to break into smaller and smaller pieces, or become rubblized. After 3 cycles of exposing the coal to liquid nitrogen and allowing the coal to warm to ambient temperatures again, the coal was reduced to grain size particles. McDaniel et al. (1997) also conducted field 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.

Cryogenic nitrogen and carbon dioxide lack significant viscosity (Rudenko and Schubnikow, 1968; Fenghour et al., 1998) and may therefore inadequately carry proppant if viscosity serves as the primary transport mechanism. Gupta and Bobier (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 velocity of the fluid allowing for adequate transport of the proppant. 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 and Bobier, 1998).

Some research has even shown that cryogenic fracturing may not rely as extensively upon proppant as does traditional hydraulic fracturing. The McDaniel et al. (1997) research, which demonstrated coal rubblization in laboratory experiments, suggests 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.

If neither traditional proppants nor self-propping mechanisms 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 et al., 2005). Kendrick et al. (2005) 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.

Along with the possibility of drastically reducing water usage, cryogenic fracturing is a stimulation method that needs to be further studied to fully understand the opportunity it offers in the oil and gas industry. Thus, in this study, we conducted laboratory experiments using cryogenic stimulation to observe whether it can create fractures. We developed laboratory setups and procedures to simulate cryogenic stimulations in wellbore conditions. Liquid nitrogen was flowed through boreholes in unconfined rock blocks. Experiments were thoroughly monitored to measure physical parameters to understand the fundamental physical phenomena associated with cryogenic fracturing. Fracture development was characterized using acoustic signatures as well as visual and X-ray computed tomography (CT) inspection. We discuss expected issues in the field, and identify further studies relevant to confined conditions and field scale study.

2. Experimental setup

In this section, we present the experimental setup for two submersion tests and then for two stimulation tests. We consider two cryogenic stimulation plans: thermal shock and thermal shock combined with borehole pressurization. Fracturing by thermal shock depends purely on thermal gradient around the borehole and the resulting tensile fracturing. In the fracturing by thermal shock and pressurization scenario, fractures initially generated by thermal shock are enhanced by borehole pressurization causing the fractures to widen and propagate farther. Two geometries have been investigated – cooling in boreholes in cubic blocks, and partial or total submersion of cubic blocks in liquid nitrogen.

2.1. Block semi-submersion tests

As a preliminary study, the effect of the thermal gradient on rock behavior was investigated. To apply a strong constant temperature gradient across a concrete block, an 8 in. cubic concrete block was set on supports in an open-top insulating enclosure. The enclosure was filled with liquid nitrogen up to the midline of the concrete block (Fig. 1). The liquid nitrogen level was maintained for 30 min and then allowed to boil off. The block was not removed until it equilibrated thermally with ambient temperature. Each side was photographed before and after the test. The images were aligned and digitally subtracted to highlight the differences.

2.2. Water-saturated block submersion test

When water becomes ice, the volume increases by 9%, which is much larger than any kind of thermally-induced expansion or shrinkage in geomaterials. To investigate potential consequences of this, the behavior of water-saturated rock under cryogenic temperature was also investigated by submersion test. In this test, the concrete sample was prepared with a water to cement ratio of 0.55, and sand to cement ratio of 2.5. The cement was injected into an $8'' \times 8'' \times 8''$ mold and sealed in a plastic bag. After 24 h, the seal and mold were removed and the concrete was cured under water



Fig. 1. Setup for semi-submersion test.

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Fig. 2. LN₂ submersion test: (a) the water-saturated concrete cube placed inside a Styrofoam container and (b) violent LN₂ film boiling at the interface between LN₂ and the block surfaces.



Fig. 3. Overall schematic for borehole cryogenic stimulation experiments (for both thermal shock and thermal shock followed by pressurization).

for 8 weeks per ASTM C192, which will maximize hydration and render the concrete very strong. The block was never removed from the water until testing, and had no visible cracks before the stimulation. In this submersion test the block was fully submerged, as shown in Fig. 2.

2.3. Borehole thermal shock

In this test, we cooled the borehole as rapidly as possible to maximize the thermal gradient. This was done by flowing liquid nitrogen (LN_2) continuously through the borehole. The basic scheme is illustrated in Fig. 3 (the left and center part of the setup). LN_2 was pumped from the Dewar by pressure difference using a liquid nitrogen withdrawal device. LN_2 was transported through a vacuum-jacketed hose to the specimen, injected into the borehole, and then directed to an outlet. A pressure transducer was attached to monitor the borehole pressure. Because this scheme does not generate much pressure, the experiment can be applied to both confined and unconfined specimens. The experimental equipment employed cryogenic-rated transport, control, and measurement systems. Parameters including pressure inside the borehole, LN_2 consumption, temperature at multiple locations, and acoustic signals were monitored in real-time. A structure that

confines a packer in place was built to prevent leakage through the packer and sustain higher pressure in the borehole (Fig. 4).

2.4. Thermal shock combined by borehole pressurization

Borehole pressurization will enhance the fractures created by the thermal shock by applying a level of pressure to the borehole during and/or after the thermal shock. One scheme of pressurization can be performed by letting existing LN_2 in the borehole evaporate while shutting off all of the inlet and outlet valves. Another is by forcing pressurized nitrogen gas into the borehole (right side of Fig. 3). While high borehole pressurization is possible for confined specimens confined by triaxial loading equipment, unconfined specimens cannot sustain much borehole pressure (rock splitting was observed in weak concrete due to pressurization).

2.5. Packer placement

Packers were used to accommodate inlet and outlet tubing and seal the borehole from the outside. At low borehole pressure in unconfined specimens, we attached a packer to the top of a borehole with epoxy and then applied loading using the confining device, depending on the expected level of pressure inside the

borehole (Fig. 4). Springs located on the bolts were used to accommodate specimen contraction due to thermal contraction while still applying almost the same stress on the packer. This device was used for our tests with unconfined specimens.

3. Experimental results

3.1. Semi-submersion experiment

In the first submersion experiments, the concrete block was semi-submerged to induce a strong temperature gradient along the LN_2 -GN₂ contact line (Fig. 1). Fig. 5 shows the results from



Fig. 4. Packer installation by using a confining structure.

image subtraction before and after the cryogenic treatment. The difference image for the top and bottom (Faces 5 and 6) do not show any fractures. The four vertical faces (Faces 1 through 4) that were semi-submerged show a fracture along the center all the way around the block (light shaded crack). This indicates that the block was fractured due to the application of the thermal gradient. During the 30+ minute test, there were no obvious indications of block fracturing such as large visible cracks or audible cracking sounds. Following the test, the block was CT scanned using a modified G.E. Lightspeed 16 medical CT scanner. Fig. 6 shows a vertical cross section indicating the presence of a fracture (darker) emanating from both sides and progressing towards the center of the block.



Fig. 6. CT scan showing a vertical slice through the middle of the block from the semi-submersion test. Arrows point to (darker) fractures in the block resulting from cryogenic fracturing.



Fig. 5. Subtracted before and after images of block sides in semi-submersion test. Face numbers are labeled. Note the light colored fracture along the midline of faces 1, 2, 3, and 4.



Fig. 7. Specimen after thermal shock – red lines are superimposed along the cracks to improve visibility. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



Fig. 8. Cracks created by the expansion of ice matrix (Face 4).

3.2. Full submersion test of a water-saturated cement sample

In the full submersion experiment conducted with the watersaturated cement sample (Fig. 2), all major cracks were created near edges during the cryogenic stimulation (Fig. 7). In this case, the ice, which is heavily interconnected through pores, was expanding against mineral (cement and sand) matrix. Thus the outer layer of the block exposed to LN_2 experienced water freezing and expanding, whereas the inner block did not. The outer layer expanded laterally, resulting in shear fractures parallel to the exposed surfaces (Fig. 8) (Kneafsey et al., 2011). If cracks were formed from rock contraction (without the effect of ice), more cracks should be located away from and perpendicular to the edges. The block bottom was in direct contact with the Styrofoam container limiting LN access resulting in the absence of cracks there. Simple calculations may help explain the competition between expansion of the ice matrix and contraction of the mineral (cement) network. The volumetric thermal expansion coefficient of concrete γ (at 20 °C) is 36×10^{-6} /K. Assuming γ is constant throughout the temperature range, the volume reduction due to a cryogenic temperature change would be

Volume reduction (%) = 200 K × 36×10^{-6} /K × 100 (%) = 0.72%

Upon phase change from water to ice, the volume increases by 9%. Assuming the porosity of concrete is 20%, the overall resulting volume change becomes

Overall volume change (%) = $(9\%) \times 0.2-(0.72\%)$ $\times 0.8 = 1.2\%$ (expansion)

There may be other complicating factors such as movement of water molecules within the block and outside during the freezing process. However, the movement of water to the outside will be negligible because the surface water will be immediately frozen upon LN_2 submersion.

3.3. Borehole cryogenic stimulation of unconfined specimens

Two $8 \times 8 \times 8$ in.³ block specimens were cryogenically stimulated by flowing LN₂ through a borehole made into the specimen. A summary of these tests is listed in Table 1. One concrete block and one sandstone block were used in these tests. Measurements included pressure, temperature, acoustic waves, LN₂ consumption, and CT scanning. For all cases, specimens were photographed before and after cryogenic stimulations.

3.3.1. Thermal shock on the concrete specimen

Two thermal shock experiments were performed on a concrete specimen without pressurization. The cement concrete block was air-cured for 5 weeks after being contained in the mold for 24 h, resulting in relatively low strength. Fig. 9 shows the experimental setup that was used for the thermal shock experiment on concrete. LN_2 was flowed from the Dewar to the specimen borehole through

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Table 1

List of cryogenic stimulation experiments.

Specimen	Cryogenic stimulation	Measurements	Cryogen (amount)	Duration (min)
Cement concrete	1st Thermal shock (unconfined packer)	<i>T</i> , acoustic (before, during, after)	LN_2 (N/A)	52
	2nd Thermal shock	T, P, acoustic (before, during, after), LN ₂ weight	LN ₂ (7 kg)	21
Sandstone	1st Thermal shock	T, P, acoustic (before), LN_2 weight	GN ₂ (12.8 kg)	57
	2nd Thermal shock	T, P, LN_2 weight	GN ₂ (14 kg)	73
	3rd Thermal shock	T, P, acoustic (after), LN_2 weight	LN ₂ (15.4 kg)	70
	4th Thermal shock+pressurizations	T, P, acoustic (after), LN ₂ weight	LN ₂ (13.8 kg)	160

Note that T, P, LN₂ and GN₂ represent temperature, pressure, liquid nitrogen, and gaseous nitrogen respectively.



Fig. 9. Experimental setup with a "packer-confining structure".





the vacuum jacketed tube and insulated stainless steel tubes. The borehole was open to the air through a vent and relatively warm N_2 flowed out to the atmosphere. There was no significant pressure buildup inside the borehole because N_2 flowed freely into the atmosphere. In this 1st test, the packer was not confined and loosely fastened to top of the borehole, so LN_2 leaked through packer/concrete interface and out of the insulation, making the thermal shock less effective.

The 2nd test was performed on the same concrete block that was used for the 1st test using the structure shown in Figs. 4 and 9, designed to withstand borehole pressure up to 500 psia. For this experiment, we only used a low pressure caused by vaporization (\sim 10 psi). A pressure transducer was attached to monitor the borehole pressure, and a scale was used to record LN₂ consumption. A set of S-wave acoustic sensors were mounted at locations on Faces 2 and 4 to monitor the wave signatures. A pulser and an oscilloscope were used to generate and record signals.

We observed that devices and materials in the setup performed well through the testing. There were no leaks through the packer/rock interface. The epoxy seal and stainless steel packer with tubes connected all remained intact under the cryogenic temperature conditions. The cryogen-rated insulation and the thermocouple's plastic insulation coat remained functional. The carbon steel plates performed at low temperatures (~ -30 °C). One reason why these were undamaged is that, unlike the borehole environment, temperature propagation was slower in the surrounding environment, preventing sharp thermal gradient. Another reason could be that the small volume of the material does not create enough contraction to fail the materials and/or interfaces. The block surfaces started to form frost once the temperature of the surface fell well below the water-freezing point. The frost was first noticed at the top and sides of the specimen, which are closer to the borehole.

3.3.1.1. Cracks. Before the 1st thermal shock, there were no visible cracks near or inside the borehole. After the thermal shocks, several noticeable cracks were found. Major cracks were visible from the top of the borehole (Fig. 10). There were some preexisting micro-cracks on the block surfaces before the thermal shock due to the natural shrinkage of concrete (Fig. 11a). Note that the dark spots are stains from the coupling material used to facilitate the transmission of acoustic energy. After the thermal shock, new cracks were generated and even visible on the surfaces, and the existing cracks became wider (see Fig. 11 for comparison).

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Fig. 11. Surface cracks. (a) Pre-existing surface cracks. (b) Surface cracks after the 1st thermal shock (c) after the 2nd thermal shock – the superimposed lines are weighted according to the crack thickness. Note that the darker spots at Fig. 11b are stains from the coupling material used to attach the ultrasonic sensors.

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Fig. 12. The CT slices at 0.4", 1", and 2" away from Face 4 (vertical side) and Face 6 (Bottom). Cracks appear as darker features on the images.

Particularly, there was a relatively small number of cracks at the bottom before applying the cryogen; however, many new cracks were created after the cryogen applications.

During the 2nd thermal shock, significant gas and liquid nitrogen permeation was observed through fractures that might have been opened up at the later stage of the experiment. The amount of leakage was dependent on the release rate from the dewar.

The specimen was scanned using an X-ray CT (Toshiba Aquilion 64). The maximum resolution of the images from the scanner is 0.3 mm \times 0.3 mm \times 0.3 mm. Thus, the CT images can only be used to detect major cracks. The CT images show that there are more fractures near the outer surfaces than the inside of the specimen. X-ray slices (a few examples are given in Fig. 12) show that cracks distributed near the exposed surfaces diminish as they move toward the inside of the specimen. There are also a few internal cracks that are not connected to any of the exposed surfaces.

3.3.1.2. Temperature, pressure, and liquid nitrogen consumption. Temperature was measured at seven locations in the specimen and the setup using thermocouples (TC) (Fig. 13). TC #2 (in black) was suspended inside the borehole, while TC #1 was attached to the borehole wall surface. Temperatures at the carbon steel plate (TC #4) and near the pressure transducer (TC #3) were monitored to protect the plates and the sensor. The temperature evolution during the experiment at the seven locations is plotted in Fig. 14c. Throughout the test, the temperature difference between the inside of the borehole and the block face was observed to be large. It is also observed that cooling at the borehole surface was slower due to a phenomenon called the Leidenfrost effect. Due to a very large temperature difference between the rock surface and the boiling point of the fluid, the liquid nitrogen immediately boils at near contact with the surface, creating a vapor cushion, which has a much lower thermal conductivity than the LN₂, thus delaying the heat transfer to the rock surface. Due to leakage of GN₂ and LN₂ through the created fractures, we had to from time to time partially close and then open the dewar valve to control the leaking. The spikes in the plots are where the LN₂ valve was closed temporarily. The temperature inside the borehole was sensitive to valve operations and the temperature increased rapidly when the LN₂ supply was stopped (Fig. 14c).

The amount of LN_2 leaving the dewar was monitored using a scale (Fig. 14a). The nonlinear LN_2 consumption vs. time before the first partial closure shows that strong vaporization occurred at the beginning, which blocked the flow of LN_2 . Then as the system cooled down, the flow rate of LN_2 increased. Pressure was generated due to vaporization in the borehole and along transport lines. In the thermal shock test setup, pressure inside the borehole was practically the same as the pressure inside the dewar (about 5–10 psi). The changes in pressure were very responsive to the opening and partial closing of the valve of the dewar (Fig. 14b).

3.3.1.3. Acoustic signatures. The characteristics of acoustic waves propagating through the medium depend on the mechanical properties of the medium. In particular, the wave velocity in jointed rock masses is a function of the density of cracks (or joint spacing) and thus has been modeled (Cha et al., 2009) as follows:

$$V_{S} = \sqrt{\frac{G_{eq}}{\rho}} = \sqrt{\frac{1}{\rho} \left(\frac{k_{S}SG}{k_{S}S+G}\right)}$$
(1)

where V_S , k_S , G, ρ , and S are shear wave velocity, shear joint stiffness, shear modulus of intact part, rock mass density, and joint spacing respectively. This equally applies to the (compressional) P-wave velocity. When other properties such as intact rock properties, density, and joint stiffness are the same, the wave velocity can be used as a monitoring tool to characterize crack generation. Geomaterials are natural low-pass filters of acoustic waves due to inherent discontinuities such as granular discreteness and cracks. Acoustic waves propagating through cracked media have their high frequency content filtered.

The differences in the acoustic signatures before and after the cryogenic stimulation were investigated by acoustic measurements before and after the tests using P and S ultrasonic transducers. Acoustic waves were also monitored during the cryogenic stimulation by mounting S transducers to the specimen surfaces. Fig. 15 shows the locations for the acoustic measurements before and after the cryogen stimulation. Acoustic signals were measured between Faces 1&3 and 2&4 (faces opposite each other). For each face set, the acoustic measurements were conducted at the 12 locations. In this work, we focus on P and S wave velocities and amplitudes. P-wave signals between Faces 1 and 3 are shown in Fig. 16.

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Fig. 13. Locations of the temperature sensors.



Fig. 14. Synchronized plots of $\ensuremath{\text{LN}}_2$ consumption, pressure, and temperature with time (the 2nd thermal shock).

In Fig. 16a, early parts of elastic wave signals are presented with their amplitudes normalized so we can focus on changes in the arrival time and in the waveforms. At most measurement locations, arrivals were delayed after cryogenic stimulation, which means that the wave velocity decreased as a result of the cryogenic stimulation, and waveforms have changed. The characteristic of acoustic signatures approximately corresponds to density of the surface cracks. For example, the acoustic signals at location 12 were the least changed in terms of the arrival time, and we also observed that the surface around location 12 was the least cracked. In Fig. 16b, the signals show early times near the arrivals,

but the original amplitudes were kept so that one can observe the changes in the amplitudes. Wave amplitudes reduced significantly after the thermal shock (except for Location 12). Signals compared with their full range of the measurements show that the global frequency of the signals became lower after completing the cryogenic fracturing at most locations.

The changes in P-wave velocities are summarized in Fig. 17. Lower velocities near the edges (#1 and 8) and center (#4 and 5) show the effects of boundaries and borehole cavity. The wave velocities decreased by 7–13% due to the application of thermal shocks.

3.3.2. Cryogenic stimulations on the sandstone specimen

Cryogenic stimulation experiments were performed on a sandstone specimen obtained from a quarry in Denver Colorado. Multiple tests were conducted due to inefficient thermal shock (the first two instances) and more resistance of the sandstone to thermal shock than the previous concrete specimen. The list of thermal stimulations and conditions applied on the sandstone specimen is documented in Table 1. Note that in the 1st and 2nd thermal shocks, cold nitrogen gas was released instead of LN₂ due to a malfunction of the withdrawal device. This problem was remedied prior to the 3rd thermal shock. Pressurization was applied to the specimen during the 4th test in addition to thermal shocks. Because there were no apparent fractures and no leaking of GN₂ and LN₂ through the fractures, the dewar valve was opened fully throughout the experiment in all tests.

3.3.2.1. First three thermal shocks without pressurization. Due to withdrawal device malfunctions, nitrogen injection into the borehole was in the gas form for the first two experiments. This was indicated by the temperatures in the borehole, which were significantly higher than LN_2 boiling point. We did not observe any drops of LN_2 from the outlet either. The temperature data did not show the Leidenfrost effect, which is yet another indication of lacking LN_2 .

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Fig. 15. Locations of acoustic measurement before and after thermal shock.





Fig. 16. P-wave signals near arrivals between Faces 1 and 3 before and after the thermal shock. (a) Velocity changes – normalized amplitude. (b) Amplitude changes – compared with original amplitude.



Fig. 17. Changes in P-wave velocities before and after the thermal shocks.

The pressure inside the borehole basically followed a trend similar to the pressure inside the dewar, except that the pressure inside the borehole was lower by about 1 psi. In this test, the release valve to the dewar was opened completely until the end of the test without any partial or temporal closure. High-frequency fluctuations of the temperature data were also indicated by fluctuations of flow sound at the outlet, which is related to the pressure fluctuation in the borehole.

No noticeable cracks were generated after the first two thermal shocks, possibly due to slower reduction of temperature, higher final temperature, lower brittleness, and the higher strength of the sandstone block.

At the 3rd thermal shock, the LN withdrawal device was improved and liquid nitrogen was injected into the borehole. Acoustic measurements were performed after the 3rd thermal shock to monitor alterations in the acoustic wave transmission properties due to the thermal shocks. The temperature data showed more immediate temperature reduction compared with the two previous experiments as well as the Leidenfrost effect, which are indications of LN_2 presence in the borehole.

3.3.2.2. The 4th thermal shock (LN_2) and borehole pressurization. The 4th test was set up to pressurize the borehole. This was done by adding cryogenic-rated valves, a pressure relief valve, an accumulator, and a compressed nitrogen cylinder (Figs. 18 and 19). The pressure release valve was set to 275 psi so that the pressure inside the borehole would not exceed that pressure. The reason for this low set pressure was that geomaterials are generally weak under tension and cannot sustain much internal pressure when they are not confined. At the end of the test, no noticeable cracks were found on the surfaces of the block by visual inspection.

3.3.2.3. Pressure, temperature, and LN_2 consumption (4th test). After the thermal shock, two different borehole pressurizations were attempted. One was by natural vaporization of liquid nitrogen in a closed system (Fig. 20a). The other was by pressurizing the borehole using compressed nitrogen gas (Fig. 20b). It was observed that LN_2 vaporization caused the pressure to increase up to 250 psi and dropped rapidly following a pressure decay curve (Fig. 21b). This is because nitrogen (most likely at a gas state) in the borehole escaped through the sandstone at a fairly high rate by permeation. This significant permeation rate was also indicated from air permeation tests performed on the sandstone block prior to the application of thermal shocks.

The temperature inside the borehole increased rapidly as the borehole was pressurized (both forced pressurization and self-pressurization) (Fig. 21). This temperature increase occurred from gas compression and heat transfer near the borehole. While the temperature increased when the LN_2 supply discontinued, the temperature increments were further accelerated by the pressurization. Due to the lengthy duration of the experiment (75 min), even the temperatures of the surfaces of the rock became very low (as low as -50 °C) at the end of experiments. In addition to conductive heat transfer, flow of cold nitrogen gas through the sandstone also contributed to the reduction of the outer surface temperature.

3.3.2.4. Acoustic measurements. The P and S wave signatures before thermal shock, after the 3rd test, and after the 4th test are compared in Fig. 22. The arrivals of P and S waves between Faces 1 and 3 with normalized amplitude are shown in Fig. 22. The velocities changed,

but in relatively small magnitude considering the number of stimulations performed. This shows that sandstone is more resistant to cryogenic stimulations compared to the cement. This may be because the thermal gradient was lower as a result of cold gas permeating through the sandstone, or that the thermal contraction relative to the strength and ductility of the sandstone was not as great. Although we did not see visible cracks on the block surfaces and in the borehole, velocity changes show that there were cracks generated within the block perhaps indicating fractures in the inter-grain cement. Wave velocity decreased by 2.5-6% for the P-waves, and 3.5–6.3% for the S-waves after the stimulations (Figs. 23 and 24). The velocity increment due to the 4th cryogenic stimulation as the thermal shocks and pressurizations turns out to be relatively small, which indicates that the effect of the pressurization is negligible at this low pressure level and permeable sandstone. The decrease or increase of velocities toward a certain direction indicates that internal mechanical properties are directional (Figs. 23 and 24).

3.3.2.5. Bubble leakage test. We found that a leak hole/crack was generated during the 2nd pressurization (forced pressurization) at the packer/rock interface by a hissing sound as well as by the pressure data (Fig. 21b). After the test, a bubble agent was used to detect the leak. In order to do this, the borehole was pressurized to about 50 psi using compressed air. The leak hole created during the 2nd pressurization may be seen in Fig. 25a at the location of the largest foam accumulation. Then the bubble agent was applied all over the top surface and Face 1 to observe permeation pattern at the block surfaces. We observed that there are several localized permeation spots (or "leaking holes") as shown in Fig. 25. The bubble leakage test showed that permeation through the stone was not homogeneous



Fig. 19. Experimental setup near the specimen and locations of thermocouple tips (the picture was taken before insulation was applied).



Fig. 18. Experimental setup for thermal shock and borehole pressurization (protection shields not shown).

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Fig. 20. Borehole pressurization schemes.



Fig. 21. $\ensuremath{\text{LN}}_2$ consumption, borehole pressure, temperature vs. time during the stimulation.

even in a rock that appears to be intact and homogeneous; there were invisible pathways (cracks, holes, or simply more permeable regions) that allowed preferential permeation of air/fluid.

4. Leidenfrost effects and formation water related issues

Clearly, there are many challenges to overcome before LN_2 can be used effectively as a fracturing fluid, e.g. transportation of LN_2 to the target reservoirs, use of proppants with LN_2 , and high heat flux from rock masses. Here we discuss two specific issues identified from this laboratory study – the Leidenfrost effects and the issue of formation water.

4.1. Leidenfrost effect – slower cooling rate

The Leidenfrost effect happens anywhere when liquid is in near contact with a surface significantly hotter than the liquid's boiling









Fig. 22. Wave arrivals across Faces 1 and 3 before and after the cryogenic stimulation – compared with normalized amplitude. (a) P-waves. (b) S-waves.

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Fig. 23. P-wave velocity comparison. (a) Measurement across Faces 1 and 3. (b) Measurement across Faces 2 and 4.

point. It slows down heat transfer to the rock by creating a vapor cushion, which has very low thermal conductivity, and thus may inhibit a sharp thermal gradient hindering fracture propagation.

An experiment was performed to observe the Leidenfrost effect at the rock-LN₂ interface. Two Lyons sandstone blocks with roughly the same dimensions $(3'' \times 3'' \times 4'')$ were used for the test (Fig. 26a). Two temperature sensors were attached to each block to monitor temperatures as they were submerged into LN₂. Another temperature sensor was placed directly in the liquid nitrogen without touching any surfaces (Fig. 26b). Before the submersion, Block 1 was heated to 120 °C, while Block 2 was at ambient temperature. Upon submersion, LN₂ violently boiled off at the interface of the blocks and liquid nitrogen, and the temperatures at the thermocouples (Fig. 26b) indicated the presence of an insulating gas layer because they did not rapidly drop to the boiling LN_2 temperature (Fig. 27). The block with the higher temperature took a longer time to reach the temperature of LN₂ at its surface as it sustained the boiling (and Leidenfrost effect) longer (Fig. 27). In spite of the Leidenfrost effect, it is observed that the temperature dropped fairly rapidly to a certain temperature (about -120 °C in these tests) which is still significantly warmer than the normal LN₂ boiling temperature (-196 °C). Although the temperatures recorded from the thermocouples at the block surface reached the temperature of liquid nitrogen in about 7 min, LN₂ continued to boil at the interfaces with gradually decreasing magnitude.

There are potential methods to reduce the Leidenfrost phenomena. The use of slush nitrogen, which is a mixture of solid



Fig. 24. S-wave velocity comparison. (a) Measurement across Faces 1 and 3. (b) Measurement across Faces 2 and 4.

nitrogen and liquid nitrogen, is a relatively proven method to increase the cooling rate (Sansinena et al., 2012), and apparatuses for producing slush of nitrogen have been devised (Kawamura et al., 2007; Machida et al., 2009). Similarly, mixing proppants in liquid nitrogen as a solid suspension might help reduce the Leidenfrost effect, and this would be advantageous as proppant may be needed to keep the fractures open. As pressure increases, the Leidenfrost effect occurs at a relatively higher temperature (Temple-Pediani, 1969). Therefore, at higher pressures, the Leidenfrost effect will diminish earlier after LN₂ application. Temperatures of the subsurface environments where fracturing is desired need to be considered as they can be very high. The Leidenfrost effect can also be decreased by creating roughness on the surfaces (Bernardin and Mudawar, 1999).

4.2. Formation water saturation

From the full submersion test of a water-saturated cement block, we learned that water saturation is an important factor in determining the thermal expansion / contraction of the formation. Depending on the water saturation level in formations, which varies greatly, the presence of water will weaken the driving force of cryogenically induced tensile fractures. Near the well-bore, water content may be high due to prior exposures to drilling and completion fluids. Volume expansion by freezing water is likely to weaken tensile stress or even create circumferential compression depending on water saturation. In addition to being an unfavorable condition for tensile failure, the compressional

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Fig. 25. Leakage/permeation test by bubbles. (a) Localized air permeation observed at the top. (b) Local permeation at the side (Face 1).



Fig. 26. Lyons sandstone block under LN₂.



Fig. 27. Rock submersion test with temperature measurements.

stress may also lead to wellbore failure and stability issues. On the other hand, frozen water on the surface or near the surface of the wellbore may inhibit nitrogen permeation into the formation and lead to stronger temperature gradient. Thus, the presence of water in or near the wellbore is likely to create a complicated situation. We will conduct more experiments in the future to carefully study its effect.

Finally, it is worthwhile to note that in this study concrete was used as a surrogate for specimen as it is easy to mold to form desired shapes and as it is a common engineering material. However, concrete and natural rocks are different in many ways. Depending on the mix ratio and the curing quality, concrete can have much residual water after hydration that cannot be easily removed unless by high heating. Water is trapped in isolated micropores, and up to 15% of water by weight can be removed by heating (100 °C) of a fully cured air-dried 100% cement concrete (tested in our lab). In our study, as thermal shocks were applied to the borehole of the concrete specimen, more fractures were observed on the outer surfaces of the block than inside the borehole, which is counter-intuitive. It may be possible that this is caused by the phase change of residual water in the micropores upon thermal shocks, which aided the fracturing of the outer surfaces.

5. Summary and conclusions

This paper reports a laboratory study of cryogenic fracturing. We designed experimental setups and laboratory procedures for cryogenic stimulation. The ability of liquid nitrogen to create fractures was tested in submersion tests and borehole stimulation experiments, and fracture assessments were made by visual inspection, X-ray CT, and acoustic signatures. The velocities and amplitudes of acoustic waves were sensitive to rock properties thus we were able to detect changes in properties that were not visible. CT images identified spatial distribution of major cracks. Measurement of both temperature and pressure data during stimulation gave us opportunities to monitor the state of the stimulating fluid as well as to assess the efficiency of the heat transfer process.

The cryogenic stimulations conducted in our study were able to create cracks in the rock blocks and alter rock properties. Fractures were created by generating a strong thermal gradient in a concrete

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block half-submerged in LN2. Application of liquid nitrogen to boreholes in the center of cement and sandstone specimens generated mixed results. For the cement block, repeated thermal shock generated visible fractures, the signatures of which were easily picked up by the acoustic measurements. Increasing the number of liquid nitrogen applications to boreholes enhances fracturing by both creating new cracks as well as widening the existing cracks. For the sandstone block, after two thermal shocks with gaseous nitrogen and two more thermal shocks with liquid nitrogen combined with elevated pressure (one from pressure buildup due to evaporation and another from an external pressurized nitrogen source), no visible fractures were detected. Clearly, the generation of fractures was highly dependent on material properties. Results from acoustic tests, however, suggest that there were invisible fractures generated inside the sandstone block.

To maximize the cooling rate, means to alleviate the Leidenfrost effect need to be sought. Immediate LN₂ flooding is equally desired, which can be helped by efficient insulation and transportation. The freezing of water in formations plays a competing role with contraction of the rock but also forms a barrier to permeation near a wellbore; the effect of these competing factors on cryogenic fracturing should be further assessed. Although many unconventional gas formations have low water saturations, near the wellbore water saturation can be significant from prior drilling and completion operations. Borehole pressurization due to LN₂ vaporization in a closed system may not generate sufficient pressure to propagate the fractures due to the limited LN₂ quantity and rate of vaporization. Gas permeation also reduces the buildup of pressure. Thus, external pressure may still be needed to propagate the cryogenically induced fractures.

Several topics that require further studies have been identified in this laboratory study. Cryogenic fracturing at reservoir stress levels by thermal shock, and the added effect of borehole pressurization are still poorly understood. The effect of stress level and stress anisotropy on the characteristics of cryogenic fracturing can be investigated by using a triaxial loading system. The effects of various material properties on cryogenic fracturing behavior should be thoroughly investigated. A full dimensional analysis considering all the relevant parameters will serve as a frame of understanding and will guide the scaling up of laboratory studies to potential field applications.

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APPENDIX D: Cha et al., Submitted to JPSE, 2014

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Abstract: Cryogenic fracturing exploits thermal gradient and resulting local tensile stress to initiate fractures / cracks on a surface exposed to cryogenic fluids. This study investigates the development and morphology of cracks generated from cryogenic thermal shock in a borehole geometry. The study evaluates cryogenic thermal shock under no external confining stress to specimens. To better understand this process in a geometry relevant to applications, a borehole was drilled through transparent acrylic specimens representing a wellbore. This borehole was partially cased with stainless steel tubing set by a high yield epoxy. Liquid nitrogen was injected into the wellbore through a stainless steel tube. The pressure was low (< 10 psia) and the fractures were initiated by the thermal shock; these initiated fractures allowed further penetration of the cryogen, which helped to propagate fractures throughout the specimen. A major advantage of performing this experiment in a transparent cryogenic specimen is the ability to observe fracture proliferation through time. It is observed that fracture growth was characterized by abrupt starts and stops, which suggest that the tensile stress generated inside the borehole must reach a certain threshold for fracture initiation and growth. Two distinctive patterns in crack development were observed: one is horizontal-planar-radial pattern created by longitudinal thermal contraction, and another is vertical cracks by circumferential contraction. The horizontal cracks appeared to be spaced by a certain length, known as the exclusion distance, which exists because a set of cracks cannot be created closer than a certain length due to limited amount of thermal contraction. The vertical tension cracks tend to initiate between the horizontal radial cracks and bridge them, as it may be energy-efficient to start from and propagate to existing defects.

Studying Cryogenic Fracturing Process and Fracture Morphology using Transparent Specimens

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Studying Cryogenic Fracturing Process and Fracture Morphology using **Transparent Specimens** Minsu Cha¹*, Xiaolong Yin¹, Taylor Patterson¹, Naif Alqahtani¹, Timothy Kneafsey², Yu-Shu Wu¹, and Bowen Yao¹ Prepared for Journal of Petroleum Science and Engineering Components: 3200 words, 0 tables, 9 figures. Keywords: Cryogenic fracturing; thermal shock; thermal stress and strain; shale and tight gas reservoirs. ¹Petroleum Engineering Department Colorado School of Mines 1600 Arapahoe St. Golden, Colorado, 80401 ²Lawrence Berkeley National Laboratory 1 Cyclotron Road Berkeley, California, 94720 *Corresponding Author. E-mail address: mcha@mines.edu

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development and morphology of cracks generated from cryogenic thermal shock in a
borehole geometry.

The study evaluates cryogenic thermal shock under no external confining stress to specimens. To better understand this process in a geometry relevant to applications, a borehole was drilled through transparent acrylic specimens representing a wellbore. This borehole was partially cased with stainless steel tubing set by a high yield epoxy. Liquid nitrogen was injected into the wellbore through a stainless steel tube. The pressure was low (< 10 psia) and the fractures were initiated by the thermal shock; these initiated fractures allowed further penetration of the cryogen, which helped to propagate fractures throughout the specimen.

A major advantage of performing this experiment in a transparent cryogenic specimen is the ability to observe fracture proliferation through time. It is observed that fracture growth was characterized by abrupt starts and stops, which suggest that the tensile stress generated inside the borehole must reach a certain threshold for fracture initiation and growth. Two distinctive patterns in crack development were observed: one is horizontal-planar-radial pattern created by longitudinal thermal contraction, and another is vertical cracks by circumferential contraction. The horizontal cracks appeared to be spaced by a certain length, known as the exclusion distance, which exists because a set of cracks cannot be created closer than a certain length due to limited amount of thermal contraction. The vertical tension cracks tend to initiate between the horizontal radial cracks and bridge them, as it may be energy-efficient to start from and propagate to existing defects.

Introduction

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 Cryogenic fracturing is a concept that looks to expand and improve on traditional hydraulic fracturing technology. The concept of 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 rock whose temperature is drastically different, the 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 contract and fail in tension, thus inducing fractures orthogonal to the contact plane of the cryogen and rock. To further develop these newly induced fractures, liquid nitrogen has a high liquid-to-gas expansion rate which creates a high pressure environment helping to propagate the fractures.

Traditional hydraulic fracturing in low permeability formations uses a highly pressurized fracturing fluid to create a complex network of fractures and uses proppants to maintain the conductivity of the main fractures after flowback. These fractures increase the contact area between the reservoir and the wellbore, allowing more reservoir fluids to flow into the wellbore and be produced. Hydraulic fracturing together with horizontal drilling and the various technological advances associated with them have drastically changed the United States' oil and gas producing capabilities. They have revolutionized the oil and gas industry in the United States and have helped sparked energy booms in many parts of the country.

The modern hydraulic fracturing practice is dominated by water-based 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 (Mazza, 1997). These formation damage mechanisms inhibit hydrocarbon flow and thus impair production rates and recovery efficiency. Second, water used 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 significant impacts on the surrounding community. Finally, downhole injection of chemicals needed in water-based fracturing operations, including slickwater and gel, are environmentally controversial. In these regards, cryogenic fracturing have the potential to offer great benefits.

In this study, we report a laboratory study of fracture initiation within transparent specimens caused by exposure to liquid nitrogen. This paper is divided into the

following sections. In Section 2, we briefly summarize the known field and laboratory
studies on cryogenic fracturing; in Section 3, we present our experimental set up; in
Section 4, we report and discuss the experimental findings.

Review of Pertinent Literature

Liquid CO₂ can be used as fracturing fluids to reduce formation damage. It can be gelled to offer a good capacity to carry proppant. Note that the temperature of liquid CO_2 is not significantly lower than that of the formation; however, there can be strong thermal effects due to rapid evaporation. King (1983) examined the use of gelled liquid carbon dioxide to stimulate tight gas sand formations to prevent formation damage. After fracturing is completed, carbon dioxide would evaporate and mingle with production gas. It was observed that the carbon dioxide would not cause clay 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. Field test used a gelled carbon dioxide capable of carrying proppant due to its higher viscosity. As a result, the fractures were able to stay open and all the wells reported in his publication showed increased rates of production (King, 1983). Unfortunately, post fracturing production data over a long period of time past a few days was not available. Although successful field data were produced, his research did not include laboratory experiments. It is impossible to determine whether the increased well production was from thermal stresses creating fractures or from fluid pressure creating the fractures.

Another fracturing technology that has recently been explored is using liquefied petroleum gas (LPG) as a fracturing fluid. An advantage of using LPG instead of water as the base fracturing fluid is the increase in the effective fracture lengths. In some hydraulically fractured wells the effective fracture length has been observed to be as low as 30% of the created fracture length (Tudor et al., 2009). This decrease in effective fracture length when using water is attributed to multiple explanations. One of these explanations is that water will react with clay and salts in the formation. This often can lead to a phenomenon where the clay swells as it comes in contact with water, thus drastically reducing the permeability in the formation. The water also can become trapped within the formation pores which hinder the flow of hydrocarbons to the wellbore (Soni, 2014). A solution to increase the effective fracturing length is using LPG as the base fracturing fluid. Fracturing formations with LPG virtually eliminates the lost fracture length that is associated with fracturing with water. In addition to that benefit, LPG is easy to gel and has sufficient viscosity to transport proppant into the formation. Since LPG is miscible with hydrocarbons the cleanup process is simple and there is

effectively no damage done to the wellbore when flowing back the fracturing fluids. A case study was performed in the McCully gas field in New Brunswick, Canada. By comparing the flowback in adjacent wells it was found that using LPG as a fracturing fluid holds promising results. The results showed that the effective fracture length doubled that of a water frac; also, in all LPG fractures 100% of the fracturing fluid was recovered during a two week period. This virtually eliminated any hindrance that may have occurred from using a water frac. The production results confirmed the speculation that fracturing with LPG is more effective than water. Long term forecasting of the production data showed an increase in productivity of 100% by using LPG as the fracturing fluid (LeBlanc et al., 2011). The results from this experiment are promising and by researching new stimulation technologies we hope to further expand on the stimulation technologies that are available to the industry.

Cryogenic fracturing using liquid nitrogen can achieve extremely low temperatures and very strong thermally induced stresses. Grundmann et al. (1998) treated a Devonian shale well with liquid 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 differentially including anisotropic stress conditions and heterogeneous reservoir conditions over short distances. To further advance the study of cryogen fluids on hydrocarbon producing formations, McDaniel et al. (1997) conducted simple laboratory studies where coal samples were immersed in cryogenic nitrogen. The coal samples experienced significant shrinkage and fracturing into smaller cubicle units, with the creation of microfractures orthogonal to the surface exposed to the cold fluid. The researchers found that repeated exposure cycles to the cryogen caused the coal to break into smaller and smaller pieces, or become rubblized. After 3 cycles of exposing the coal to liquid nitrogen and allowing the coal to ambient temperatures again, 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 et al. (1997) also conducted field experiments by re-simulating four coal-bed methane (CBM) wells and one tight sandstone well with liquid nitrogen. The wells were retrofitted with stainless steel surface piping, manifolds, and wellhead component to prevent thermal contraction problems. A free hanging fiberglass tubing was used to inject the liquid nitrogen without compromising the casing integrity. The results were mixed: All 5 wells showed promising re-stimulating initial production rates 10-20 times the before-re-stimulation production average; however, those rates quickly dwindled. The CBM wells showed sustained 6 month re-stimulation production increase of 0-45%. The tight sand well

initially had higher flow rates for 2 months after re-simulating, but then had a 65% loss in
production from pre re-stimulation performance. It is believed that the initial success in
re-stimulating these wells was not that new fractures were created but that the damage
from the gel filter cake of previous fracturing treatments were greatly reduced.

These prior researches suggest that field application of liquid nitrogen with special equipment rated for liquid nitrogen temperatures is possible, and may bring in some promising benefits. They did not, however, identify the specific fracture initiation and propagation mechanisms at work in downhole conditions. There are also uncertainties on how to transport proppants to prop open the newly formed fractures. Liquid and gasified nitrogen both lack significant viscosity (see e.g. Rudenko and Schubnikow (1968)) and may therefore inadequately carry proppants if viscosity serves as the primary transport mechanism. Gupta and Bobier (1998), for the case of liquid carbon dioxide, argued that low viscosity could not enable adequate proppant transport; however, it is possible to use a higher velocity of injection to create high-Reynolds-number turbulent flows to enhance transportation of the proppants, at least through the wellbore to the perforations, if not through the fractures (Gupta and Bobier, 1998). The McDaniel et al. (1997) research, which demonstrated coal rubblization in laboratory experiments, suggests 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. Production enhancement observed in some of the wells tested was indeed long term without proppants. Ultra-light weight proppants (ULWPs) may also be used with liquid nitrogen. ULWPs are manufactured proppants that consist of a chemically hardened walnut hull core with multiple layers of epoxy resin coating acting as the outer shell (Kendrick et al., 2005). Prior research (Kendrick et al., 2005) showed improved post-stimulation production in Devonian Shale wells fractured 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 nitrogen foam fractured wells with traditional proppants.

In summary, fracturing with liquid nitrogen is viable for the field and may bring with it substantial benefits in reducing the formation damage and water and chemical use. However, when it comes to the understanding of the fracture initiation and propagation mechanisms and the consequence of low viscosity on proppant transport, not much work, especially laboratory work, has been done. In this study, we injected liquid nitrogen into the center of a transparent acrylic cylinder to visualize fracture initiation due to temperature effect. It is the first step toward understanding the mechanisms of liquid nitrogen fracturing and toward developing and improving the process for field applications.

Experimental Setup

Devices and Procedure

We consider fracturing cryogenic thermal shock, which depends on pure thermal gradient and resulting thermal tensile fracturing and subsequent cryogen transportation into fractures.

In the test setup, we are mainly concerned about cooling the borehole as rapidly as possible to maximize thermal gradient. This is done by flowing LN₂ continuously through the borehole. In this lab-scale experiment, LN_2 was pumped from the Dewar by pressure difference using a liquid nitrogen withdrawal device (Figure 1). Liquid nitrogen was transported by a vacuum-jacketed hose to the specimen, and injected into the borehole and then directed to an outlet. A pressure transducer was attached to monitor the borehole pressure. In this thermal shock setup, pressure inside the borehole was basically the same as the pressure inside the Dewar. Cryogenic fracturing was done purely by thermal gradient; little pressure existed inside the boreholes (less than 10 psi) throughout the thermal shock. Pressure inside the borehole, LN₂ consumption, and temperature were monitored and logged. A pressure transducer (Omega model # PX309-300G5V) was located at the top of 8"-long stainless steel extension tube (1/8"OD), which create vapor cushion and dissipate low temperature to limit heat transfer (temperature above 0°C (32°F) observed at the top of the tubing throughout testings). Temperature was measured by T-type thermocouple (Omega TT-T-30-SLE) to measure cryogenic temperature, and its thin wire to allow prompt response to temperature changes. Having data for both temperature and pressure provide reliability about data interpretation. More complete information about the devices can be found in Cha et al. (2014).

Taking advantage of the specimen being transparent, we observed the flow characteristics inside the borehole. Upon the start of the experiment, nitrogen inside the borehole was flown initially as a gas (for about 1~2 minutes), and then flown as a gas mixed with droplets of liquid, and finally flown in a more continuous phase of liquid with still a significant amount of gas phase intermixed.

Specimen preparation: transparent acrylic specimens

Two acrylic specimens are used as transparent specimens. Acrylic specimens are chosen because they are transparent, and relatively brittle, which is one of the important characteristics of rocks.

Specimen 1. The dimensions of the acrylic specimen 1 are illustrated in Figure 2a. The acrylic cylinder is 4" in diameter and 9.1" in height and the borehole is drilled from top, and 7" in depth and 0.5" in diameter. A 0.5" O.D. stainless steel tube was inserted and

228 attached to the borehole wall using epoxy to the depth of 2.5". An LN_2 inlet tubing was 229 inserted to 2.25" beyond the casing end.

231 <u>Specimen 2</u>. The sample dimensions of Specimen 2 are the same as those of the Specimen
232 1. However, unlike Specimen 1, both the steel casing and the inlet point were 1.5" in
233 depth (Figure 2b). The injection point was purposely placed higher than Specimen 1 to
234 study the effect of the injection point location.

Results

237 Temperature, pressure, and LN₂ consumption

Temperature of the specimen dropped rapidly with the introduction of LN_2 , and reached LN₂ boiling point within five minutes in this laboratory setups. The temperature distribution at the surface was also dependent on the proximity to the cracks due to transportation of LN2 through cracks (as this will be shown in Figure 8). Temperatures dropped by non-negligible amount shortly after the end of the test (e.g. TC #2 of Specimen 1 and TC #2, TC #4, and TC #5 of Specimen 2 in Figure 3), which probably caused by the pressure drop at the borehole.

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Although a lot of LN_2 (20kg) was flown in the experiment 1, most of the fractures occurred at an early stage (within 15 minutes). The Dewar lever was opened fully during releasing LN_2 without intermediate closure. The experiment for Specimen 2 was terminated by depletion of the LN_2 tank. The duration of the experiment 2 was 11 minutes and the amount of nitrogen consumption was 7.6kg. Pressure measured at the borehole was in the range of 3~5 psig, which was exerted by the Dewar tank.

- 41 252
- ⁴²₄₃ 253 Crack development

Images of specimens were captured throughout the testings using a digital camera(Olympus PEN EPL1 12.3-megapixel).

Specimen 1. Images of specimens were captured in a video throughout the experiment (Figure 5). It was observed that fracture growth was not continuous, but rather jumpy, characterized by abrupt starts and stops. This suggests that the tensile stress generated inside the borehole must reach a certain threshold for fracture initiation and growth. The increased material's brittleness at low temperature may have also contributed to this behavior. During the experiment, clear audible sounds were emitted, when the fractures were observed to grow. The magnitude/amount of instantaneous growth between starts and stops tends to decrease as the fracture grew larger. Most of the cracks occurred

within 20 minutes. Two distinctive patterns in crack development were observed:horizontal, planar, radial fractures, and vertical cracks joining the horizontal fractures.

The horizontal, planar, radial fractures form the dominant pattern of crack morphology. This can be explained by the fact that the specimen is cylindrical with a borehole height greater than the diameter, which makes thermal contractions more pronounced in the longitudinal direction. The horizontal fractures were clearly spaced by a certain length, which can be considered as an "exclusion distance". An exclusion distance exists because a set of crack cannot be created closer than a certain length due to a limited amount of thermal contraction (Figure 6). The behavior of exclusion distance also exist in other phenomena, such as mud crack, frost heaving area, and dissolution pipes etc. (Buijse, 2000; Jenkins, 2005; Toramaru and Matsumoto, 2004).

Fractures were generated in the vertical direction in a less magnitude compared to the horizontal fractures, caused by the circumferential thermal contraction. The vertical tensile fractures tend to initiate from or form between the horizontal fractures and bridge them. It is energy-efficient to start from one pre-existing defect (i.e., a horizontal fracture) and propagate toward another pre-existing defect (Figure 5 ~ Figure 6). At the end of the experiment, the specimen showed a complex fracture morphology created by the interplay between longitudinal and circumferential thermal contractions (Figure 5).

286 <u>Specimen 2</u>. Two horizontal fractures were observed: one in the steel cased part of the
287 acrylic sample and another right next to the inlet port (Figure 7). Following the initiation
288 and propagation of the horizontal fractures, vertical cracks were created and they bridged
289 the horizontal fractures.

Similar to the Specimen 1 test, the fracture in the uncased part was located close to the inlet port. In this test, however, one big radial wing was created (compared to three in the previous test), which means that there was not enough driving longitudinal contraction to generate more horizontal fractures. The lack of thermal driving and multiple horizontal fractures could be due to the shorter stimulation time – 11 min vs. 36 min in Specimen 1 test, thus lower amount of LN_2 applied - 7.6kg vs. 20kg in Specimen 1 test, by the early depletion of the LN_2 tank.

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299 Effect of presence of fracture on temperature distribution

Temperature distribution at the surface and inside the acrylic samples was dynamically coupled to the initiation and growth of fractures. During the experiments, it was observed that liquid nitrogen moved into and flowed through created fractures, which helped fractures to further propagate. This, in turn, accelerated the temperature propagation. Figure 8a show that some cracks approached the surface at the later stage of the

experiment, and the temperature on surface near the crack was readily affected by theproximity to the cracks.

308 Effect of casing / inlet location

For both Specimen 1 and Specimen 2 tests, one major horizontal fractures were initiated from early stage at the steel cased parts of the acrylic cylinders (Figure 5 and Figure 7) although they are far from inlet ports. This is perhaps due to the efficient heat transfer of the casings, which has a high heat conductivity. The steel casing will also have shorter period during which it is under Leidenfrost effect. These fractures, however, did not propagate as far as those in the borehole. This, perhaps, is due to the steel casing blocking the flow of LN_2 into these fractures; this may be also due to the steel casing, epoxied to the acrylic cylinder, changing the stress condition and preventing further growth of the fracture (Figure 9). Clearly, the steel casing and the epoxy have influenced heat transfer, flow of LN₂, stress distribution and eventually affected the fracture distribution.

We notice that the distribution of cryogenic temperature inside the borehole was affected by the location of the injection point. Fractures were mainly generated near the injection point, which suggests colder temperatures near the injection point (Figure 9).

325 Conclusions

Experiments were performed to study the development and morphology of fractures generated by a cryogenic thermal shock in a borehole geometry. We designed our experimental apparatus and procedures specifically for thermal stimulation using liquid nitrogen. Direct observations of fracture formation were made possible by the use of transparent acrylic specimens. The study provides key observations on fracture initiation and propagation when sufficient thermal contraction / tensile stress is achieved in a borehole.

Cryogenic fracture growth was observed to be abrupt due to the brittleness of the material at the cryogenic temperature and the accumulation-release of tensile stress coupled with fracture propagation and heat transfer. The area of fracture created at each growth event tends to decrease as the fractures become larger. Two distinctive patterns in the fracture development were observed: one is horizontal, planar, and radial propagation created by longitudinal thermal contraction, and another is vertical fractures created by circumferential contraction. The horizontal fractures were initiated first and were more dominant than the vertical fractures. This is perhaps because the borehole height is much greater than the borehole diameter, which makes thermal contractions more pronounced in the longitudinal direction. The horizontal fractures tend to be spaced by a certain length (exclusion distance), which exists because a set of fractures cannot be created closer than a certain length due to limited amount of thermal contraction. The vertical fractures tend to initiate from or form between the horizontal fractures and bridge them. We expect that the sequence of initiation and patterns will also depend on the stress condition of the specimen and this will be examined in future experiments.

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Highlights

- We study cryogenic fracture development and morphology in a borehole geometry.
- Cryogenic fracture growth was observed to be abrupt due to materials' brittleness.
- Two distinctive fracture patterns, horizontal and vertical propagations, were observed.
- Horizontal, radial propagation created by longitudinal contraction is more dominant.
- Vertical fractures by circumferential contraction is created between the horizontal patterns.



Figure 1. Setup for cryogenic stimulation experiments.



(b) Acrylic Specimen 2

Figure 2. Acrylic specimens tested – Dimensions and locations of the stainless steel (SS) casing and the inlet tube.



(a) Temperature vs. time - Specimen 1



(b) Temperature vs. time - Specimen 2

Figure 3. Locations of thermocouple tips and temperature evolutions during the cryogenic thermal shock experiments.



Figure 4. Borehole pressure and LN_2 released during the test – Specimen 1.



Figure 5. Crack development. The steps do not represent all the crack growth steps – Specimen 1.


Exclusion distance

Horizontal radial planar cracks caused from longitudinal thermal contraction

Figure 6. Crack morphology and driving thermal tensile stresses – Specimen 1.

Vertical circumferential

tensile crack due to

circumferential thermal contraction



Figure 7. Crack development – Specimen 2.



Figure 8. Effect of crack propagation on the surface temperature. Local frost in the circled area of Specimen 1 is due to water vapor condensation in an area where a fracture reached to the surface.



(a) Specimen 1

Figure 9. Effect of casing / inlet location.

A crack plane aided by the proximity to the inlet port



Effect of steel casing

(b) Specimen 2