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Experimental Investigation of Cryogenic Fracturing of Rock Specimens Under True Triaxial Confining Stresses

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Abstract

We have performed a laboratory study of cryogenic fracturing for improving oil/gas recovery from low-permeability shale and tight reservoirs. Our objective is to develop well stimulation techniques using cryogenic fluids, e.g. liquid nitrogen (LN) to increase permeability in a large reservoir volume surrounding wells. The new technology has the potential to reduce formation damage created by current stimulation methods as well as minimize or eliminate water usage and groundwater contamination.

The concept of cryogenic fracturing is that sharp thermal gradient (thermal shock) created at the rock surface by applying cryogenic fluid can cause strong local tensile stress and initiate fractures. We developed a laboratory system for cryogenic fracturing under true triaxial loading, with a liquid nitrogen delivery/control and measurement system. The loading system simulates confining stresses by independently loading each axis up to about 5000 psi on $8'' \times 8'' \times 8''$ cubes. Both temperature in boreholes and block surfaces and fluid pressure in boreholes were continuously monitored. Acoustic and pressure-decay measurements are obtained before and at various stages of stimulations. Cubic blocks ($8'' \times 8'' \times 8''$) of Niobrara shale, concrete, and sandstones have been tested, and stress levels and anisotropies are varied. Three schemes are considered: gas fracturing without cryo-stimulation, gas fracturing after low-pressure cryogen flow-through, gas fracturing after high-pressure flow-through.

Pressure decay results show that liquid nitrogen stimulation clearly increases permeability, and repeated stimulations further increase the permeability. Acoustic velocities and amplitudes decreased significantly following cryo-stimulation indicating fracture creation. In the gas fracturing without the stimulation, breakdown (complete fracturing) occurs suddenly without any initial leaking, and major fracture planes form along the plane containing principal stress and intermediate stress directions as expected theoretically. However, in the gas fracturing after cryogenic stimulations, breakdown occurred gradually and with massive leaking due to thermal fractures created during stimulation. In addition, the major fracture direction does not necessarily follow the plane containing principal stress direction, esp. at low confining stress levels. In tests, we have observed that cryogenic stimulation seems to disrupt the internal stress field. The increase of borehole temperature after stimulation affects the permeability of the specimen. While a stimulated specimen is still cold, it keeps high permeability because fractures remain

open and local thermal tension is maintained near the borehole. When the rock becomes warm again, fractures close and permeability decreases. In these tests, we have not used proppants. Overall, fractures are clearly generated by low and high-pressure thermal shocks. The added pressure of the high-pressure thermal shocks helps to further propagate cryogenic fractures generated by thermal shock. Breakdown pressure is significantly lowered by LN stimulation with breakdown pressure reductions up to about 40% observed.

Introduction

To improve well productivity and and hydrocarbon recovery, almost all oil and gas wells drilled in unconventional plays are completed with stimulation. Massive hydraulic fracturing has been demonstrated as one of the most effective technologies and are widely applied in combination with horizontal drilling for developing shale reservoirs (Steward 2013). Due to the general availability and low cost of water, fracturing fluids of hydraulic fracturing are mostly water-based, containing proppants and some chemical additives (Sharma et al. 2004, Shaefer 2005).

Several major shortcomings accompany the application of water-based fracturing fluids. First, water can cause significant formation damage to shale formation in the sense that clay-rich shale tends to absorb water and swell, narrowing the conductive fractures and pores. Also, capillary retention of water would partially or completely block the flowpath of hydrocarbon from matrix to fracture networks (Mazza 1997). Secondly, water usage in large quantities is concerning, placing demands upon local water supply and environments, especially in areas where water supply is at shortage. For instance, during the period of 2009-6/2011 in Texas, the median water usage in hydraulic fracturing for each horizontal well in Barnett, Eagle Ford, and Haynesville were 10,600, 16,100, and 21,500 m³, respectively (Nicot and Scanlon 2012). The lower volume, 10,600 m³ can fill more than two Olympic swimming pools or supply water for 65 families for one year. Finally, high pressure downhole injection of fracturing fluids containing chemical additives has led to a contentious community and political climate over underground water contamination. In contrast to hydraulic fracturing, cryogenic fracturing using liquid nitrogen (LN) offers potentially greater fracturing capabilities without any of the issues associated with water-based fracturing fluids.

Cryogenic fracturing is a relatively new stimulation technology that looks to expand and improve the traditional hydraulic fracturing technology. Cryogenic fracturing rests on the idea that a sharp thermal gradient caused by contacting with and vaporization of a cryogen, can induce fractures when brought into contact with a much warmer rock under downhole conditions. Cryogen exists in the gaseous phase at standard conditions but takes a liquid form at low temperatures, such as liquid nitrogen and liquid carbon dioxide. Specifically, when liquid nitrogen is injected into a borehole, heat from the rock near the borehole will quickly transfer to the liquid nitrogen at boiling point (-195.8°C or -320.4°F at atmospheric pressure), resulting in rapid cooling of the near-borehole area, which will cause the surface of the rock or borehole wall to contract. Once the tension due to contraction is sufficiently increased, fractures orthogonal to the interface of cryogen and rock can be initiated. These newly induced fractures can be further extended by high pressure gas from LN vaporization. Note that nitrogen has a liquid-to-gas expansion ratio of 1:694 at 20°C (68°F) and atmospheric pressure.

Although cryogenic fracturing has not been widely deployed for developing unconventional reservoirs, it was tested in a few field cases during the 1980s and 1990s. Instead of water, Lillies and King (Lillies and King 1982, King 1983) pumped gelled liquid carbon dioxide at -28.9°C to -40°C (-20 °F to -40°F) to stimulate tight gas sand formations using standard tubing and casing configurations. On average, 3-4 days after the fracturing treatments, oil and gas wells were cleaned up with complete flowback of vaporized liquid carbon dioxide, without producing any formation damage. In these cases, the gelled carbon dioxide was capable of carrying proppants due to its higher viscosity than liquid CO_2 , hence the fractures were able to stay open. Accordingly, all the wells for which they published results experienced increased production rates (Lillies and King 1982, King 1983).

McDaniel et al. (1997) conducted simple laboratory studies in which coal samples were immersed in LN for observation of their fracturing process. The coal samples experienced significant shrinkage and broke into smaller cubic units, creating microfractures orthogonal to the surface exposed to the liquid nitrogen. The researchers found that repeated exposure cycles to the cryogen caused the coal to break into smaller pieces, or become rubblized. After three cycles of submersion into liquid nitrogen and warm-up to ambient temperatures, the coal sample was reduced to grain-size particles. McDaniel et al. (1997) then continued field tests with liquid nitrogen, and published before-and-after production rates for five wells. The results were mixed: three CBM wells showed increased production, one CBM well showed equivalent production, and one low permeability sandstone well initially completed with slick water fracturing showed decreased production. By injecting liquid nitrogen, Grundmann et al. (1998) treated a Devonian shale well 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.

Liquid nitrogen and liquid carbon dioxide have low viscosities (Rudenko and Schubnikow 1968, Fenghour et al. 1998) and therefore are inadequate in carrying high density proppants if viscosity serves as the primary transport mechanism. Gupta and Bobier (1998) concluded that liquid carbon dioxide could not enable adequate proppant transport; however, it is possible to increase the flow rate for improved proppant transport. The turbulence caused by high Reynolds number flow allows sufficient transport of proppant, at least through the wellbore into the perforations, if not through the fracture (Gupta and Bobier 1998). Some research even showed that cryogenic fracturing may not rely as extensively upon proppant as traditional hydraulic fracturing. The research of McDaniel et al. (1997), in which coal rubblization was demonstrated in laboratory experiments, suggests a self-propping mechanism. That is, if rock undergoes sufficient thermal-induced breakage on the fracture planes, the rubblized rock particles may keep the fracture open against in-situ compressive stresses after the cessation of thermal contraction. If neither traditional proppants nor self-propping mechanisms can effectively prop the cryogenic fractures, ultralight weight proppants (ULWPs) may fill the gap. ULWPs are artificial proppants that consist of a chemically hardened walnut hull core with multiple layers of epoxy resin coating as the outer shell (Kendrick et al. 2005). in In Devonian shale, Kendrick et al. (2005) observed improved post-stimulation production from wells treated by nitrogen foam fracturing with ULWPs. It also showed that the majority of the wells treated with the ULWPs performed as good if not better than those treated with traditional proppants.

Although several field cases have been implemented as mentioned above, during the past 15 years no further studies were continued for better understanding and application of this promising fracturing technology. The fracturing processes, mechanisms, and controlling factors of cryogenic fracturing are still poorly understood. As more and more unconventional plays are being developed, it becomes necessary to conduct research to further investigate this formation and environment-friendly fracturing technology and to discover the significant potentials it can offer for the oil and gas industry. This experimental study aims to better understand and optimize the cryogenic fracturing processes using liquid nitrogen and discusses how it can be integrated into our current fracturing technology to enhance the oil and gas recovery of unconventional reservoirs.

We conducted preliminary cryogenic tests to understand the cryogen and material behaviors by performing submersion tests and applying cryogen to boreholes in unconfined concrete specimens (Cha et al. 2014). Initial data gathered from the previous study provided basic understanding in its physical mechanisms. In this study, we present the study on the effect of cryogenic stimulation on rock fracturing under true triaxial confining stresses. We developed laboratory setups and procedures that are designed for conducting cryogenic fracturing tests under wellbore conditions. In these tests, LN is flowed through

boreholes drilled in rock blocks (concrete, shale, and sandstone) under triaxial confining stresses. Processes are thoroughly monitored to observe the behaviors associated with cryogenic fracturing. Fracture development was assessed by acoustics, permeability, visual inspection, and X-ray CT. Comparisons are made between pre- and post-stimulation conditions to evaluate the efficiency of the method. The research results will help to select efficient applications of cryogenic stimulations as a completion technique and will build the foundations for continuing avenues of research.

Laboratory Studies

The devices and procedures used in our tests are significantly improved and optimized based on our understanding on cryogen and fluid behavior from our preliminary studies. Artificial and natural rock specimens were prepared for the experiments. Shale and sandtones were collected from outcrop of oil and gas producing formations.

Experimental Equipment

We developed a laboratory system for cryogenic fracturing study under true triaxial loading conditions. It consists mainly of a triaxial loading system, a LN delivery, and control/measurement system (Figures 1 and 2).



Figure 1—True triaxial loading device: (a) Containment ring housing the specimen and actuators, (b) Closeup inside the ring showing axes and faces of specimen.



Figure 2—Cryogenic fracturing setup (true triaxial loading device not shown).

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Triaxial loading system The true triaxial loading system was developed to simulate effects of stress levels and stress anisotropy manifested *in situ* on the characteristics of cryogenic fracturing. The containment is designed for the selected specimen size $8'' \times 8'' \times 8''$. The triaxial loading (TX) system can load the specimen 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. One advantage of our system is the vertical loading frame can be moved by rolling sideways on the bed. This provides a user with space to work on specimens and inside the containment. An open system is preferred in our study to better deal with any unexpected situation such as a cryogen spill and for ease of instrumentation (Figure 1).

PTFE or silicon pads are placed between rock specimens and the loading pad to provide uniform contacts and loading to specimen surface. PTFE or silicon pads resist temperature down to -350°F and -80°F, respectively. Automatic servo control system for load control is not available in our system nor required as our problem is quasi-static. Constant forces can be maintained by either manual control or in quasi-automatic manner using pressure relief valves attached to the hydraulic lines. In the manual control, a small additional amount of pressure is applied by pumping when certain amount of natural decay occurs.

Liquid Nitrogen delivery Liquid nitrogen is released out of the dewar once an outlet is opened by the internal gas nitrogen pressure builtup inside the dewar (Figure 2). This pressure is usually kept at relatively low levels ~10-20 psi. The fluid injection/delivery system for cryogenic fracturing needs to be different from that for hydraulic fracturing. For cryogenic thermal fracturing, cryogenic fluid needs to keep flowing in order to cool the borehole down, because stagnant liquid nitrogen will quickly boil and vaporize because the speciemen is much hotter. Compressed nitrogen gas was used to either push liquid nitrogen into borehole under higher pressure ("higher-pressure LN flow") or directly pressurize boreholes for breakdown tests. Pressure can be applied as shown on the right-hand side of Figure 2.

Tubing and fittings in the cryogen transport lines must withstand cryogenic temperature (down to -321°F in our study). Materials for tubings and fittings we used for cryogen transport are stainless steel 316 and brass, which generally provide such an ability as their brittleness-ductility transition temperature is lower than liquid nitrogen boiling point. Stainless steel has higher pressure rating at the cryogenic temperature must be seamless and annealed. 1-inch stainless steel tubing was used for borehole casing and is mounted to the borehole wall by epoxy for sealing and pressure resiliance at cryogenic temperature. Epoxy generally worked well for that purpose, but could deteriorate after 3-5 times of uses. Insulation is applied between Dewar and specimen inlet to minimize heat loss.

Measurements & monitoring The measurements made include pressure, temperature, photography of specimens, and liquid nitrogen consumption. Pressure is measured using a pressure transducer attached to the end of a standoff pipe (Figure 2), where a vapor cushion is created to prevent conductance of the cold temperature to the sensor. Data show that the temperature at the top of the stand-off pipe remains above 0°C throughout the stimulation (Figure 3). The pipe length should not be longer than necessary as a long narrow pipe can create drag force and thus may decrease responsiveness to rapidly-changing pressure. For temperature measurement, T-type thermocouples were chosen for range and accuracy. Very thin thermocouple wires are inserted into borehole between casing and borehole walls. One thermocouple is suspended in the borehole to measure temperature in the borehole, and another is attached to the borehole wall to observe the actual temperature of the wall. Liquid nitrogen consumption is measured by placing Dewar on a scale. In addition, acoustic and X-ray computed tomography (CT) scanning are used for fracture assessment before and after stimulations.



Figure 3—Low-pressure LN borehole flow-through for an unconfined specimen - temperatures in various locations.

Test procedure

Cryogenic stimulation is accomplished by applying liquid nitrogen from liquid nitrogen sources into the borehole of the specimen. Baseline measurements of rock conditions relevant to fracture indication are done before any treatment, and then the same measurements are performed during treatments or after completing the treatments for comparison. Fracture assessment include acoustics, pressure decay tests, visual inspection, and CT.

Stimulation procedure As mentioned previously, boiling LN is needed to lower temperature and since the specimen is much hotter than liquid nitrogen any stagnant condition will increase the borehole temperature rapidly. Thus, liquid nitrogen is continuously flowed through the borehole. Using our cryogenic fracturing apparatus under triaxial loading conditions, we performed two different cryogenic stimulation schemes: 1) low-pressure liquid nitrogen flow and 2) high-pressure liquid nitrogen flow. In low-pressure liquid nitrogen flow, liquid nitrogen is directly flowed from the dewar from a pressure difference between inside the dewar and outside the dewar upon opening the dewar's release valve. Pressure ranges from 5-20 psi in the borehole, depending on the internal pressure level inside the dewar. In the high-pressure scheme, liquid nitrogen is flowed through the borehole under higher pressure (300-400 psi) for faster cooling in the borehole due to reduced film boiling effects. In high-pressure liquid nitrogen flow, there is also a pressure effect, which facilitates fracture opening by helping to reach tensile strength of the rock. Normally we apply the higher-pressure stimulation multiple times because our vessel for storing liquid nitrogen for higher-pressure injection is small (1 liter), which makes each stimulation cycle very brief (1-2 min).

Fracture assessment Fracture assessment is carried out by borehole pressure decay test, acoustic measurements, and specimen breakdown by gas pressure. Borehole pressure decay is performed by applying a pressure to the borehole, shutting the borehole in, and monitoring the pressure decay. This was tested before cryogenic treatments as a baseline, and then tested between treatments and after completing cryogenic treatments for comparison. After all planned stimulation is completed for each specimen, specimens were subject to gas nitrogen (GN) pressure to fully fracture ("breakdown") the specimen. These breakdown pressures are compared with baseline breakdown pressure of untreated specimen, and also with those of specimens that were treated in different situations. Breakdown tests are done at the last stage after all other tests and measurements are done as it will fully fracture to the surface.

Transmission of elastic waves in both P and S-modes are measured using ultrasonic transducers before and after the treatments. The post-stimulation measurements were done before applying breakdown pressure, so that we can know the effect of cryogenic stimulations. 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 fractures (or fracture spacing) (Cha et al. 2009). 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 fracture generation.

Specimens

Collection and preparation Shale and sandstone were collected from outcrops of producing formations to make specimen types more relevant to the purpose of this study. A fairly large specimen size $(8'' \times 8'' \times 8'')$ is selected for the study in order to create sufficient thermal gradient in the specimen for an extended time, which may be required for thermal tensile fracturing. Basic index properties of intact rock of the tested specimens are listed in Table 1. Three different types of rock specimens are tested in this study and listed in Table 2.

Rock Type Properties	Concrete	Sandstone	Shale
Density (g/cm ³)	2.041	2.18	2.389
Unconfined compressive strength (MPa)	37.34	41.46	54.59
Splitting tensile strength (MPa)	2.88	4.505	8.45
Young's Modulus (GPa)	30.0	44.9	49.3
Constraint modulus (GPa)	36.3	51.2	61.2
Shear Modulus (GPa)	12.5	18.4	19.5
Poisson's ratio	0.243	0.22	0.268
P-wave velocity (m/s)	4220	4850	4970
S-wave velocity (m/s)	2455	2906	2796
Specific heat capacity (J/(kg·K))	891	857	990
Porosity (%)	9.56	11.5	6.64
Permeability (md)	8.72×10 ⁻³	0.349	1.06×10^{-3}

Table 1-Mechanical, thermal, and flow properties used for cryogenic fracturing test under confinement

Table 2-Matrix of cryogenic fracturing experiments performed under triaxial confining stresses

Specimen #	Specimen type	Stresses (x:y:z)	Test procedure	Measured P _{BD} [psi]	Notes
16	Concrete	500:750:1000	Fracturing by GN	583*	* Weakness created near the casing
17	Concrete	500:750:1000	Fracturing by GN	1180	
18	Concrete	500:750:1000	Low-pressure LN flow (30 min) + Fracturing by GN	685	
19	Concrete	500:750:1000	High-pressure LN flow + Fracturing by GN	778	
20	Concrete	500:750:1000	High-pressure LN flow + Fracturing by GN	759	
21	Concrete	1000:1500:2000	Fracturing by GN	1317	
22	Concrete	1000:1500:2000	Low-pressure LN flow (30 min) + Fracturing by GN	-	
23	Concrete	1000:1500:2000	High-pressure LN flow + Fracturing by GN	1094	
S1 (24)	Shale 1	1000:1500:2000	Low-pressure LN flow (40 min)		CT scanned before & after the LN test.
			High-pressure LN flow	1394	3 LN treatment cycles.
S2 (25)	Shale 2	1000:3000:4000	High-pressure LN flow (1 st round) High-pressure LN flow (2 nd round)	1417	3 LN treatment cycles for each round. LN flowed until getting similar
					decay curves at last runs.
S3 (26)	Shale 3	1000:1500:2000	Low-pressure LN flow (40 min)		Pressure decay before & after show an permeability enhancement
			High-pressure LN flow	168	Fractured at early stage when doing pressure decay before testing & before loading (fractured at 180 psi)
S4 (27)	Shale 4	1000:1500:2000	Fracturing by GN	2439	
SS1	Sandstone	1000:1500:2000	High-pressure LN flow	219	Fractured during pressure decay test when $\sigma_z = 60$ psi
SS2	Sandstone	$\sigma_{\rm z}$ = 60 psi	Fracturing by GN	689	

Mortar concrete blocks are used as surrogate for real rock. A fresh concrete with a water to cement ratio of 0.55, and sand to cement ratio of 2.5 was poured into the $8'' \times 8'' \times 8''$ mold and sealed in a plastic bag. After 24 hours, the seal and mold were removed and the concrete was cured under water (ASTM 2014a) for at least two months, which maximizes hydration enhancing concrete strength. Niobrara shale blocks were gathered from an outcrop of the Niobrara formation in Lyons, Colorado. The sandstone blocks were gathered from an outcrop of Williams Fork Formation in Western Colorado. Both the shale and sandstone formations are oil and gas producing strata at some regions in Colorado and other states.

Large shale and sandstones blocks were precisely cut into $8'' \times 8'' \times 8''$ cubic specimens using a bridge saw. After cutting, a 1-inch diameter wellbore was drilled by using a diamond imbedded coring drill bit with 1.06 inch outer diameter under dry conditions and the wellbore was drilled to a depth of six inch. Following drilling a 1-inch stainless steel-316 tube (casing) were attached to the wellbore by applying epoxy after the thermocouples were placed inside the wellbore. The casing extends two inches into the wellbore.

Intact rock properties Key intact rock properties were obtained and may allow more quantitative approaches to further analyze results. Mechanical, thermal, and flow properties of the specimens were tested and obtained. Permeability and porosity were measured using CMS 300 (CoreLab). Elastic constants were obtained from measurements of elastic wave velocities (Cha and Cho 2007). Specific heat capacity was obtained by using a calorimeter. Splitting tensile strength and unconfined uniaxial compressive strength were obtained using procedures from the ASTM standards (ASTM 2008, 2014b).

Results and analyses

Eight concrete speciemens, four shale speciemens, and two sandstone specimens were tested under different triaxial confining stresses and with different cryogen treatment procedures as shown in Table 2. The results are analyzed, compared and discussed here.

Temperature and pressure evolution in boreholes

Breakdown tests were performed on specimens that were not treated with LN (Specimens #16 and #17). In these tests, the GN was injected into the specimen to pressurize the borehole and fracture the specimen (Figure 4). These breakdown pressure values are compared with values for specimens that were treated with LN in different situations.



Figure 4—Breakdown fracturing test (using gas pressure) of a specimen without LN treatment (Specimen #17).

Temperature and pressure evolutions during low-pressure liquid nitrogen flow and high-pressure liquid nitrogen flow through boreholes are presented (Figure 5-6). For low-pressure injection, cryogen pressure in the borehole remains low (10-15psi) (Figure 5b). Temperature drops fairly quickly; however, on the borehole wall, the rate of temperature drop is retarded compared to that of the borehole (Figure 5a). For high-pressure injection, the cryogen pressure during the flow-through is 250-450psi, but very brief e.g., 1-2 min (Figure 6b). The temperature drops more quickly than for the low-pressure treatment due to reduced effect of surface film boiling under higher pressure flow-through (Figure 6a). For high-pressure treatment, multiple cycles were performed because of the small volume of the LN container (1 liter) and thus small amount of LN available for each treatment. Upon ceasing the LN supply, temperature in the borehole increased quickly back after these durations of stimulations.



Figure 5—Temperature and pressure during low-pressure LN flow-through (Specimen #18).



Figure 6—Temperature and pressure during multiple higher-pressure LN flow-through (Specimen #19).

Leidenfrost effects (i.e., surface film boiling) were more prominent in the unconfined specimen tests than the confined specimen tests (Compare Figure 3 with Figure 5-6). In confined test, under the similar low pressure flow condition in borehole, the temperature drop was faster than that of the unconfined tests. This could be due to contraction of borehole wall circumference when subjected to high confining stress; the thermocouple may have been loosened and even detached under flow.

There were indications of specimen fracture from the pressure response in hydraulic lines for loading actuators (e.g., Figure 4b). The x-axis hydraulic line responded with the high jump in hydraulic pressure for its hydraulic piston at the time of the breakdown fracture. This indicates that the fracture opened in the minimum horizontal stress direction (x-axis) (propagated in the direction of the maximum horizontal stress), which has been also confirmed later by surface photos of the breakdown fractures.

Breakdown by gas pressure on untreated specimens is characterized by a sudden loud fracture without any pre-leaking of gas. However, approaching the breakdown point on cryogenically treated specimens involves significant high-pitch leaking sounds which increases with increasing borehole pressurization before final breakthrough. There may be possibly progressive type of fracture as well.

We performed tests that showed cryogen leaking during cryogenic stimulation which means breakthrough just by cryogenic stimulation. It showed at both low and high pressure injections (Figure 5b and Figure 6b). In higher-pressure LN flow, liquid nitrogen leaked during 3rd high-pressure LN flow through, indicating there were already significant fractures created that extend to the surface.

Figure 7 summarizes and compares breakdown pressures of tested specimens. It clearly shows that cryogenic treatment decreases breakdown pressure (PBD) level in concrete and shale specimens by both low and high pressure liquid nitrogen flow-through. PBD reduction is 37% for concrete specimens and 43% for shale specimens occurred.



Figure 7—Effect of cryogenic stimulation on breakdown pressure under confining stresses.

Fracture pattern

Cryogenic stimulations alone generally did not lead to fracture propagation to the block surface. Only in some long-duration stimulations, fractures propagated to the external surface; they were identified by visually identified, localized leakage of liquid nitrogen during stimulation. In this section, we present fracture patterns visible on the external surfaces of blocks after breakdown tests.

Direction of breakdown fractures - Untreated specimen As explained previsouly, when breakdown fracturing of specimens that did not undergo liquid nitrogen stimulation, they tend to fracture suddenly without any pre-leaking. The major fracture direction in the specimens that were not stimulated with LN is perpendicular to the minimum horizontal stresses direction (i.e., x-axis) from the borehole wall (Figure 8). The x-axis is the minimum horizontal stress direction and y-axis is the maximum horizontal stress direction. In other words, their major fracture planes tend to exist along y-z plane, which is the plane containing the principal stress direction and the intermediate stress direction. This behavior is theoretically expected from rock mechanics and also observed in hydraulic fracturing. The top view picture from inside the wellbore after breakdown also shows the fractures propagate perpendicular to the minimum horizontal stresses direction from the borehole wall (Figure 8a).



Figure 8—Surfaces after breakdown fracturing (using gas nitrogen) without any LN stimulation. Note that fractures are located along y-z plane (i.e., the plane with the principal stress and intermediate stress directions). Breakdown fractures occurred suddenly without any pre-leaking.

Effect of cryogenic stimulations on direction of fracture propagation We observed that if specimens are cryogenically stimulated, the breakdown fracture direction can deviate from the conventionally predicted directions. This is particularly true when the confining stress is low. For examples, fractures are in rather arbitrary directions (Figure 9a&b), or occurred only locally (Figure 9c&d). In Figure 9b, the fracture opened in a half wing configuration along the minimum horizontal stress direction. Another fracture wing was along the maximum horizontal stress direction. In Figure 9d, the main fracture propagation was around the wellbore, affected by casing's much higher thermal conductivity, creating weakness in that region. These fractures occurred gradually without clear fracture sound and with massive pre-leaking. Clearly, cryogenic stimulation disrupts the internal stress field. However, at higher confining stress and also perhaps with higher anisotropy in the applied stress, fractures follow theoretically predicted directions; the fractures propagated as expected, in a plane perpendicular to the minimum horizontal stress (x-direction). At high stress level, we surmise that confining stress would govern the fracture direction (e.g., Figure 10).



ל) Specimen #23

Figure 9—Surfaces after breakdown after LN stimulations. Fractures do not follow y-z plane, and are in rather arbitrary directions (a & b), or occurred only locally (c & d). Breakdown occurred more gradually with massive pre-leaking.



Front of cryogenic fracture



Figure 10—Fracture plane after breakdown fracturing of LN treated shale specimen. Yellow dotted lines show the front of cryogenically created fractures (Specimen #S2).

Cryogenic fractures as "seed fractures" for pressure-induced fracturing After the breakdown test, two major fractured pieces are opened, and fracture surfaces are visually inspected. There were clear discontinuities in surfaces between the cryogenically induced fractures and the fracture plane opened up by the breakdown pressure. The planes of cryogenic cracks are slightly rounded, while the planes opened up by breakdown gas pressure are straight (Figure 10). This cryogenic pre-fracturing or seed fracturing is not fully developed to the external surface, but will signicantly lower the breakdown pressure. The pore-fracturing observed during applying breakdown pressure to the borehole. It should be noted that this set of cryogenic fractures may not be the only set of cryogenic fractures generated. They are, however, the major ones through which the block is fractured by the breakdown pressure. Another charcteristics of cryogenically created seed fractures is that they all start from the borehole, which is favorable from the production's point of view. When the specimen was not treated with LN, a sharp straight fracture formed from the wellbore directly to the block surfaces (Figure 11).



Figure 11—Fracture plane after breakdown fracturing of untreated specimen (Specimen #S4).

Permeability changes

After each treatment procedure, enhancements in the permeability were assessed by borehole pressure decay tests.

Permeability increase due to cryogenic treatment Permeation (pressure decay) test results clearly show that low and high-pressure liquid nitrogen stimulations clearly increase permeability of stimulated specimens. In one test, low-pressure LN flowed for 36 min resulting in a significant increase in permeability (Figure 12) indicated by a more rapid pressure decay. Three cycles of higher-pressure stimulation under triaxial loading also led to significant permeability further. For example, at a higher-pressure LN flow test, permeability increases after each brief injection cycle (Figure 14).



Figure 12—Effect of low-pressure LN flow on permeability (specimen unconfined except 60 psi vertical loading) (Specimen #S1).



Figure 13—Effect of multiple high-pressure LN flow on permeability – first and last (specimen unconfined except 60 psi vertical loading) (Specimen #S1).



Figure 14—Effect of repeated high-pressure LN stimulations on permeability (Specimen #S1).

Effect of temperature We also observed the effect of temperature on the permeability of the specimen that has experienced cryogenic stimulation. Permeability decreased when the specimen returned to the room temperature after the 3^{rd} high-pressure LN flow (Figure 15 and Figure 12 for low-pressure treatment). When the specimen is at lower temperature, the specimen is in the state of local thermal contraction near the borehole and the crack will remain open around the borehole but closes as the specimen relaxed.



Figure 15—Effect of specimen temperature on permeability (under confining stresses) (Specimen #S1).

However, in untreated specimens or weakly treated specimens, the permeability is lower when the specimen is at its low temperature, and increases when it returned to the room temperature. In other words, if no crack exist near the borehole, permeability is lower at lower temperature. At low temperature, the formation is under thermal contraction and consequently, the pore sizes are reduced, and perhaps the natural fracture widths are also smaller. This behavior is sometimes observed after the first cycle of high-pressure flow through.

Effect of confining stress We observed that the presence of confining stresses decreases specimen's permeability. Figure 16 shows the shale specimen's pressure decay obtained before applying confining stress and the pressure decay curve after the specimen was placed under loading (Figure 16). This permeability reduction comes from compaction of pores or closure of micro cracks under confining stress.



Figure 16—Effect of confining pressure on permeability (Specimen #S1).

Acoustic wave signature

The differences in the acoustic waves before and after the stimulations were investigated. Figure 17 shows the locations for the acoustic measurements on a specimen surface. Acoustic signals were measured between Faces 1&3 and 2&4 (pairs of opposing faces). For each set of faces, acoustic measurements were conducted at twelve locations. We observed that acoustic velocities and amplitudes decrease (original amplitudes not shown) after cryogenic stimulations, indicating fracture creation in the specimens. For shale specimens, we also observed the effect of layering on acoustic velocity and attenuation.



Figure 17—Waves measured along Face 1 & 3 and 2 & 4 (a). Measurement location in a surface (b).

Acoustic measurement results before breakdown generally agree with the fracture profile after breakdown. In particular, velocities and amplitudes significantly decreased where a major fracture exist (Figures 18 and 19 lines #5-#10). Because waves interrogate the whole block, changes in wave signals (wave velocity and amplitude) reflect the average the whole block; however, the effect of the locality of the sensor is significant. Signals compared with their full range of the measurements show that the global frequency of the signals became lower after cryogenic fracturing at nearly all locations.



Figure 18—P-wave signals (amplitude adjusted) across Face 2-4 before and after stimulation (Specimen #19).



Before and after: delay in arrivals

Figure 19—S-wave signals (amplitude adjusted) across Face 2-4 before and after stimulation (Specimen #19).

The changes in P-wave velocities are summarized in Figure 20-21. Lower velocities near the edges (#1 & 8) and center (#4 & 5) show the effects of boundaries and borehole cavity. In rare cases, velocity slightly increased in a local area, which is indication of local compressed zones especially in the corner.



Figure 20—P-wave velocity comparison – Face 1-3 (Specimen #19).



Breakdown pressure analysis The breakdown pressures (P_{BD}) are compared in this section. A number of factors may affect the breakdown pressure including rock properties, the confining stress magnitude and anisotropy, whether the sample is treated with LN, and the duration of LN exposure. A simple estimation of expected P_{BD} can be given by using Equation 2, which assumes that when the tangential compressive stress in the wall of the well is equal to the tensile strength of the rock, the fracture will be created (Guo et al. 1993). The calculated P_{BD} are listed in Table 3 and compared with the measured values obtained in the lab.

Table 3—Comparison between measured P_{BD} and calculated P_{BD} .					
Specimen #	Specimen type	Stresses (x:y:z)	Measured P _{BD} [psi]	Calculated P _{BD} [psi] by Eq.	Mea.P _{BD} /Cal. P _{BD}
16	Concrete	500:750:1000	583*	1168	0.5
17	Concrete	500:750:1000	1180	1168	1.01
18	Concrete	500:750:1000	685	1168	0.58
19	Concrete	500:750:1000	778	1168	0.67
20	Concrete	500:750:1000	759	1168	0.65
21	Concrete	1000:1500:2000	1317	1918	0.68
22	Concrete	1000:1500:2000	-	1918	-
23	Concrete	1000:1500:2000	1094	1918	0.57
S1	Shale	1000:1500:2000	1394	2726	0.51
S2	Shale	1000:3000:4000	1417	1226	1.15
S4	Shale	1000:1500:2000	2439	2726	0.89

$$P_{BD} = 3\sigma_h - \sigma_H + T_0$$
 Equation 1

= Breakdown pressure (psi) P_{BD}

 $\sigma_{\rm H}$ and $\sigma_{\rm h}$ = Maximum and minimum horizontal stresses, respectively (psi)

= Tensile strength (psi)

Specimen #17 (concrete) was fractured by GN pressure without LN treatment. The calculated P_{BD} is close to the value obtained from the test. The P_{BD} for Specimen 17 can therefore be used as a baseline P_{BD} for the other tests using concrete specimens. Specimens 18-20 treated with LN with different procedures have lower P_{BD}'s than that of Specimen 17. P_{BD} value in Specimen 18 was reduced by 42%. Specimens 19 and 20 were reduced by ~ 36%. Specimens 21-23 were tested under higher triaxial loadings than Specimens 16-20. P_{BD} for Specimen 21, which was not treated with LN, is lower than the calculated $P_{\rm BD}$. Specimen 23 was treated, and the $P_{\rm BD}$ value reduced by 17% compared with the measured $P_{\rm BD}$ for Specimen 21. Shale S1-S4 were tested under the same triaxial loading as concrete specimens 21-23. S1 was treated with LN and shows a P_{BD} reduced by 43% compared to the P_{BD} for S4, which was not treated with LN. Based on these results, LN reduces the breakdown pressure needed to initiate the fracture and that the LN is creating cracks to the wellbore surface.

Permeability analysis using a well testing equation The permeability of specimen could be affected by factors such as the number of treatment cycles, the wellbore temperature during and after the treatment, and the loading applied. In Specimen S1, the permeability increased after each treatment cycle. To see how this affected the permeability, a well testing analysis was used to find the permeability factor f (K) and obtain the normalized value and then compare it with other f(K) values. Drawdown pressure analysis was used to arrive at a "value" for f (K). These calculations do not reflect the change in the permeability of the entire block; they are used as a procedure to quantify and compare the effect of wellbore cryogenic treatment region. From a pressure decay test, a semi-log plot of pressure versus logt is constructed. The slope for a straight line after the hump in this plot is used in Equation 2 (Sabet 1991, Horne 1995).

$$f(K) = \frac{K}{q} = -162.6 \frac{B \cdot \mu}{m \cdot h}$$
 Equation 2

- Κ = permeability (md)
- = gas flowrate (scf/day) q
- В = gas formation volume factor (RB/STB)
- = gas viscosity (cp) μ
- = the slope (psi/cycle) m
- = interested zone length (ft) h

The flowrate was unknown in the lab and is assumed to be constant. The output value is the K/q term and this value is calculated for each pressure decay situation and LN cycle. Finally, the value of how much the K/q increased or decreased after each test procedure was found. Figure 22a shows the value of the first permeability function (K_o) with the specimen under loading (x:y:z = 1000:1500:2000psi) and before any treatments divided by the permeability function after each treatment cycle. It shows that after each treatment, the permeability increases. After the first treatment cycle, the permeability increased 60% more after the second treatment. After the third treatment cycle, the permeability increased 37% from the second treatment cycle.



Figure 22—K/Ko vs. number of LN treatment cycles applied showing increasing in the permeability after each cycle.

Figure 22b shows the value of the first permeability function (K_o) for shale S2 when the specimen is under loading (x:y:z = 1000:3000:4000 psi) and before the treatment divided by the permeability function after each treatment cycle. Two runs were conducted on shale S2, and each run it was treated with three cycles (the total number of cycles for specimen S2 is six). All cycles are included in Figure 22b and show that after the second treatment, the permeability increases. However, after the first LN treatment cycle, the permeability decreased by 15%, possibly because of the higher stress applied in the y direction preventing thermal failure from occurring. With more LN cycles, there was more permeability enhancement which could be an indication of higher extension of the created formation cracks with each treatment cycle.

Effect of triaxial confining loading on the permeability The effect of loading on the permeability existed on all shale specimens. With loading, the permeability decreased significantly. The value of f(K) from laboratory results and the calculated permeability using Equation 3, values are comparable. The value of f(K) reduction (using the well testing technique) was 0.07609 while the value using Equation 3 (Kikani and Pedrosa Jr 1991) was 0.07906. Figure 23 show the f(K) values before and after loading without any treatment for shale S1 and S2, respectively.



Figure 23—Permeability reduction in shale after applying loading, calculated using Equation 3 (before applying crogenic treatment)

$$K = K_{a} \times e^{(-c_{f} \cdot \sigma_{avg})}$$

K, K_o= permeability after and before loading, respectively (md) c_f = formation (shale) compressibility (1/psi) $\sigma_{av\sigma}$ = the average in-situ stresses (psi)

Conclusions

We have conducted a number of cryogenic fracturing tests of concrete, sandstone, and shale blocks under true triaxial stress conditions using an integrated experimental system, designed and manufactured for this special purpose, and performed analyses and measurements on the samples before and after the LN stimulation.

Cryogenic treatments of concrete, sandstone, and shale samples all show reductions in their respective breakdown pressures. The effectiveness of cryogenic fracturing processes depends on rock properties, injection pressure, treatment time and cycles, and triaxial stress conditions. High injection pressure of LN through the borehole enhances the rock breakdown process.

In confined stimulation tests under triaxial stresses, cryogenic fractures with low horizontal stress contrasts opened along or between the directions of the minimum and maximum horizontal stresses. For higher horizontal stress contrasts, the fractures always propagated as expected, in a plane perpendicular to the minimum horizontal stress. Cryogenic stimulation changes the local stress field around the wellbore, as evidenced by gas fracturing profiles.

Acoustic measurements confirm that liquid nitrogen stimulation increases the matrix permeability by generating fractures inside the rock blocks. Multiple cycles of treatments in shale samples demonstrate that greater permeability enhancement can be achieved after each cycle, indicating that each LN treatment cycle not only creates new fractures, but also widens the existing ones. In addition, as temperature returns to ambient, the fractures narrow, as is evidenced by a decrease in permeability. Also, due to compressibility, permeability decreases under high triaxial stress conditions.

Analytical analyses show that cryogenic stimulation is capable of reducing breakdown pressures of concrete and shale blocks by up to $\sim 40\%$; for shale samples, permeability can be enhanced by up to $\sim 300\%$ after multiple cylcles of cryogenic stimulation; also, intact shale samples show noticeable compressibility under triaxial stress conditions.

No dramatic fracture generation is observed during cryogenic fracturing, indicating that additional injection pressure may be needed to induce significant fractures and deeper penetration.

The results and knowledge obtained in this study may be applicable to any wellbores that need stimulation such as oil and gas wells, and enhanced geothermal wellbores. If successfully deployed for field-scale applications, the cryogenic fracturing technique could reduce water usage in stimulation jobs, and thus mitigates the damages incurred by water-based fracturing fluids in water-sensitive formations, and will greatly reduce flowback disposal. Cryogenic fracturing can also be used in combination with other stimulation technologies, e.g. to reduce the breakdown pressure by creating seed fractures prior to a conventional hydraulic fracturing treatment.

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Equation 3

can effectively deliver hydrocarbons from domestic resources to the citizens of the US. RPSEA, operating as a consortium of premier US energy research universities, industry, and independent research organizations, manages the program under a contract with DOE's National Energy Technology Laboratory.

Nomenclature

μ	= gas viscosity (cp)
$\sigma_{ m avg}$	= the average in-situ stresses (psi)
$\sigma_{ m h}$	= Minimum horizontal stresses (psi)
$\sigma_{ m H}$	= Maximum horizontal stresses (psi)
c _f	= formation (shale) compressibility (1/psi)
h	= interested zone length (ft)
m	= the slope ($psi/cycle$)
q	= gas flowrate (scf/day)
В	= gas formation volume factor (RB/STB)
G	= giga
Κ	= permeability after loading (md)
Ko	= permeability before loading (md)
Μ	= mega
P _{BD}	= Breakdown pressure (psi)
T _o	= Tensile strength (psi)

SI metric conversion factors

inch \times 2.54	E-01	= m
$g/cm^3 \times 1.0$	E+03	$= kg/m^3$
$md \times 1.01325$	E+12	$= m^2$
psi × 1.45038	E-04	= Pa
$(T_{\circ_{\rm F}} + 459.67)/1.8$		$= T_{\circ C}$

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