

Waterless fracturing technologies for unconventional reservoirs-opportunities for liquid nitrogen

Lei Wang<sup>a\*</sup>, Bowen Yao<sup>a</sup>, Minsu Cha<sup>b</sup>, Naif B. Alqahtani<sup>c</sup>, Taylor W. Patterson<sup>a,d</sup>, Timothy J.

Kneafsey<sup>e</sup>, Jennifer L. Miskimins<sup>a</sup>, Xiaolong Yin<sup>a</sup>, Yu-Shu Wu<sup>a</sup>

<sup>a</sup> Petroleum Engineering Department, Colorado School of Mines; <sup>b</sup> Department of Civil Engineering, Texas A&M University; <sup>c</sup> King Abdulaziz City for Science and Technology; <sup>d</sup> now with Devon Energy; <sup>e</sup> Lawrence Berkeley National Laboratory

\*Corresponding author, email: lwang@mines.edu.

## **Abstract**

During the past two decades, hydraulic fracturing has significantly improved oil and gas production from shale and tight sandstone reservoirs in the United States and elsewhere. Considering formation damage, water consumption, and environmental impacts associated with water-based fracturing fluids, efforts have been devoted to developing waterless fracturing technologies because of their potential to alleviate these issues. Herein, key theories and features of waterless fracturing technologies, including Oil-based and CO<sub>2</sub> energized oil fracturing, explosive and propellant fracturing, gelled LPG and alcohol fracturing, gas fracturing, CO<sub>2</sub> fracturing, and cryogenic fracturing, are reviewed. We then experimentally elaborate on the efficacy of liquid nitrogen in enhancing fracture initiation and propagation in concrete samples, and shale and sandstone reservoir rocks. In our laboratory study, cryogenic fractures generated were qualitatively and quantitatively characterized by pressure decay tests, acoustic measurements, gas fracturing, and CT scans. The capacity and applicability of cryogenic fracturing using liquid nitrogen are demonstrated and examined. By properly formulating the technical procedures for field implementation, cryogenic fracturing using liquid nitrogen could be an advantageous option for fracturing unconventional reservoirs.

**Keywords:** hydraulic fracturing; shale; tight sandstone; waterless fracturing; cryogenic fracturing; liquid nitrogen

## **1. Introduction**

Without a doubt hydraulic fracturing has revolutionized the exploitation of unconventional oil and gas resources in the United States and around the world. Hydraulic fracturing for developing reservoirs of micro- and nano-Darcy permeability entails pumping highly pressurized fracturing fluids at high flow rates into the reservoir to create fractures in this rock. Hydraulic fractures predominantly propagate perpendicular to the minimum horizontal stress in a single plane around the perforations. As pumping stops and the fracture closes, the proppants suspended in the fracturing fluid prop open the complex network of fractures. These fracture networks increase the

contact area between the reservoir and the wellbore and serve as highly conductive pathways for reservoir fluids to flow into the wellbore for production, controlling a region surrounding the wellbore known as the stimulated reservoir volume (Mayerhofer et al. 2010; Yuan et al. 2015). Hydraulic fracturing and its associating technologies have drastically increased the oil and gas production in the United States (Steward, 2013).

Modern hydraulic fracturing technology is being applied world-wide in the field; more than 90% of gas wells and 70% of oil wells drilled in recent years have been hydraulically fractured (Brannon, 2010). Hydraulic fracturing typically relies on water-based fracturing fluids, including the popular slick water, due to the general availability and low cost of water as well as its ability for proppant transport; however, a dependence upon water presents several major shortcomings. First, water can cause significant formation damage, which is manifested as capillary end effects, effective permeability decreases, and clay swelling stemming from water imbibition and mineral hydration, respectively (e.g. Sinal and Lancaster, 1987). Formation damage mechanisms inhibit hydrocarbon flow from rock matrix into fracture network and thus impair production rates and recovery efficiency. Second, water use in large quantities may place significant stress upon local water resources, especially for areas experiencing droughts, as well as local environments. Based on statistics from thousands of wells drilled in unconventional reservoirs across the United States in 2014, the median annual water volume of hydraulically fracturing horizontal oil and gas wells are 15,275 and 19,425 m<sup>3</sup>, respectively (Gallegos, 2015). Third, improper disposal or treatment of large amounts of flow back fluids with contaminants could lead to contentious public concerns and political issues. Several cases of felt seismicity that are below the damage threshold of modern building codes were probably induced by injecting flow back fluids into disposal wells (Davies et al. 2013; Ellsworth, 2013). Unless being re-injected into deep formations, flow back fluids containing chemical additives, high concentrations of suspended solids, salts, and hydrocarbons, etc. needs to be properly treated in order to avoid environmental pollution, lifting the cost of implementing hydraulic fracturing treatments (Hayes et al. 2014). All of these water related issues necessitate the research and development of waterless fracturing technologies.

Foams, by stabilizing CO<sub>2</sub>, N<sub>2</sub> or their combination in liquid with the aid of surfactants, have been introduced for fracturing to reduce water usage and formation damage over 40 years (Blauer and Kohlhaas, 1974). The volume of gas in the foam system can amount to as much as 95%, and its subsequent expansion during flow back would assist and accelerate the cleaning up of the liquid phase from rock matrix (Gupta, 2005). Generally, the higher the density and viscosity of foam, the better it carries proppants and the deeper they can be transported. However, foam is sensitive

and fragile to high temperature, high salinity, and oil/condensate presence, therefore its efficacy is restrained by the reservoir conditions. In foam formulas, water still remains as an important constituent thus the water related issues cannot be completely eliminated.

## **2. Waterless Fracturing Technologies**

A few waterless fracturing technologies have been developed and tested in the field during the past several decades. In the following section, we review mechanisms, case studies, advantages and disadvantages of commonly used waterless fracturing technologies, including oil-based and CO<sub>2</sub> energized oil fracturing, explosive and propellant fracturing, gelled alcohol and LPG fracturing, gas fracturing, liquid/supercritical CO<sub>2</sub> fracturing, and cryogenic fracturing using liquid nitrogen (LN<sub>2</sub>).

### **2.1 Oil-based and CO<sub>2</sub> Energized Oil Fracturing**

Oil-based fracturing field tests were first implemented in Colorado, Kansas, Texas, and Wyoming in the late 1940s (Clark, 1949). In these tests 11 out of the 23 stimulated wells experienced increased productivity, while productivity decreased at 3 wells in Rangely, Colorado. In these field tests, gasoline was gelled to form high-viscosity fracturing fluid enabling sand transport by adding Napalm. Oil-based fracturing fluids are preferable in cold regions where water-freezing can be a problem, e.g. the North Slope of Alaska and Canada in winter (McCabe et al., 1990). Fluids including condensate, kerosene, diesel, or even crude oil can be used alone as or mixed to form the base fluid (Maberry et al., 1997). Usage of gasoline was initially thought to avoid most of the water-related formation damage. Oil-based fracturing fluids can however impair the effective permeability of gas reservoirs (Smith, 1973). Considering the confinement effect in small pores (~10 nm and smaller) in shale and tight reservoirs as well as the heavy hydrocarbon components introduced by oil (Wang et al., 2014), effective permeability decreases could get worse and capillary end effects could become much more significant than those in conventional reservoirs. Moreover, oil-based fracturing fluids are expensive and hard to dispose of.

To aid with the flow back of fracturing fluid and reduce the amount of oil required, gas (CO<sub>2</sub> or N<sub>2</sub>) has been used to “energize” the oil-based fracturing fluids (e.g. Vezza et al., 2001; Gupta and Leshchynshyn, 2005). Due to the good miscibility with hydrocarbons, CO<sub>2</sub> is often selected as the energizing gas. Experiments on Montney cores with average permeability of 6.6 μD demonstrated that a 50/50 vol% miscible CO<sub>2</sub>/C<sub>7-11</sub> fracturing fluid regained 99.9% methane permeability after a 7-day exposure under representative reservoir conditions, as is comparable to gelled propane, a 95-quality foam, and an 80-quality foam formed by a biopolymer gel (Taylor et al., 2009). Vezza et al. (2001) implemented CO<sub>2</sub> energized oil fracturing in a well in Morrow formation in

Oklahoma, which is a water-sensitive fine sandstone reservoir with measured core permeability of 1.26 mD. The target well was stimulated by energized diesel gel containing 30-40 vol% CO<sub>2</sub>. Comparison to two offset wells with similar formation properties that were treated by non-energized gelled diesel showed that initial production rate of the target well was 140% higher and its estimated ultimate recovery was estimated to be increased by 110%. Additionally, pressure build-up analyses obtained a fracture half-length of 65 ft and a total skin of -3 for the target well; superior to 35 ft half-length and -1 skin of the two offset wells. Gupta and Leshchyshyn (2005) compared short-term gas production data for 55 wells completed in the Rock Creek gas formation in Alberta, which has a typical porosity range of 10-14% and a permeability range of 1-5 mD (Stepic and Strobl, 1996). Of these wells, 7, 16, and 32 were stimulated by CO<sub>2</sub> energized, N<sub>2</sub> energized, and non-energized oil gel, respectively. Although different proppant types and concentrations were used, on whole CO<sub>2</sub> energized fracturing approximately doubled the average cumulative gas production achieved by N<sub>2</sub> energized and non-energized oil fracturing. Recently, in the Karr field of the Montney gas play, Hlidek et al. (2012) investigated the performance of energized oil gel and energized water gel in fracturing 6 horizontal wells. The pay zone is characterized by fine sandstone, and has an average porosity of 7% and an average gas permeability of 0.28 mD. Initial production rate data showed that three wells stimulated with 20 vol% CO<sub>2</sub> energized oil outperformed the other three stimulated with 25 vol% CO<sub>2</sub> energized water by 107%, even though the slurry volume of the oil-based fluid used was much smaller. Furthermore, flow back of oil-based fluid occurred 8 times faster than that for water-based fluid, with 75% more recovery. It is obvious that CO<sub>2</sub> significantly improves the compatibility of conventional oil-based fracturing fluid with unconventional reservoirs.

Nowadays, flow back that accounts for about 40-50% of the fracturing fluid is cleaned and recycled for subsequent fracturing, reducing the fluid cost and environmental impacts (Edwards, 2009; Hlidek, 2012). Spent oil is generally returned with produced oil to refinery for processing (Fyten et al., 2007). Still there are a few concerns associated with CO<sub>2</sub> energized oil fracturing, such as permeability damage due to residual fluid (Veza et al., 2001), CO<sub>2</sub>/hydrocarbon vapor separation, possible produced water separation from recovered flow back, and safety risks.

## **2.2 Explosive and Propellant Fracturing**

One of those earliest explosive fracturing technologies started in 1964 (Miller and Johansen, 1976), nitroglycerin and TNT were detonated in small 5-spot wells that were 60-100 feet deep to fragment oil shale formations in Wyoming. Extensive fractures were formed to a radius of 90 feet and significant airflow enhancement up to 800% was measured from different oil shale wells,

enabling sustained *in-situ* combustion. Application of explosive fracturing to deeper wells around 380 feet deep demonstrated enhancement of air flow as well. As trials to stimulate low permeability gas reservoirs, a series of nuclear fracturing were implemented in New Mexico and Colorado from 1967 to 1974 (Stosur, 1977). Tens of nuclear explosions for stimulating oil and gas wells were also carried out by the USSR during that period. These peaceful nuclear attempts proved to be very successful in increasing natural gas production, but were abandoned due to prohibitive accompanying radioactivity.

Rather than generating compressive shock waves formed in explosive fracturing, propellant deflagration fractures rock matrix by producing slower propagating pressure peaks, which are still rapid enough to create multiple fractures that can reduce the dominance of *in-situ* stresses. Sandia National Laboratory carried out a series of high energy gas fracturing experiments by burning or deflagrating propellant in boreholes, aiming to fracture oil, gas, and geothermal wells. Multiple fractures were obtained in wellbores with perforations (Warpinski et al. 1979; Schmidt et al. 1980; Cuderman, 1982, 1986). Propellant fracturing is not capable of carrying proppants into fractures, instead, shear slippage or spalling of the fracture planes might provide support for fracture openings (Page and Miskimins, 2009). Wieland et al. (2006) conducted laboratory propellant fracturing experiment on a 30" × 30" × 36" Mancos shale block, in which induced fractures propagated perpendicular to the minimum horizontal stress and were constrained by two well-cemented siltstone layers with high compressive strength. Page and Miskimins (2009) further extended propellant fracturing to stimulate three Mancos shale wells in Colorado, and one well that experienced a pressure increase of 200 psi after perforation showed slow gas flow and an elevated build-up of 1,375 psi over two weeks after deflagration. One year later, this well was stimulated with CO<sub>2</sub> energized slick water, however, breakdown pressure comparisons suggested no significant improvements lasting from the previous propellant treatment. But in agreement with the laboratory propellant fracturing, radioactive tracer logs after the slick water treatment exhibited clear containment of the fracture height growth, which might be a combined result of high strength layers, *in situ* stresses or shear slippage. To control the peak pressures, progressive burning propellants have been developed and successfully implemented in hundreds of wells in sandstone, limestone, shale, and coal formations (GasGun, 2016). Effective control of peak pressures maximizes the fracture growth by allowing more gas to be squeezed into formation without damaging the casing. In December 2005, propellant stimulations were tested against hydraulic fracturing in stimulating twelve shallow gas wells in Basal Belly River formation in Canada (Schmidt, 2009). Pressure transient analysis showed that the six wells stimulated with propellant exhibited comparable performance to the six wells treated with 5,000 kg sand

fracturing. The advantage is that propellant stimulation is considerably less expensive than hydraulic fracturing. However, it should be noted that propellant stimulations will never replace high volume hydraulic fracturing because of its lower fracture length and height (Schmidt, 2009). Since explosive and propellant fracturing do not inject fluids into the reservoir, flow back is avoided and production can be started immediately after treatment. For the same reason, no proppant will be transported into fractures. Instead, rock deformation, shear slippage, and rock spalling caused by rapid deflagration or explosion help prop the fracture open as conductive pathways.

### **2.3 Gelled Alcohol and LPG Systems**

Non-aqueous liquids, such as alcohol and liquefied petroleum gas (LPG), have long been recognized as promising fluids for fracturing unconventional reservoirs (Smith, 1973). Crosslinked gels have been employed to facilitate proppant transport by increasing the viscosity of non-aqueous fluids during fracturing. Laboratory studies and field application of crosslinked gelled methanol-based fracturing fluid demonstrated higher regained permeability than crosslinked water-based gel and gelled oil (Thompson et al. 1992). Miscibility in water and favorable interfacial tension help achieve more efficient clean up and recovery of the flow back after fracturing treatment, thus increasing the permeability to the gas phase from matrix to fracture networks. In spite of these advantages, high safety risks are associated with methanol-based fracturing fluid operations due to its unfavorable properties, including a low flash point of 11 °C, wide flammable limits ranging from 6.7% to 35%, and higher specific gravity of the vapor phase (Hernandez et al. 1994).

Recently, LPG has been gelled for efficient fracturing treatment and proppant transport in low permeability formations. In 210 cases, these treatments consistently demonstrated complete recovery of the fluid from the invaded zone within 24 hours and decreased formation skin (less than - 4.0) after gel breaking (Tudor et al. 2009). In these cases, wells stimulated by gelled LPG varied over a depth range from 750 feet to 11,500 feet and with permeability from 0.007 to 3.0 mD. In the unconventional McCully gas field, compared with water-based fluid, gelled LPG fracturing treatments generated longer effective fracture half lengths as evidenced by larger microseismic event area and higher gas production rates due to much better clean up performance maximized after 24 hours (LeBlanc et al. 2011). As a fracturing fluid, gelled LPG has several desirable properties, such as improved viscosity, low interfacial tension, and high solubility with hydrocarbons. Also, the flow back of LPG, unlike other foreign gases, does not need to be flared (Lestz et al. 2007). Similar to methanol, LPG has potential safety risks. For example, as one

major component, propane has much lower flash point, flammable limits of 2.0-10% (Cashdollar et al. 2000), and specific gravity of 1.52. Unfortunately, the cost of gelled alcohol and LPG is much higher than other fracturing fluids due to limited sources.

## **2.4 Gas Fracturing**

Formations can be fractured by injecting a gas at pressures high enough to split the rock matrix. The most widely available and economical gas species is gaseous nitrogen ( $\text{GN}_2$ ), which makes up 78% of air (Freeman et al. 1983). In the laboratory, Gottschling et al. (1985) demonstrated that gas created fractures are capable of accepting sand proppant transported in high velocity gas streams through perforations based upon tests carried out on physical perforation and fracture models. Then, in their field application in Ohio, multiple vertical wells were stimulated using gaseous nitrogen for the 3,000 feet deep Devonian shale formation, with the treatment procedure consisting of a perforation breakdown by HCl, high rate nitrogen pumping, injection of nitrogen gas and sand, and a nitrogen flush. The maximum sand concentration implemented was 0.4 lbm/gal, and the highest placement was 5,400 lbm for 20/40 sand. Comparison of two offset wells fractured by gas nitrogen showed that sand placement significantly enhanced the oil production by a factor of several times during the initial half year; long term production data were unavailable though. Since gas has a low density, gaseous nitrogen fracturing is primarily used in shallow unconventional oil and gas reservoirs less than 5,000 feet deep (Rogala et al. 2013). With gas, the low density and viscosity limit the capability of nitrogen fracturing to transport proppants. Although self-propping by rubbing fracture surfaces is considered as a contribution for fracture opening at shallow depths, this possibility is minimized for deep formations having tight cementation. However, with the recent development of ultralight weight proppants (Rickards et al. 2006; Gu et al. 2015), hundreds of gas fracturing jobs pumping 100% nitrogen were successfully performed in shallow shale and siltstone gas plays in Canada during 2006-2010 (Canyon, 2016). As an inert gas without water related issues and chemical additives, the economics and applicability of gaseous nitrogen fracturing has been well justified, as compared to foam and hydraulic fracturing (Kothare, 2012). Additionally, gaseous nitrogen is miscible with hydrocarbons, although its miscibility pressure with oil is generally higher than that of  $\text{CO}_2$  (Hudgins et al. 1990; Orr Jr. and Silva, 1987a, 1987b). In view of the advantages, inadequate proppant transport could be the last obstacle to be resolved before gas nitrogen fracturing becomes the best choice for shallow oil and gas well stimulation.

## 2.5 CO<sub>2</sub> Fracturing

The primary motivation for using liquid carbon dioxide as a stimulation fluid is to eliminate the permeability damage of residual fluid after the hydraulic fracturing of low permeability reservoirs, especially, for the low and slow fluid return problem. After a liquid carbon dioxide treatment, the carbon dioxide would evaporate and return to the surface under controlled rates as a gas, resulting in a more rapid and complete cleanup. Reservoir temperature and pressure are normally higher than the critical temperature 31 °C and pressure 7.38 MPa of CO<sub>2</sub> (Suehiro et al, 1996), hence CO<sub>2</sub> is generally at liquid or supercritical state while being injected for fracturing low permeability oil and gas reservoirs. To guarantee relatively high viscosity for efficient proppant transport, e.g. 0.12mPa·S at 20 Mpa and 280 K (Fenghour et al. 1998), the bottom hole temperature should be kept below 31 °C for CO<sub>2</sub> to exist in the liquid form (Lillies and King, 1982). There have been numerous attempts to add CO<sub>2</sub>-philic solvents or create gelled systems to improve proppant transport, and several successful formulas have been found (Enick and Ammer, 1998). Although CO<sub>2</sub> can be stored at about -30 °F in tanks on site, subsequent pressurization and transport through pumps and wellbore would heat it up to above 30 °F before entering the perforations, these temperatures are considered as non-cryogenic (Lillies and King, 1982). For a 6,000 feet deep well with bottom hole temperature of 150 °F and the minimum horizontal stress of 2,000 psi, and given the average thermal expansion coefficient of  $6 \times 10^{-6}$  (inch/inch)/°F and Young's modulus of  $2.5 \times 10^6$  psi (Grundmann et al. 1998), then a 120 °F temperature difference will result in a thermal stress of 1,800 psi, which alone is unlikely to initiate fractures in tight reservoir rocks confined by in situ stresses. But the thermal stress could contribute significantly to fracturing reservoir rocks.

Lillies and King (1982) and King (1983) presented results of over 40 gelled liquid carbon dioxide fracturing treatments in vertical tight sandstone wells, some of which, for example, were drilled into Codell sandstone with 8-13% porosity and 0.008-0.05 mD (Smith et al. 2014) as well as Dakota sandstone with 2.8-9.6% porosity and less than 0.2 mD permeability (Matuszczak, 1973) in Colorado. Since the liquid carbon dioxide is gelled, it has sufficient carrying capability for bringing proppants into fractures and holding them open. After treatment, all of the reported wells showed increased production rates, and for some of them significant enhancements of tens of fold were observed. Unfortunately, there are no post-fracturing production data over a long period that are publicly available for these wells. The field tests also did not show how much the thermal effect from the gelled liquid carbon dioxide contributed to the fractures generated underground. Five years later, Sinal and Lancaster (1987) analyzed over 450 fracturing treatments using 100% liquid CO<sub>2</sub> and sand proppants that were implemented in various low permeability gas formations



in Canada, such as Cardium sandstone with 9-12% porosity and less than 0.1 mD permeability as well as Cadomin with less than 10% porosity and 0.01-0.74 mD (Coskuner, 2006). These liquid CO<sub>2</sub> treatments achieved sand concentrations up to 1,100 kg/m<sup>3</sup> and emplacement of sand up to 44 tons by turbulence, without viscosifying agents eliminating residue. Six-month production data showed that on average liquid CO<sub>2</sub> treatments have twice as much production increase as gelled reformatte fracturing treatments. After fracturing, rapid clean up generally finished in 1-2 days. It is noteworthy that liquid CO<sub>2</sub> field treatments showed several constraints, which are lower sand concentration, smaller sand size, and inapplicability to high permeability reservoirs due to low viscosity. What's more, for oil reservoirs, liquid CO<sub>2</sub> does not work well in that the fractures generated are narrower than those created by conventional fracturing, unfavorable for viscous oil flow (Sinal and Lancaster, 1987).

In the early 1990s the United States DOE started to introduce liquid CO<sub>2</sub> fracturing into vertical Devonian shale wells in eastern Kentucky (Yost et al. 1993). Two-stage stimulations with CO<sub>2</sub>/sand, GN<sub>2</sub>, and N<sub>2</sub> foam/sand were performed in four, seven, and four gas wells within a distance of 20 km, respectively (Yost et al. 1994). Nine-month production data showed that wells stimulated with CO<sub>2</sub>/sand produced 1.9 and 4.9 times as much as those stimulated with GN<sub>2</sub>, and N<sub>2</sub> foam/sand, respectively. Note that GN<sub>2</sub> treatments did not place sand proppants in fractures. A follow-up review updated the 5-year production data for the seven wells (11 stages) stimulated by CO<sub>2</sub>/sand, the nine wells (17 stages) by GN<sub>2</sub>, and the five wells (9 stages) by N<sub>2</sub> foam/sand (Mazza, 2001) from those previously reviewed by Yost et al. (1993, 1994). It demonstrated that the ratio of cumulative production per stage from CO<sub>2</sub>/sand stimulations to GN<sub>2</sub> and N<sub>2</sub> foam/sand stimulations had increased from 1.9 and 4.9 to 3.0 and 6.5 times over the 5-year period, respectively. Additionally, production history indicated that production rate of wells stimulated by N<sub>2</sub> foam/sand decreased faster than the other two well groups (Mazza, 2001). In 1999, 16 vertical wells drilled in the 4,000 feet deep Lewis Shale, San Juan Basin were treated using CO<sub>2</sub>/sand, and measured pitot gauge rates showed an average 86% increase over 23 control wells treated with nitrogen foam (Campbell et al. 2000).

From 1981 to 1994, over 1,000 wells were stimulated with 100% liquid CO<sub>2</sub>, with improving techniques (Tudor et al. 1994). For example, densitometers were mounted on the blender to control the slurry quality, and a nitrogen tank was used to maintain liquid CO<sub>2</sub> at desired pressure. Although pumping equipment of standard high pressure was used, thermal contraction was considered in the design. The fracturing process was optimized for high permeability reservoirs, and has been applied to coalbed methane wells. In addition, nearly 200 treatments were

conducted by adding gas N<sub>2</sub> into CO<sub>2</sub> for economic optimization in Canada by 1997 (Gupta and Bobier, 1998); case studies with nitrogen quality of 50-67% showed that on average the total cost of commingled N<sub>2</sub>/CO<sub>2</sub> treatments was 23% less than 100% CO<sub>2</sub> while still achieving similar production enhancements (Tudor, 1995; Luk and Apskrum, 1996).

Compared with hydraulic fracturing, CO<sub>2</sub> is believed to be capable of creating more complex and extensive fracture networks under much lower breakdown pressures due to 1) its low viscosity (Ishida et al. 2013), 2) it is difficult for CO<sub>2</sub> to be trapped, and 3) it poses no formation damage threat, because of its miscibility with hydrocarbons (Middleton et al. 2015). In addition, attraction between CO<sub>2</sub> molecules and the organic matter is stronger than that between methane molecules and the organic matter in shale, which could result in enhanced desorption of methane during CO<sub>2</sub> injection into the shale formation (Cracknell et al. 1996). This preferential adsorption behavior and stronger adsorption capacity offers additional potential for CO<sub>2</sub> sequestration in fractured shale reservoirs during enhanced gas recovery or after the depletion of the reservoirs. Nevertheless, a few drawbacks are associated with CO<sub>2</sub> fracturing, such as obtaining and transporting CO<sub>2</sub>, and post-stimulation separation of this greenhouse gas from hydrocarbon stream.

## **2.6 Cryogenic Fracturing with Liquid Nitrogen**

Cryogenic fracturing is a relatively new technology in the petroleum industry and not much research has been done in this area. Rather than exerting high hydraulic pressure, cryogenic fracturing utilizes sharp thermal shock generated by cryogenics to induce drastic contraction to break reservoir rocks. Liquid nitrogen, for example, as one of the most common cryogenics, has a boiling point of -196 °C or -320 °F (Jacobsen et al. 1986). With the same assumption of reservoir conditions and rock properties in last section, bringing LN<sub>2</sub> into contact with reservoir rocks will result in a thermal contraction of 7,050 psi, which is 5,050 psi over the minimum horizontal stress, far exceeding the tensile strength of typical shale and sandstone rocks (Lin, 1983).

As one of the earliest studies of cryogenic fracturing, McDaniel et al. (1997) conducted several laboratory liquid N<sub>2</sub> submersion tests on coal samples to prove that cryogenic fracturing may have an advantageous effect on enhancing gas production from tight and low-rate coalbed methane wells. The coal samples experienced significant shrinkage during the submersion tests with creation of micro-fractures orthogonal to the surfaces which were exposed to cryogen. After three cycles of submersion in liquid nitrogen and warming up to the ambient temperature, the coal samples were shattered into grain size particles. This research showed that cryogenic fracturing can effectively increase the production in coal-bed methane formation and may also have a

promising effect on other rock formations. Based on the laboratory experiments, McDaniel et al. (1997) further applied liquid nitrogen through fiberglass tubing to re-fracture four coalbed methane wells, which were originally fractured using foam and gel and one low-permeability sandstone well previously treated with slick water. The production responses from all these wells were very positive during the first several months. However, in the long term, only the two coalbed methane wells that were treated by two 10-min stages with water mist injection between them had increment in production. The other two coalbed methane wells treated for 30 mins experienced nearly equivalent production as before and the low-permeability sandstone well that was also treated for 30 mins experienced decreased production. During the same period, early results of two slick water re-fractures in similar coalbed methane wells with comparable total cost showed no production enhancement. Grundmann et al. (1998) later conducted a similar cryogenic fracturing treatment in a vertical Devonian shale well that was 3,020 ft deep and 4.5" in diameter with liquid nitrogen flowing through a 2.375" OD fiberglass tube at about 12 bbl/min and 3,000 psi. Two stages injecting 197 bbl and 136 bbl of liquid nitrogen were performed, separated by a water mist spacer, which blocked the first stage fractures and diverted the liquid nitrogen to other zones. The well showed an 8% increase in the initial production rate when compared to a nearby offset well that underwent a traditional nitrogen gas fracturing treatment. In addition, liquid nitrogen injection used only about half of the pumps required by hydraulic fracturing, thus avoiding construction of large setups.

Although field tests have shown some promising benefits from cryogenic fracturing, they did not identify the fracturing mechanisms that work at downhole conditions. Additionally, there are also concerns about the effectiveness of cryogenic fracturing, such as equipment used for injection cryogenic fluids and proppant carrying capability. Liquid nitrogen, similar to liquid CO<sub>2</sub>, lacks significant viscosity for carrying proppants into fractures in downhole conditions (Rudenko et al. 1934), which may result in inadequate proppants in fractures to hold them open. But it is also very feasible for liquid nitrogen to transport adequate amounts of proppants by increasing the injection velocity. Gupta and Bobier (1998) concluded that the turbulence accompanied by the high velocity permitted proppant to be carried efficiently from the wellbore to the perforations or even to the fractures during liquid CO<sub>2</sub> or liquid CO<sub>2</sub>/N<sub>2</sub> fracturing. In addition, with the rubblization effect discovered in the research from McDaniel et al. (1997), the rock formations treated with cryogenic fluid may undergo a self-propping process. The rubblized rock may enable the fractures to stay open against in-situ stress after cessation of treatment pressure. Most of the work performed to date on cryogenic fracturing was simple laboratory work or fieldwork that does not show consistent results. None of the previous work actually reflects the fracture

initiation by cryogenic fracturing under different states of stress and temperature. Therefore, there is a need to define the fracture mechanisms presented during the cryogenic fracturing under such conditions.

Without the issues associated with the use of water-based fracturing fluids, cryogenic fracturing offers potentially greater fracturing capabilities. As for formation damage, there are no concerns with cryogenic fracturing, since nitrogen will not react with minerals inside the formation, neither as liquid nor as gas. In addition, there is no liquid flow back after cryogenic fracturing because nitrogen gas is miscible with natural gas and has little retention when displaced by liquid hydrocarbons inside the formation. After cryogenic treatment, the formation is unaffected by issues affecting water-based fracturing. Cryogenic fracturing can also minimize water consumption in the stimulation process, which will save millions of gallons of water compared to traditional hydraulic fracturing operations. In contrast with other waterless fracturing technologies reviewed above, cryogenic fracturing does not involve violent explosions, combustible fluids, gelled systems, or greenhouse gas control, totally eliminating these public concerns. If extensively deployed, liquid nitrogen can be obtained by separating and compressing nitrogen gas from air by commercial air separation equipment at operation sites, which also minimizes the cost for transportation or pipeline construction.

### **3. Experimental Study of Cryogenic Fracturing**

To investigate the fracturing mechanisms and characteristics of cryogenic stimulation, we designed three types of experiments to investigate the effect of varying experimental parameters and conditions on the fracturing processes. It is vital that all samples tested are similar to each other so comparisons can be made among them. To ensure this, all of the rock samples tested are processed into 8" × 8" × 8" blocks, an intermediate scale between cores and reservoirs. The types of artificial and natural rock samples tested include concrete, low permeability sandstone, and shale. Submersion tests were performed on concrete samples to demonstrate the concept of cryogenic fracturing by sharp thermal gradient and allowed refined observation of fractured surfaces. Borehole thermal shock tests that flowed liquid nitrogen into the borehole were conducted to assess whether cryogenic stimulation is capable of generating fractures from borehole wall by thermal stress alone. Tests with borehole pressurization mimicked the field situation investigating the means to improve the penetration of liquid nitrogen into the cryogenically generated fractures and enlarges the region of cryogenic contact with rock.

To better evaluate and illustrate the efficacy of cryogenic fracturing, we established several kinds of measurement methods, including the pressure decay test and breakdown pressure measurement

by gas nitrogen fracturing. Pressure decay testing provides the rate of gas leak-off, which is an indication of the gas permeability. All the pressure decay tests were performed by pressurizing the wellbore of the rock sample up to about 180 psi, and after that the borehole was shut in, allowing the nitrogen gas in the wellbore to leak off while measuring the pressure. Assuming that gas flow during the pressure decay tests is radial and steady-state through homogeneous medium, then the effective permeability in mD can be determined by  $k = -162.6 q \mu B / (mh)$ , where  $q$  is the gas flow rate (STB/day),  $\mu$  is the gas viscosity (cp),  $B$  is formation volume factor (RB/STB),  $h$  is formation thickness (ft), and  $m$  is the slope of the first straight line in semilog pressure-time plot (Horner, 1951; Horne, 1995). Herein, permeability values are quantified as the reciprocal of  $m$ , i.e. all other parameters are assumed as constants. Thus it is only an approximation rather than an accurate calculation neglecting many factors. For instance, the gas nitrogen viscosity almost doubles from -160 °C to 20 °C at 145 psi (Span et al. 2000), which would result in an underestimated permeability at ambient temperature. The intent of breakdown pressure measurement by gas fracturing was to quantify the breakdown pressure values and compare them with those of intact rock samples to quantitatively characterize the degree of cryogenic damage occurred inside the rock blocks.

### 3.1 Concrete Samples

Concrete samples with consistent formula (mass ratio of sand : Portland cement : water = 2.50 : 1.00 : 0.55) were treated in two ways to investigate the effectiveness of cryogenic fracturing on generating fractures on the contact interface and inside the samples. The average tensile strength of concrete samples used in these experiments measured by Brazilian test is 418 psi (Yao, 2016).

#### 3.1.1 Fracture Pattern

As proved by the simple laboratory experiments done by McDaniel et al. (1997), liquid nitrogen submersion created orthogonal fractures to the surfaces of the coal sample and multiple treatments broke it into fine cubic particles. As preliminary tests, simple semi-submersion and full-submersion experiments under no confining stress were conducted to examine the impact of sharp thermal gradient on concrete sample surfaces (Cha et al. 2014). In the 30-minute semi-submersion test, when liquid nitrogen was filled up to the midline of the concrete block, a visible fracture was created along the midline all the way around, while other regions didn't show apparent fractures. CT scans showed that the fracture penetrated into the center of the concrete block. Although the concrete was not cracked as drastically as coal, it did verify that thermal

gradient alone is capable of cracking high strength rock samples (Alqahtani, 2015). Full-submersion tests were then carried out by fully immersing concrete samples into liquid nitrogen for 50 minutes. Pictures were taken for the top surface of the dry concrete sample before and right after the full-submersion test, as shown in **Figure 1**. When the sample was still cold the fractures were easily observed by the naked eye and formed a polygonal network, which is in qualitative agreement with previous recognition and observation of cryogenic fracture patterns (Grundmann et al. 1998). Since there were 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. For example, **Figure 2(b)** shows a new fracture on the front face, next to it is a pre-existing fracture that was extended vertically by liquid nitrogen treatment. After the sample temperature rose back to the room temperature some fractures were observed to close.

A wet concrete block, cured in the same environment with the dry one, was placed in water for one week. Following that, it was treated with liquid nitrogen in the same manner. As was seen with the dry sample, on the concrete block surface several new fractures were identified, and similar fracture networks were formed with polygonal shapes around the exterior of the block. However, these polygonal shapes are bigger in size and much sparser compared to those on the dry sample, as shown in **Figure 3**. In borehole cryogenic stimulation of concrete samples under no triaxial stress loading, this polygon fracture pattern was also observed on the surfaces of the samples; besides, after each cryogenic treatment existing fractures were widened and new fractures were created (Cha et al. 2014; Alqahtani, 2015). Also, the dry sample has more fractures that propagate away from the block edges, while the fractures on the wet sample were created near the edges. This probably have been due to the effect of ice formation in the pores of the wet sample. Ice formation would only occur in the pores on the outer layer of the sample, causing additional stress on the block. The freezing of outer layer caused lateral expansion, resulting in shear fractures parallel to the exposed surfaces (Kneafsey et al. 2011).

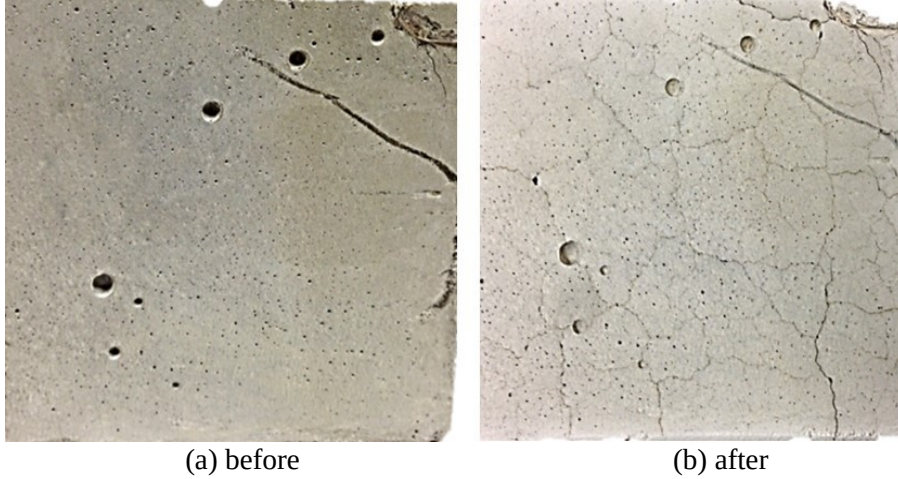


Figure 1. Top surface of the concrete block before (a) and right after (b) the full-submersion test.

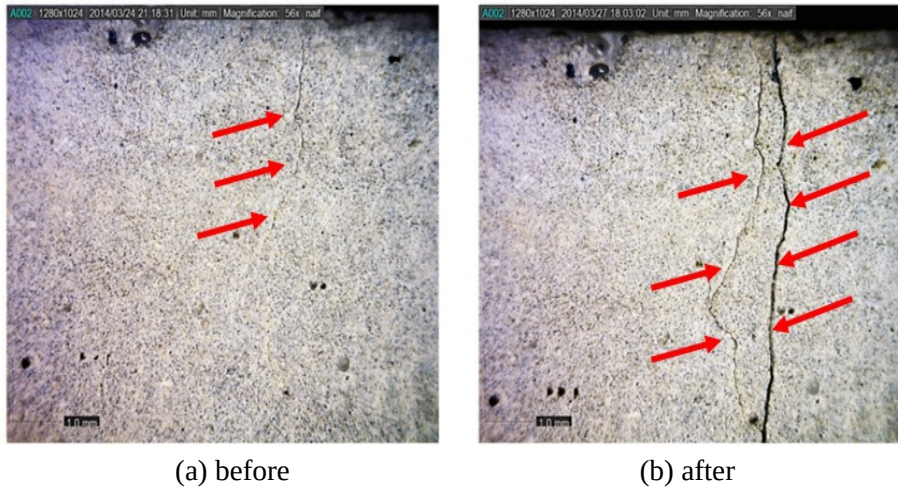


Figure 2. Images of the concrete block front face after the full-submersion test show both the extension of existing fractures and the creation of large, new fractures (1 mm resolution).

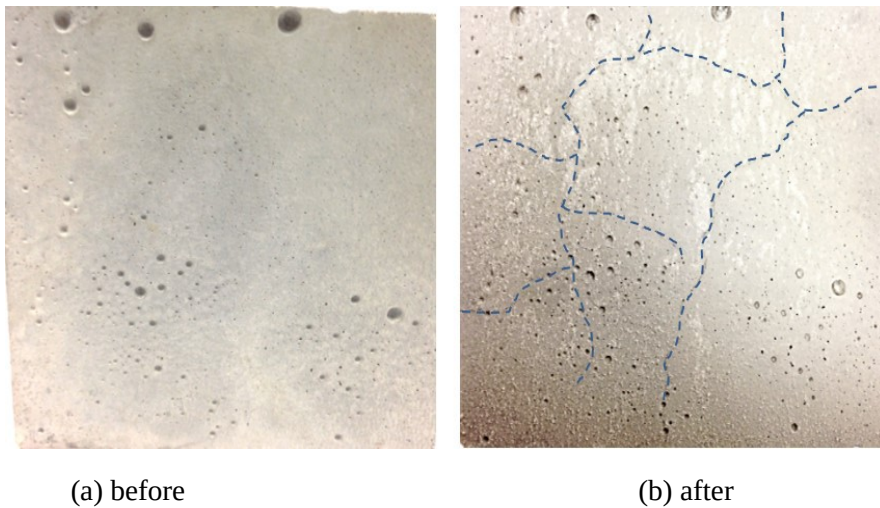


Figure 3. Top surface of the wet concrete block before (a) and after (b) the full-submersion test.

### 3.1.2 Breakdown Pressure

Under true triaxial stress conditions applied by a custom-built laboratory system (Cha et al. 2016), five dry concrete samples initially at room temperature were stimulated by flowing liquid  $N_2$  into the borehole. After the cryogenic treatment, we injected gas nitrogen into the borehole with increasing pressure to break down the rock samples. The peak pressure at which the samples broke down were recorded for comparison to highlight the deterioration of the rock strength after cryogenic treatment. These breakdown pressure values are compared with baseline values for three intact samples that were only fractured with gas nitrogen under different triaxial conditions, as summarized in **Figure 4**. The “triangle” data points were acquired from experiments done with triaxial stresses of  $x : y : z = 500 : 750 : 1000$  psi. The “square” data points were obtained from experiments with triaxial stresses of  $x : y : z = 1000 : 1500 : 2000$  psi. It can be seen that as the triaxial stresses and the stress difference doubled, the breakdown pressure of the samples was elevated, which is reasonable because the thermal stress induced by fracturing fluid must first overcome the confining stresses, then conquer the tensile strength of the concrete to split it. Also, for each triaxial stress loading condition, there is a negative correlation suggesting that liquid  $N_2$  treatments weaken the rock strength, though one of the  $GN_2$  fractured concrete samples deviates from this correlation.



Figure 4.  $GN_2$  breakdown pressure values of intact and  $LN_2$  stimulated concrete samples.



### 3.2 Shale Samples

Shale samples were acquired from the Niobrara outcrop in Northern Colorado, XRD analysis of the shale unit gave average mineralogical composition of 50.9% carbonate, 21.8% clays, 13.0% quartz, 2.6% TOC, and 11.7% other minerals by weight (Matthies, 2014). Three shale samples were stimulated with different LN<sub>2</sub> treatment procedures under different elevated triaxial stress conditions, and one sample was directly fractured by GN<sub>2</sub> to measure the breakdown pressure for comparison. Shale sample availability was very limited, therefore, multiple tests were completed on each sample with different LN<sub>2</sub> treatment procedures. After each treatment procedure, enhancements in permeability were quantified by pressure decay tests for comparison. Finally, after GN<sub>2</sub> fracturing, created fractures were observed and recorded.

#### 3.2.1 Pressure Decay Curves and Permeability Enhancement

Shale sample S2, under triaxial stresses of  $x : y : z = 1000 : 3000 : 4000$  psi, was treated with two rounds of liquid N<sub>2</sub> treatment, each of which contained three cycles of 15-second LN<sub>2</sub> injection at 450 psi. **Figure 5** shows the pressure decay tests with different stress conditions after the first round of treatment. It is obvious that the pressure decay is affected by the stresses applied on the sample, and the permeability decreased when the sample was placed under stress loading. **Figure 6** shows the pressure decay tests for this shale sample before LN<sub>2</sub> treatment and after each LN<sub>2</sub> treatment cycle, all of them were conducted under triaxial stress loading, displaying significant reduction in the pressure decay time after each treatment cycle. Permeability evaluation from semilog pressure-time showed that after two rounds of cryogenic treatment, the permeability of shale sample S2 reached about 14 times of the original value of 0.0003 mD right after the cryogenic treatment. Meanwhile, our finite difference matching of the pressure decay curves by incorporating Mogi-Coulomb criteria (Al-Ajmi and Zimmerman, 2006) into TOUGH2-EGS (Fakcharoenphol et al. 2013; Xiong et al. 2013; Zhang et al. 2015, 2016) showed a 10-fold improvement in permeability (Yao, 2016). **Figure 7** shows the pressure decay tests with different temperature conditions while the sample was still under triaxial stress loading. The permeability decreased by about two-thirds according to semilog pressure time analysis when the sample returned to room temperature, demonstrating that the cryogenic fractures partially closed as the sample warmed up, as was observed in the submersion tests of concrete samples.

Figure 5. Pressure decay tests under different stress conditions before the second round of liquid N<sub>2</sub> treatment on shale sample S2.

Figure 6. Pressure decay tests before and after each LN<sub>2</sub> injection cycle of the second round of treatment for shale sample S2 under true triaxial stresses.

Figure 7. Pressure decay tests at different temperature conditions for the third cycle of the second round of treatment on shale sample S2.

Shale sample S1 was treated twice by LN<sub>2</sub> under triaxial stresses of  $x : y : z = 1000 : 1500 : 2000$  psi. The first treatment was a 40-minute low pressure ( $\sim 15$  psi) circulation in the borehole, the second treatment consisted of three cycles of 15-second injection at 450 psi. After all cryogenic treatments, permeability evaluation from semilog pressure-time showed that the permeability of shale sample S1 increased to about 3.5 times of the original value of 0.0013 mD right after the cryogenic treatment. Finite difference matching of the pressure decay curves gave a 2.6-fold improvement in permeability (Yao, 2016). Shale sample S3 unexpectedly broke down during LN<sub>2</sub> treatment, so no pressure decay curves were measured.

### 3.2.1 Breakdown Pressure and Fracture Profile

Shale sample S2, was fractured by injecting gas N<sub>2</sub> under triaxial stress loading after two high pressure LN<sub>2</sub> treatments. **Figure 8** shows that the breakdown pressure for shale sample S2 is 1417 psi, which is 41.9% lower than that of the untreated shale sample S4. **Figure 9** shows photographs of cryogenically fractured sample S2 before and after the gas N<sub>2</sub> fracturing. The major fracture propagated along the maximum horizontal stress, which is the horizontal direction here. **Figure 10** shows photographs of the fracture plane in shale sample S2 after the liquid N<sub>2</sub> treatment and gas N<sub>2</sub> fracturing. The outer gas fracture growth direction was slightly deviated

from the inner cryogenic fracture profile, which displays an eggplant shape encircling the wellbore. The inconformity between cryogenic fracture and subsequent gas fracture indicates that cryogenic treatment changed the local stress distribution.

Shale sample S1 stimulated under triaxial stresses of  $x : y : z = 1000 : 1500 : 2000$  psi broke down at 1394 psi, which is 42.8% lower than that of untreated shale sample S4. Shale sample S3 under the same stresses as S1 unexpectedly broke down during  $LN_2$  treatment, after which the pressure decay only built up to 168 psi. More details of these cases can be found in the studies of Alqahtani (2015) and Alqahtani et al. (2016).

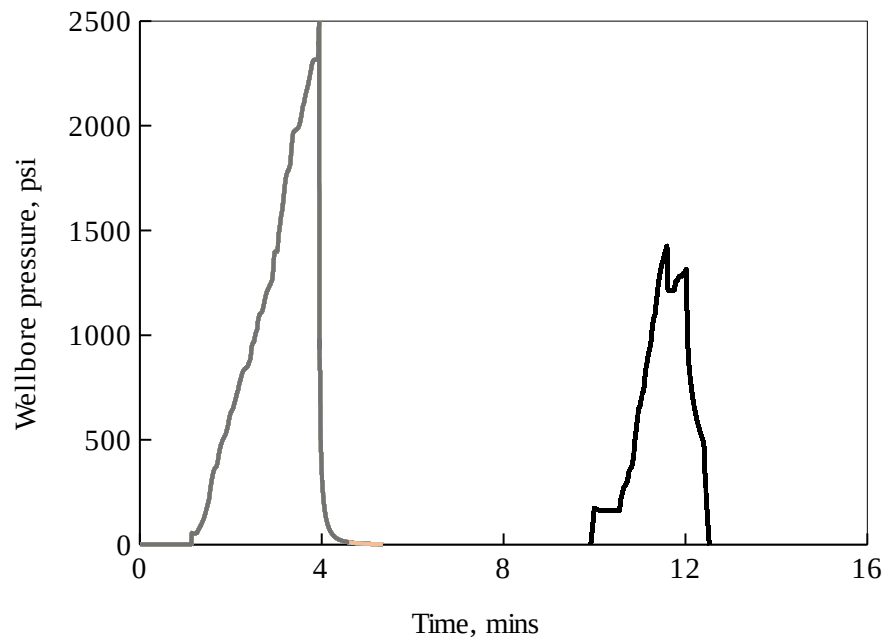


Figure 8. Breakdown pressure profiles for shale samples S2 and S4 using  $GN_2$ .

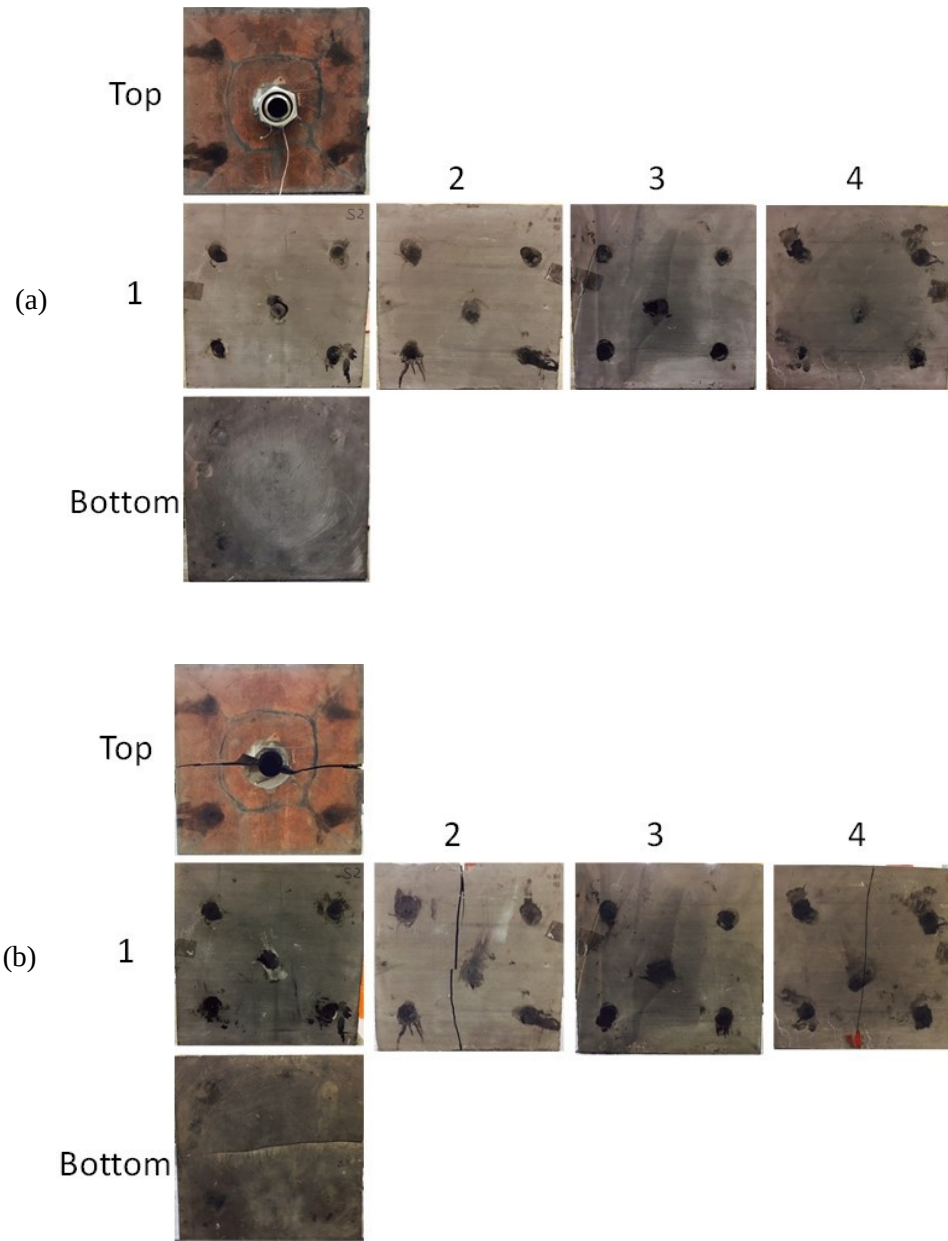


Figure 9. Shale sample S2 before (a) and after (b) the gas N<sub>2</sub> fracturing.

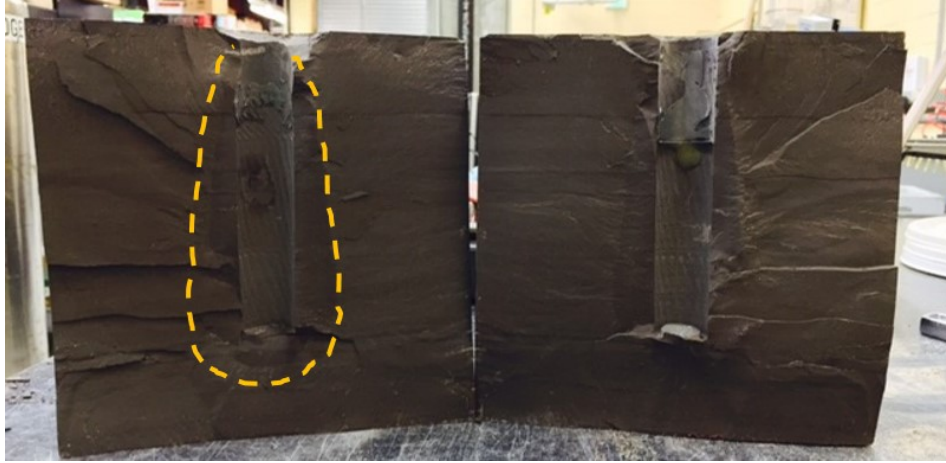


Figure 10. Fracture plane in shale sample S2 after liquid N<sub>2</sub> and gas N<sub>2</sub> fracturing.

### 3.3 Sandstone Samples

In our previous tests under no confining stresses (Cha et al. 2014), four cycles of thermal shock and pressurization were performed on a low permeability sandstone sample obtained from a quarry in Denver, Colorado. XRD analysis showed that average mineralogical composition of the fluvial sandstone beds where these outcrop samples were obtained is 51% quartz, 10% feldspar, 8% carbonate, 26% clay, and 5% others in weight (Pitman et al. 1989). No observable cracks were found on the sandstone surface, suggesting more resistance to thermal shock than concrete samples. After liquid N<sub>2</sub> was injected for 60 minutes, the block surface reached as low as -50 °C and thick frost formed on the side and top faces. Later, bubble agent tests detected several leakage spots on all the side and top faces of the block, which allowed fast gas flow and could be simply high permeability pathways instead of cryogenic fractures. Fortunately, subsequent acoustic measurements helped demonstrate that cryogenic treatments did cause invisible internal structural damage, which retarded the compressional and shear wave velocities through the block (Cha et al. 2014).

We continued to fracture two more sandstone samples: SS1 and SS2, of the same type under true triaxial stresses by LN<sub>2</sub> and GN<sub>2</sub>, respectively. Pressure decay tests were carried out to assess the permeability change after each cycle of 15-second liquid nitrogen treatment to SS1, and finally the sandstone sample was fractured by gas nitrogen. The breakdown pressure of sandstone sample SS1 was compared with that of SS2, which was directly fractured by high pressure gas N<sub>2</sub> without any liquid nitrogen treatment.

### 3.3.1 Pressure Decay Curves and Permeability Enhancement

**Figure 11** shows the pressure decay curves obtained from sandstone sample SS1 before cryogenic treatments under different triaxial stress conditions. The permeability decreased when the sample was compressed under stress loading of  $x : y : z = 1000 : 1500 : 2000$  psi. **Figure 12** shows the pressure decay tests conducted under triaxial stresses for SS1 before LN<sub>2</sub> treatment and after each LN<sub>2</sub> treatment cycle, indicating that there is no distinct observable difference in the pressure decay curves after even 3 cycles of LN<sub>2</sub> treatments. In addition, permeability evaluation with semilog pressure time plots showed an increment of only about 30% right after the third cryogenic treatment. This could be because sandstone has a much higher average permeability than the concrete and shale ( $k_{\text{sandstone}} = 0.349$  mD,  $k_{\text{concrete}} = 0.009$  mD, and  $k_{\text{shale}} = 0.001$  mD), also there were possibly numerous natural fractures present in the sample. Fast leak off of liquid N<sub>2</sub> through high conductivity pores or natural fractures cools the porous medium in a relatively uniform way that cannot generate sufficient local stress to split the sandstone block. It is noteworthy to point out that typical thermal expansion coefficient of quartz, which is a major component in sandstone, is larger than that of carbonate, which is a major component in shale (Robertson, 1988).

**Figure 13** shows the pressure decay curves measured at different temperature conditions while sandstone sample SS1 was still under triaxial stresses. Wellbore pressure decay accelerated as the sample returned to room temperature. Permeability evaluation showed that the sandstone permeability at room temperature is about five times as high as the value right after the cryogenic treatment. At low temperature, the sandstone matrix was under thermal contraction and consequently, the pore sizes and perhaps the natural fractures widths were smaller. Additionally, it is speculated that LN<sub>2</sub> partially destroyed the cement structure among the framework grains, then as the sample returned to room temperature, the slightly shifted grains and cement expanded and self-propped the micro apertures around, preventing their closure. It appears that there were no large cryogenically generated fractures in sandstone sample SS1.

Figure 11. Pressure decay tests under different loading conditions for sandstone sample SS1.

Figure 12. Pressure decay tests before LN<sub>2</sub> treatment on sandstone sample SS1 and after each LN<sub>2</sub> treatment cycle. All tests were conducted under triaxial stress loading.



Figure 13. Pressure decay tests with different temperature conditions for sandstone sample SS1.

### 3.2.1 Breakdown Pressure and Fracture Profile

**Figure 14** shows the breakdown pressure measured during the pressure decay test at the end of the experiment with a slight vertical stress loading of  $z = 60$  psi, as was designed for comparison with the first decay test before any treatments, but the sample broke down at a very low pressure of 215 psi. **Figure 15** presents photographs of sandstone sample SS1 before and after the gas fracturing. It seems that the created fractures followed the paths of the natural fractures, which was initially set perpendicular to the minimum horizontal stress though.

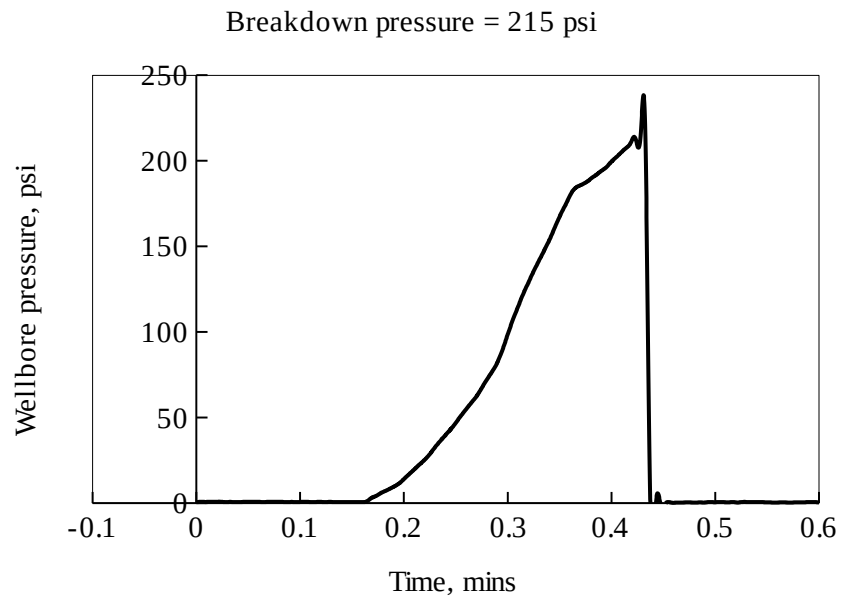
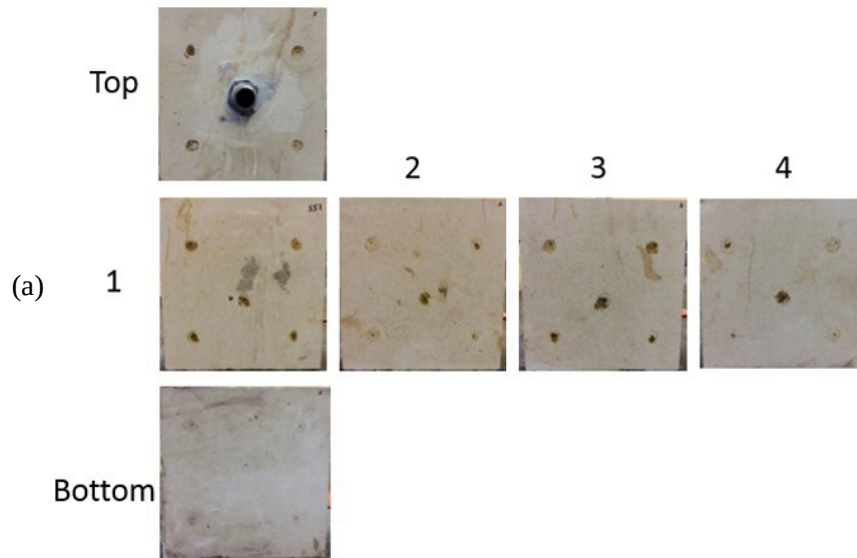


Figure 14. Pressure decay test at the end of the experiment on sample SS1. During this step, the sample fractured at a very low pressure of 215 psi.



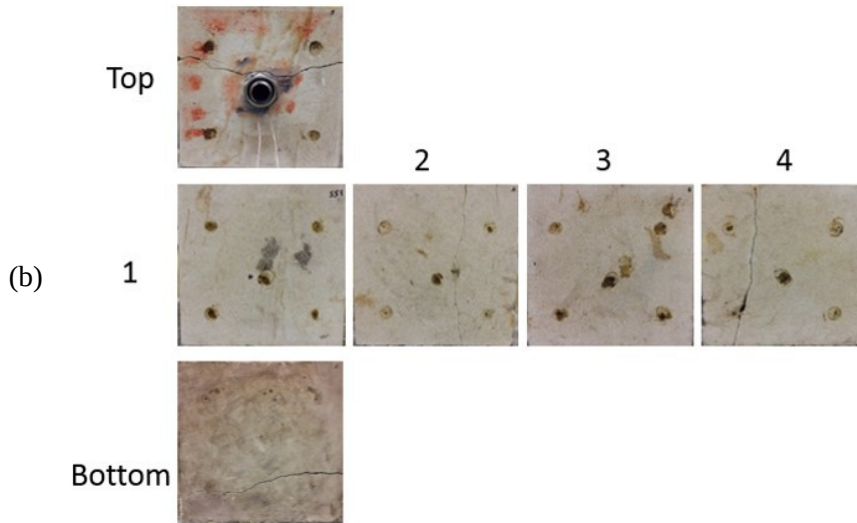


Figure 15. Sandstone Sample SS1 before (a) and after (b) the fracturing. It appears that the created fractures follow the natural fractures.

Sandstone sample SS2 was directly fractured with GN<sub>2</sub> under the vertical stress loading of  $z = 60$  psi. **Figure 16** shows that the breakdown pressure for sandstone sample SS2 is 689 psi, much higher than 215 psi of SS1. This breakdown pressure drop verified the speculation that considerable cryogenic damage to the inter-grain cement bonds was caused inside the sandstone block by liquid nitrogen treatments, although no significant difference was observed from the pressure decay curves. **Figure 17** shows photographs of SS2 before and after the gas fracturing.

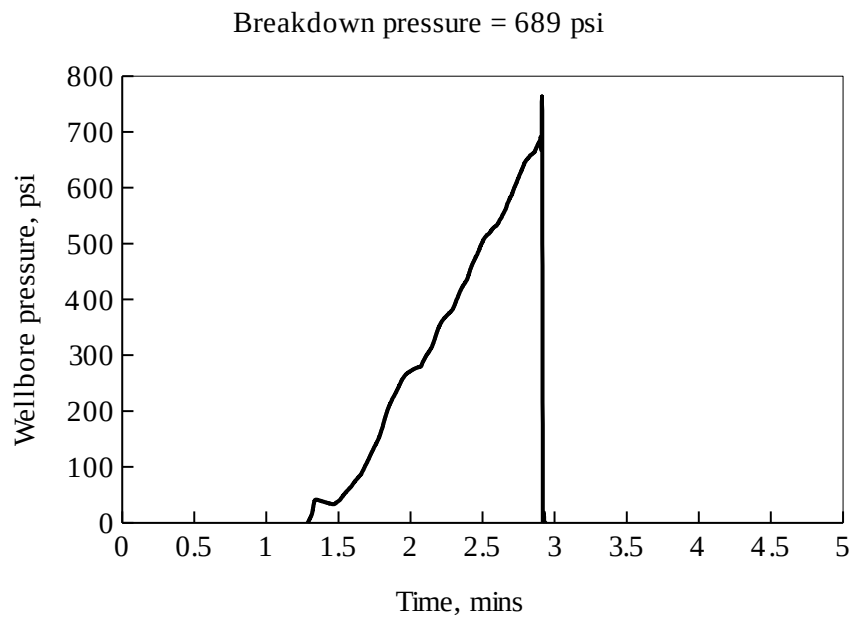
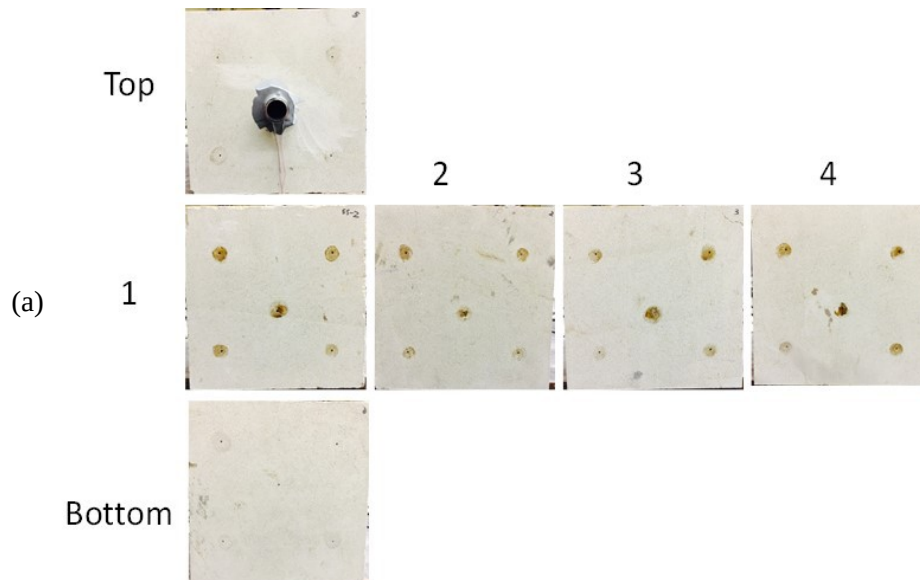


Figure 16. Breakdown pressure for sample SS2 by injecting only GN<sub>2</sub>.



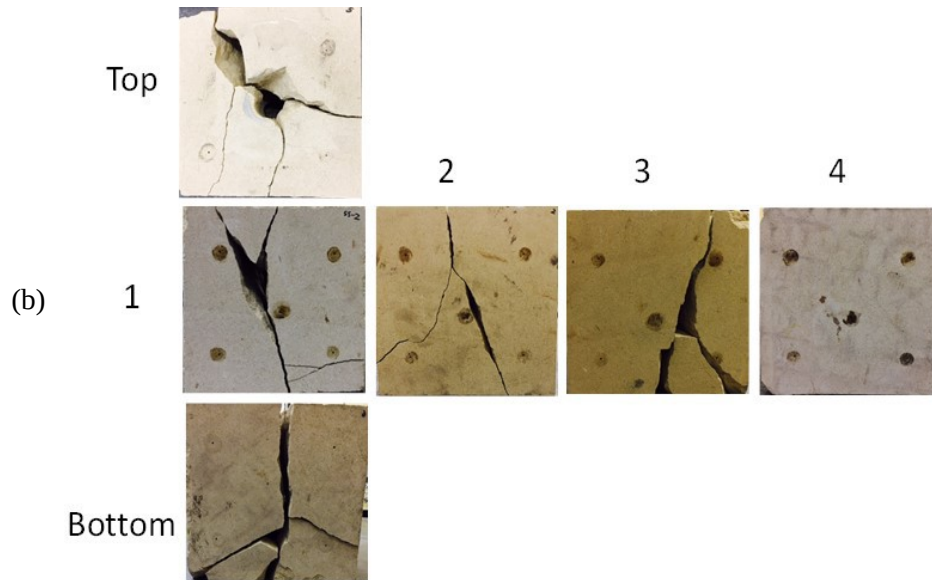


Figure 17. Sandstone sample SS2 before (a) and after (b) the gas nitrogen fracturing. This block exhibited significant fracturing, as can be seen in (b).

#### 4. Discussion

Significant progress has been made in developing waterless fracturing technologies for unconventional reservoirs. The main features of the waterless fracturing technologies discussed are compared in **Table 1**. Conventional oil-based fracturing gains new compatibility with unconventional reservoirs attributing to CO<sub>2</sub> energizing and base fluid recycling. Yet high initial cost, environmental pollution, and safety risks remain concerning. Explosive and propellant fracturing is the least costly treatment considering its small-scale operations; however, field implementations are confined to small treatments due to its limited fracture length and height. Gelled LPG or alcohol is the most expensive fracturing treatment in terms of the fluid availability, but it excels in generating long effective fracture lengths and providing miscibility with hydrocarbons. Safety risks with these flammable fluids should be given special attention. Gas nitrogen fracturing is the most cost-effective and environment-friendly treatment for unconventional wells of shallow depths. Liquid CO<sub>2</sub> fracturing has been proved as an efficient treatment for unconventional wells, still its application is restricted by source, transportation, and recovery of CO<sub>2</sub>. Development of unconventional reservoirs provides an option for CO<sub>2</sub> utilization as well as additional opportunities for CO<sub>2</sub> storage and sequestration. In comparison, cryogenic fracturing is a relatively new fracturing technology that have not been well investigated in either laboratory or field.

Table 1. Comparison of waterless fracturing technologies

Features	Oil/CO <sub>2</sub>	Exp./Prop.	LPG/Alcohol	GN <sub>2</sub>	CO <sub>2</sub>	LN <sub>2</sub>
Eco-friendly	No	Yes	No	Yes	No	Yes
Source abundance	Yes	No	No	Yes	No	Yes
Flow back treatment	Yes	No	No	No	Yes	No
Gel/Clean up	Yes	No	Yes	No	No	No
Wellbore protection	No	Yes	No	No	No	Yes
Proppant transport	Yes	Self-Propped	Yes	Yes	Yes	Yes
Fracture complexity	low	medium-high	low	medium	low	high
Miscibility	high	low	very high	low	medium	low
Safety risk	high	low	high	low	medium	low
Fluid cost	very high	very low	very high	low	high	medium
Reported field cases	hundreds	>1000	>1000	hundreds	>1000	~6

Our laboratory study demonstrated that cryogenic treatment with liquid nitrogen is capable of initiating and generating fractures in reservoir rock blocks. Cryogenic treatment tended to form more complex polygon patterns than conventional hydraulic fracturing, and generally propagated perpendicular to the minimum horizontal stress. Cryogenic treatments of concrete, sandstone, and shale samples all resulted in reducing breakdown pressures for gas fracturing. Gas fracturing after cryogenic treatment is demonstrated to be a feasible technical combination where liquid nitrogen treatment served as a technique to generate seed fractures for other fracturing technologies. Analyses of tests on concrete and shale samples confirm that liquid nitrogen stimulation reduces breakdown pressures 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<sub>2</sub> treatment cycle not only creates new fractures, but also widens the existing ones. In addition, as temperature returned to ambient, the fractures narrowed, as is evidenced by a decrease in permeability. Compared with concrete and shale, sandstone samples did not show obvious enhancement in permeability after each LN<sub>2</sub> treatment cycle, due to very high original permeability. It appears that high permeability pores and existing natural fractures may have diminished the impacts of cryogenic fracturing. On the contrary, when temperature of sandstone returned to ambient, permeability of sandstone increased, as a result of combined effect of thermal relaxation and inter-grain cement damage.

No water and chemical additives were used for fracturing in cryogenic stimulation experiments, which completely avoids the formation damage and environmental concerns caused by water-based fracturing fluids. Formation-damage-free stimulated reservoir volume can provide low-resistant fracture networks that will increase the effective oil and gas drainage area. In addition, no flow back or cleanup is expected after cryogenic fracturing in field, thereby stimulated wells are directly ready to be put into production. In field operations, liquid nitrogen can be directly

obtained by separating and compressing nitrogen gas from air, minimizing the cost for fluid transportation. Thus, time and money are saved in terms of zero water usage, no water storage pits and tanks, smaller site construction, faster development pace, and larger reservoir drainage area, etc.

To be implemented in the field, a few challenges associated with liquid nitrogen injection should be expected. Cryogenic temperature is detrimental to both steel tubing, casing, and cement sheath, thus fiberglass is currently preferred for liquid nitrogen delivery and warm gas nitrogen should be injected downward in the annulus to protect the casing (McDaniel et al. 1997). As temperature rises, cryogenic fractures partially close, therefore proppants are needed to sustain fracture conductivity. However, low viscosity of liquid nitrogen and vaporized gas poses difficulties in sufficient proppant transport. Successful field experience of placing sand proppants during liquid CO<sub>2</sub> fracturing is of great value to technically ensure that liquid nitrogen serves as an efficient proppant carrier. As have been used in gas fracturing, currently available ultralight weight proppant with low specific gravity of 1.08 (Gaurav et al. 2012) should be compatible with liquid nitrogen stimulation. The Leidenfrost effect, when an insulating gas layer develops between the rock and the LN<sub>2</sub>, delays the process of cryogenic fracturing. It may be minimized by optimizing the LN<sub>2</sub> flow rate during the cryogenic treatment (Patterson, 2015).

Through experimental investigation and comparative review of the current waterless fracturing technologies, cryogenic fracturing using liquid nitrogen is demonstrated as an advantageous process for developing unconventional reservoirs in that liquid nitrogen is inert with reservoir rock/fluids and facility, nontoxic, environment-friendly, and can be produced on-site.

## **5. Conclusions**

Based on the review of currently available waterless fracturing technologies and our laboratory study of cryogenic fracturing using liquid nitrogen, the following conclusions can be drawn:

There are several well-developed waterless fracturing technologies, the applications of which are restricted by fracturing fluid source, operating complexity, cost, environmental and safety concerns, etc.

Cryogenic fracturing with liquid nitrogen is demonstrated as a formation-damage-free stimulation technology that can effectively generate fractures in shale and sandstone reservoir rocks.

Permeability enhancement can be achieved after each cycle of liquid nitrogen treatment, and the breakdown pressure of subsequent gas fracturing after cryogenic stimulation can be significantly reduced.

Cryogenic fracturing using liquid nitrogen can either be implemented alone or be combined with other fracturing technologies to cost-effectively enhance the hydrocarbon recovery of unconventional reservoirs.

### **Acknowledgement**

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