

# Breakdown pressure and fracture surface morphology of hydraulic fracturing in shale with $H_2O$ , $CO_2$ and $N_2$

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**Abstract** Slick-water fracturing is the most routine form of well stimulation in shales; however  $N_2$ , LPG and  $CO_2$  have all been used as “exotic” stimulants in various hydrocarbon reservoirs. We explore the use of these gases as stimulants on Green River shale to compare the form and behavior of fractures in shale driven by different gas compositions and states and indexed by breakdown pressure and the resulting morphology of the fracture networks. Fracturing is

completed on cylindrical samples containing a single blind axial borehole under simple triaxial conditions with confining pressure ranging from 10 to 25 MPa and axial stress ranging from 0 to 35 MPa ( $\sigma_1 > \sigma_2 = \sigma_3$ ). Results show that: (1) under the same stress conditions,  $CO_2$  returns the highest breakdown pressure, followed by  $N_2$ , and with  $H_2O$  exhibiting the lowest breakdown pressure; (2)  $CO_2$  fracturing, compared to other fracturing fluids, creates nominally the most complex fracturing patterns as well as the roughest fracture surface and with the greatest apparent local damage followed by  $H_2O$  and then  $N_2$ ; (3) under conditions of constant injection rate, the  $CO_2$  pressure build-up record exhibits condensation between  $\sim 5$  and 7 MPa and transits from gas to liquid through a mixed-phase region rather than directly to liquid as for  $H_2O$  and  $N_2$  which do not; (4) there is a positive correlation between minimum principal stress and breakdown pressure for failure both by transverse fracturing ( $\sigma_3$  axial) and by longitudinal fracturing ( $\sigma_3$  radial) for each fracturing fluid with  $CO_2$  having the highest correlation coefficient/slope and lowest for  $H_2O$ . We explain these results in terms of a mechanistic understanding of breakdown, and through correlations with the specific properties of the stimulating fluids.

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## 1 Introduction

Hydraulic fracturing is a mature completion technique which has been extensively applied in tight and unconventional gas reservoirs. For unconventional reservoirs such as shale with extremely low permeability, long horizontal laterals with multi-staged hydraulic fractures are necessary to deliver economic production. The introduction of hydraulic fractures significantly increases flow rate because of large surface contact area between fractures and the reservoir, enhanced permeability around the wellbore, and reduced fluid diffusion lengths (King 2010; Vincent 2010; Faraj and Brown 2010).

Water-based fluids have become the predominant type of fracturing fluid. Sometimes  $N_2$  or  $CO_2$  gas is combined with the fracturing fluids to form foam as the base fluid. Other additives can also be combined with  $N_2$  or  $CO_2$  to improve the efficiency, e.g. coupling solids-free viscoelastic surfactants (VES) with a carbon dioxide ( $CO_2$ )-emulsified system to further enhance cleanup in a depleted reservoir, extend the application to water-sensitive formations, and maintain reservoir gas saturation to prevent any potential water blockage (Hall et al. 2005); or incorporating low-polymer-loading carboxymethyl guar polymer and a zirconium-based crosslinker to minimize the damage and maximize production (Gupta et al. 2009). For unconventional reservoirs in arid areas the availability of water is sparse. In these cases,  $N_2$ , liquefied petroleum gas (LPG) or  $CO_2$  may become an “exotic” option for stimulation fluid. For example, fracturing with  $CO_2$  has been used in places such as Wyoming where carbon dioxide supply and infrastructure are available (Bullis 2013).

Using  $CO_2$  or  $N_2$  as stimulation fluid has a number of potential advantages. Not only can it eliminate the need for large volume of water—approximately 5 million gallons per treatment—but it can also reduce the amount of wastewater produced and therefore reduce the need for re-injection, which is known to induce seismicity in some cases (Weingarten et al. 2015) and the environmental footprint of these operations. Energized fluids with a gas component can facilitate gas flowback in tight, depleted or water sensitive formations and may be required when drawdown pressures are smaller than the capillary forces in the formation (Friehauf and Sharma 2009; Friehauf 2009). Some recent studies suggest that

using carbon dioxide can also result in a more extensive and interconnected network of fractures, making it easier to extract the resource (Ishida et al. 2012). Other work argues that fractures created with  $N_2$  are more complex than  $CO_2$  which in turn are more complex than those formed by  $H_2O$ , where fracture pattern complexity is based on the ratio of fracture surface area to rock volume, with rough, intricate fracture having high complexity and greater potential to access pore space in tight shales and other formations (Alpern et al. 2013; Gan et al. 2013).

Classic geomechanics models suggest that breakdown pressure is independent of fluid type (composition) or state (gas or liquid) in that failure is controlled by effective stress, alone for a given rock tensile strength (Hubbert and Willis 1957; Biot 1941; Haimson and Fairhurst 1967). However recent research suggests that fluid composition and/or state may have great influence on breakdown pressure (Alpern et al. 2012; Gan et al. 2013). The purpose of this study is to explore the development and behavior of fractures in Green River Shale (GRS) when injected with  $H_2O$ ,  $CO_2$  and  $N_2$ . We focus in particular on breakdown pressure and fracture morphology, including fracture surface roughness and the complexity of the resulting fracture network.

## 2 Experimental method

The introduction and behavior of induced fractures in shale by  $H_2O$ ,  $CO_2$  and  $N_2$  are investigated with respect to breakdown pressures and morphology of the resulting fracture networks. These experiments are conducted on Green River shale.

### 2.1 Approach

Hydraulic fracturing experiments are conducted using intact cylindrical cores containing a blind central borehole ( $\sim 1/10$ -inch-diameter to depth of 1-inch). These experiments measure breakdown pressure and examine the morphology of the resulting fracture. Cores are 1-inch diameter and 2-inches long, sheathed in a jacket, and subjected to mean and deviatoric stresses in a simple triaxial configuration. Multiple cores of GRS are tested with  $H_2O$ ,  $CO_2$  and  $N_2$ . Post-experiment fracture surfaces are measured using a



**Fig. 1** Hydraulic fracturing system. Containment vessel with platen and fluid feed assembly and cell end-caps in foreground

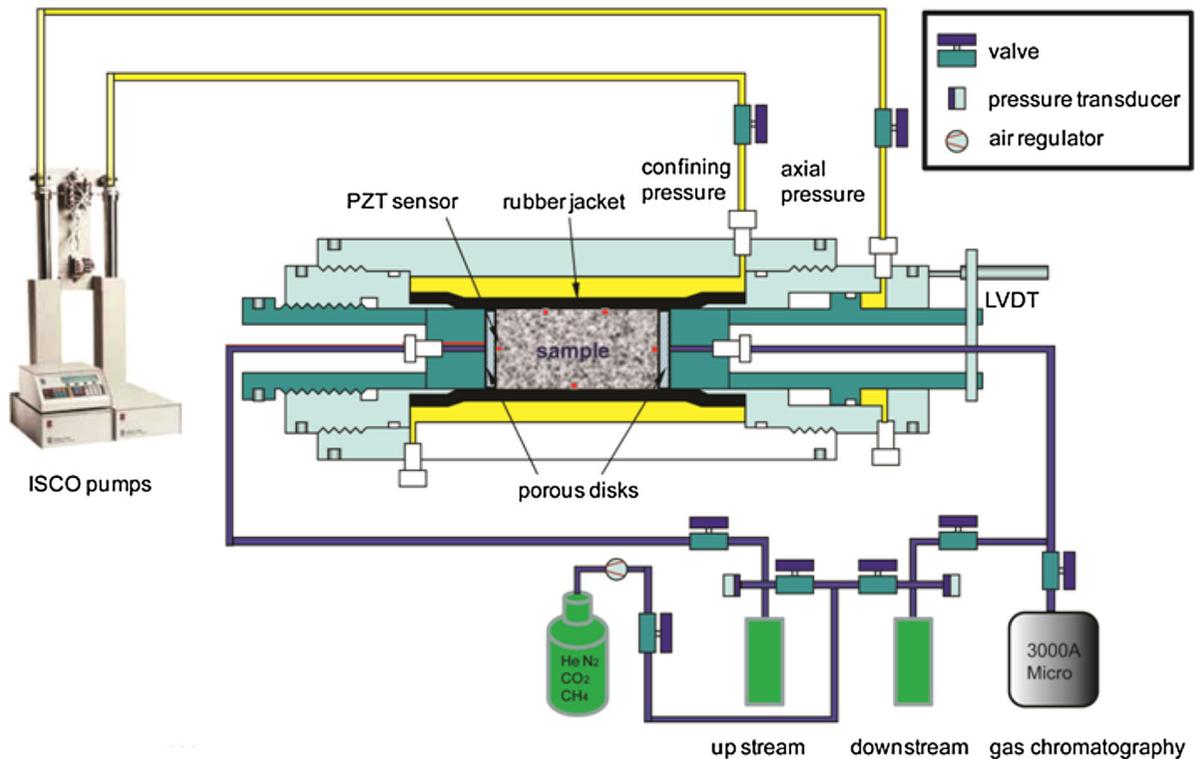
Zygo NewView 7300 scanning white light interferometer for surface roughness and complexity.

### 2.2 Apparatus

All experiments in this study are completed using a standard triaxial apparatus configured for hydraulic fracturing as shown in Figs. 1 and 2. The triaxial core holder (Temco) accommodates the membrane-sheathed cylindrical samples (1-inch diameter and 2-inches long) and applies independent loading in the radial and axial directions via syringe pumps.

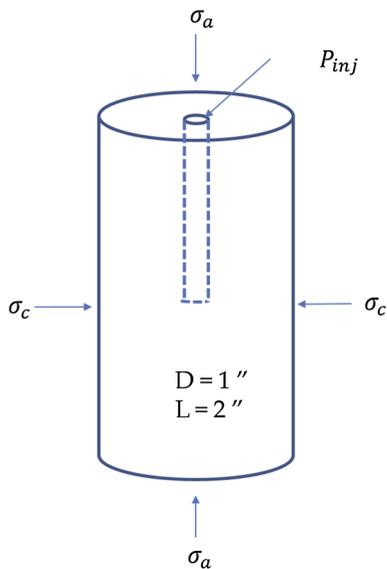
### 2.3 Sample design and seal method

Green River shale samples with a diameter of 1-inch are trimmed by saw to a length of 2-inches and then end-grounded. A central borehole (1/10-inch-diameter) is drilled to a depth of 1-inch (Fig. 3).



**Fig. 2** Schematic of pulse test transient/hydraulic fracturing system (Wang et al. 2011). ISCO pumps supply monitored confining and axial pressure; upstream reservoir supplies injection fluid and the fluid pressure is monitored; downstream is sealed at the *bottom* of the sample; sample is sealed with a

rubber jacket and a porous disk/end plug is used to inject fluid into the sample. This set-up is also capable of acoustic emission and strain measurement as well as gas concentration measurement, however these features are not used in this study)



**Fig. 3** Sample design

Calibration experiments are conducted with 27 samples of GRS to explore the effectiveness of the method of sealing the sample, especially with corrosive and low viscosity  $CO_2$ . Calibration experiments are performed with three methods of sealing (Fig. 4) to ensure congruent results—with the simplest and least invasive of the methods used for the experimental suite. The sealing methods are: (1) a platen with a single concentric O-ring encircling the central injection port (Fig. 4a) (2) a double O-ring design (Fig. 4b); and (3) use of a Swagelok fitting epoxied into the top borehole within the sample (Fig. 4c). Of these, the double O-ring design is the preferred

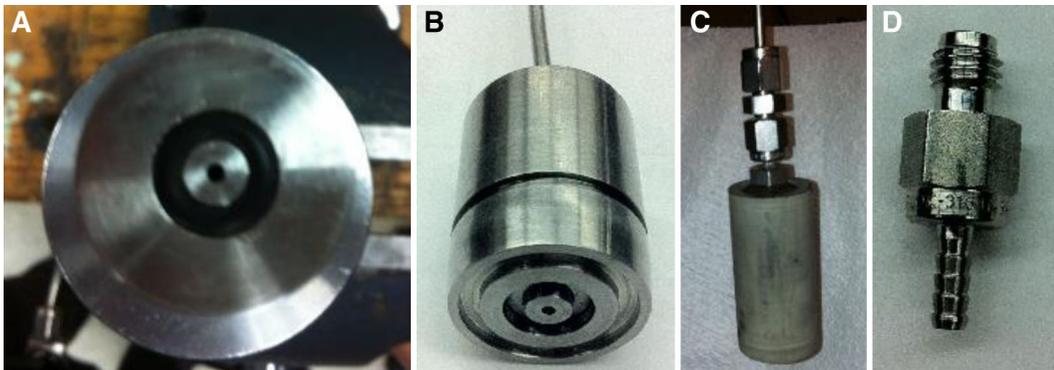
method—simple and adequate. The single O-ring is an effective seal for  $H_2O$  but not for  $CO_2$ . The high pressure fitting is an effective but unnecessary seal compared to the dual O-ring design.

## 2.4 Standard experiment procedure

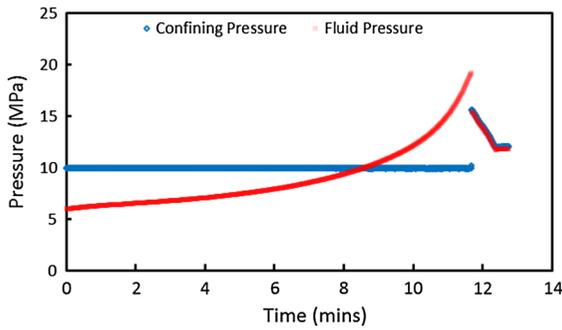
The jacketed sample is placed in the apparatus and axial and confining stresses are applied. Once at the desired pressure, the axial stress is held constant and the pump controlling the confining stress set to constant volume with a pressure relaxation of  $\sim 0.6\%$  which means during the experiment the confining stress can be decreased by  $\sim 0.6\%$  due to the instability of the pump. With confining stress set to constant volume, a rapid increase in confining pressure can also be used as a sign for sample failure. Fluid is then injected into the blind borehole at a constant flow rate (1 ml/min for  $H_2O$ ; 5 ml/min for  $CO_2$  and  $N_2$ ). Breakdown in the sample is observed as a rapid drop in the borehole pressure and a simultaneous jump in the confining pressure (Fig. 5). This defines the breakdown pressure with a typical log shown in Fig. 5.

## 3 Results

Previous studies (Alpern et al. 2012; Gan et al. 2013) have shown that the breakdown pressures and morphology of induced fractures are dependent on both the fracturing fluid and the applied stress regime. We explore the mechanistic underpinnings of these



**Fig. 4** Sealing methods: **a** Single O-ring seal within the platen; **b** Double O-ring seal; **c** Fitting design; **d** Close-up of the fitting with barbs that are epoxied into the blind borehole within the sample



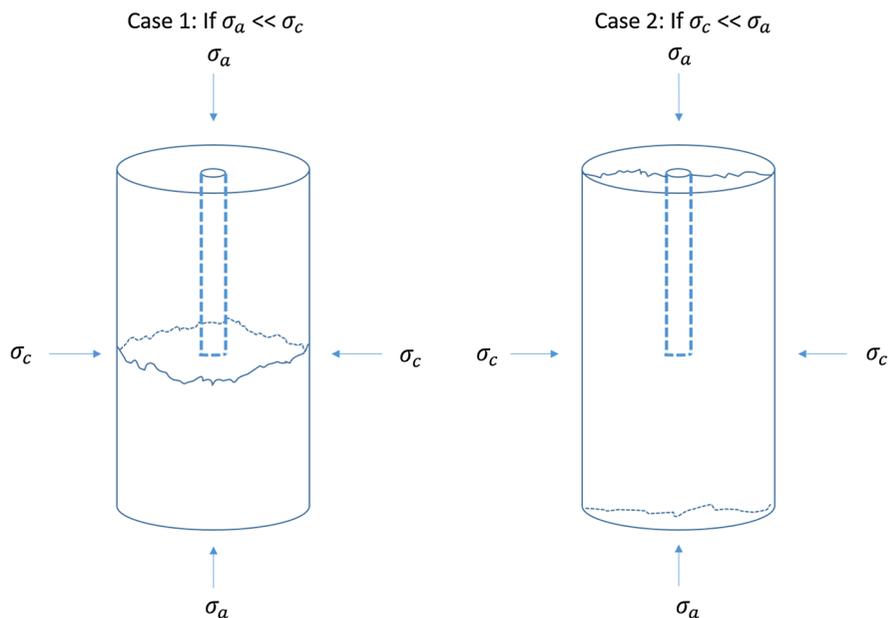
**Fig. 5** Typical pressure response during an hydraulic fracturing experiment. (Sample: Green River shale; Stimulant:  $CO_2$ ; Confining stress: 10 MPa; Axial stress: 20 MPa; Breakdown pressure: 19.3 MPa)

observations in the following, together with their consistency with the observed results in this study.

### 3.1 Theoretical considerations

Hydraulic fractures initiated from a cylindrical borehole in a simple-triaxial stress regime will open against the minimum principal stress (i.e. in the plane of the maximum principal stress). In our configuration, the fractures should develop either across the borehole (Fig. 6 left) when the axial stress is less than the confining stress, or along the borehole (Fig. 6 right) when the axial stress is the maximum stress.

**Fig. 6** Potential failure modes for different stress configurations



When the axial stress is the maximum principal stress (Fig. 6 right), failure is based on the Hubbert and Willis (H–W) hydraulic fracturing criterion where the fracture evolves perpendicular to the local minimum principal stress at the borehole wall, when the rock tensile strength is exceeded. If there is no initial pore pressure in the rock, and assuming an elastic medium, the breakdown pressure is given by:

$$p_b = 3\sigma_{h_{min}} - \sigma_{h_{max}} + \sigma_t \tag{1}$$

where  $p_b$  is breakdown pressure,  $\sigma_{h_{min}}$  is minimum horizontal stress and  $\sigma_{h_{max}}$  is maximum horizontal stress (both perpendicular to the borehole), and  $\sigma_t$  is the tensile strength of the rock.

In our experiments, and for the specific case of the longitudinal fracture of Case 2 then  $\sigma_{h_{min}} = \sigma_{h_{max}} = \sigma_c$  and the breakdown pressure is given by

$$p_b = 2\sigma_c + \sigma_t \tag{2}$$

where  $\sigma_c$  is the confining pressure ( $\sigma_{min} = \sigma_{max} = -\sigma_c$ ). Thus, for these cylindrical samples, the breakdown pressure should be solely a function of confining pressure for a defined tensile strength.

When the axial stress is the minimum principal stress (Fig. 6 left), the sample fails transversely to the borehole. In this case the stress concentration around the tip of the borehole is undefined at the sharp boundary of the borehole termination—acting as a

stress concentrator. Although theoretically undefined and large, it will be limited by blunting of the termination geometry and local failure. In this case the breakdown pressure may be defined generically as

$$p_b = A\sigma_a - B\sigma_c + C\sigma_t \quad (3)$$

where A, B and C are coefficients for axial stress, confining stress and tensile strength. Thus a similar arrangement may be applied to the H–W solution for a longitudinal fracture, with only the magnitudes of the coefficients A and B changing. Absent a stress concentration, the coefficients for Case 1 (when the confining stress is larger) would be  $A = C = 1$  and  $B = 0$ , and for Case 2 (when the axial stress is larger),  $A = 0$ ,  $B = -2$ ,  $C = 1$ .

The results for the above equations are for the case that no fluid penetrates the borehole wall (Hubbert and Willis 1957). Where fluid penetration occurs, based on poroelastic theory considering the poroelastic stress induced by the fluid permeation into rocks (Haimson and Fairhurst 1967), the revised expression for both Cases 1 and 2 may be redefined as:

$$p_b = (A'\sigma_a + B'\sigma_c + C'\sigma_t) \frac{1}{1 + \eta} \quad (4)$$

$$\eta = \frac{v\alpha}{(1 - v)} \quad (5)$$

where  $A'$  is the coefficient for axial stress;  $B'$  is the coefficient for confining stress and  $C'$  is the coefficient on the tensile strength (always unity);  $v$  is the Poisson ratio and  $\alpha$  is the Biot coefficient which reflects the poroelastic effect (Biot 1941);  $\frac{1}{1 + \eta}$  ranges between 0.5 (permeable, where fluid is allowed entry into the borehole wall with  $\eta = 1$ ,  $\alpha = 1$  and  $v = 0.5$ ) and 1 (impermeable, where fluid is excluded from the borehole wall with  $\eta = 0$  and  $\alpha = 0$  which results in Eq. (4) collapsed into Eq. (3)).

Similar to the impermeable cases, when  $\sigma_c < \sigma_a$  the coefficients  $A' = 0$  and  $B' = 2$  for longitudinal fracture (Case 2); when  $\sigma_c > \sigma_a$  and neglecting the stress concentration effect,  $A' = 1$  and  $B' = 0$  for transverse fracture (Case 1).

### 3.2 Experimental results

A large number of experiments are completed on GRS under various stress conditions at ambient temperature. These experiments are completed for the three

fracturing fluids  $H_2O$ ,  $CO_2$  (gas and liquid state) and  $N_2$  (gas state). Results are grouped according to stress conditions and failure modes. For those failing longitudinally where the breakdown pressure is solely a function of confining stress and a given constant tensile strength, breakdown pressures are shown scaled with confining stress (Fig. 7).

Even though the results are somewhat scattered, the general trend is that  $CO_2$  has larger breakdown pressures than  $N_2$ , which in turn has higher breakdown pressures than  $H_2O$ . If interpreted using the concepts (Eqs. 3–5) discussed previously, the magnitudes of the tensile strength are on the order of 4–10 MPa and the multiplier for the confining stress (B) is  $\sim 0.8$ –1.3. The Brazilian test also shows the tensile strength of GRS is  $\sim 10$  MPa (Table 1).

When the samples fail in a transverse mode, ignoring the stress concentration effect, the breakdown pressure is principally controlled by axial stress. Breakdown pressures are shown as a function of axial stress in Fig. 8.

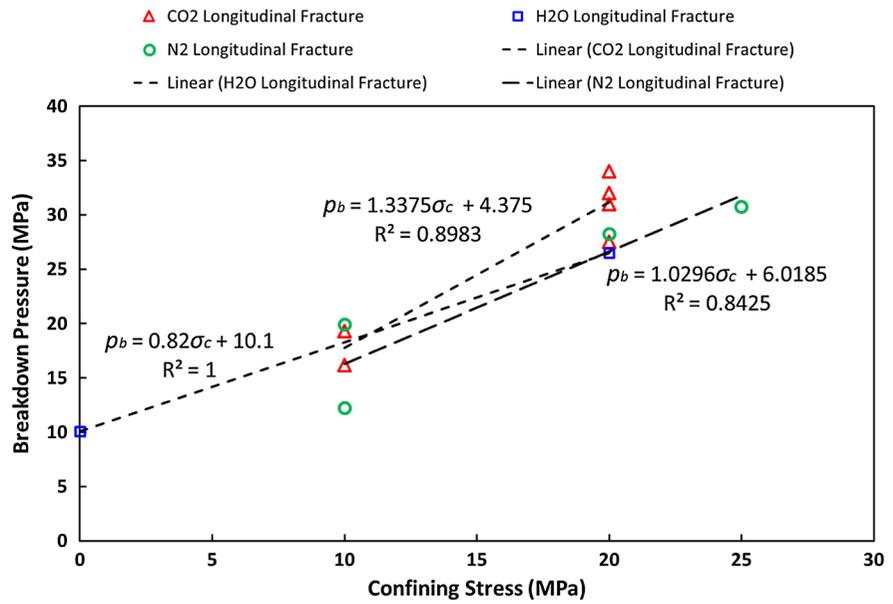
Again, the breakdown pressures are greatest for  $CO_2$ , lower for  $N_2$  and lowest for  $H_2O$ . Projected tensile strength, is in the order of 12–20 MPa. The coefficient of the axial stress (A) is 0.7–1.2.

One thing to notice is that under a constant injection rate, the  $CO_2$  pressure profile presents an extended plateau of constant pressure (Fig. 9b) due to condensation between  $\sim 5$  and 7 MPa. This condensation period implies that the  $CO_2$  transits from gas to liquid via a mixed-phase region. Due to the nature of the other fluids, this is not observed for  $H_2O$  and  $N_2$  (Fig. 9).

### 3.3 Application to other rock types

Extensive attempts have been made to estimate the magnitude of wellbore breakdown pressure through analytical, semi-analytical and numerical approaches (Kutter 1970; Newman 1971; Tweed and Rooke 1973). The suitability of using GRS as an analog for other rock types may be established through comparison of index properties of strength, deformability, porosity and permeability, as well as organic content. These are given in Table 1. More specifically, direct scaling of fracture breakdown is possible when indices of extensional strength (tensile strength) and capillary behavior (scaled from permeability and porosity) are applied.

**Fig. 7** Breakdown pressure as a function of confining stress (Case 2: longitudinal fracture)



The Green River shale is fine-grained, highly laminated, and with low-grade kerogen. Its geomechanical properties are shown in Table 1.

The various responses for breakdown for GRS in each of the configurations are:

Longitudinal fracture ( $\sigma_{\min} = \sigma_c < \sigma_{axial}$ ):

$$CO_2: p_b = 1.34 \sigma_{\min} + 4.4 \text{ MPa} \tag{6}$$

$$N_2: p_b = 1.04 \sigma_{\min} + 6.0 \text{ MPa} \tag{7}$$

$$H_2O: p_b = 0.82 \sigma_{\min} + 10.1 \text{ MPa} \tag{8}$$

Transverse fracture ( $\sigma_{\min} = \sigma_{axial} < \sigma_c$ ):

$$CO_2: p_b = 1.18 \sigma_{\min} + 11.8 \text{ MPa} \tag{9}$$

$$N_2: p_b = 0.66 \sigma_{\min} + 19.7 \text{ MPa} \tag{10}$$

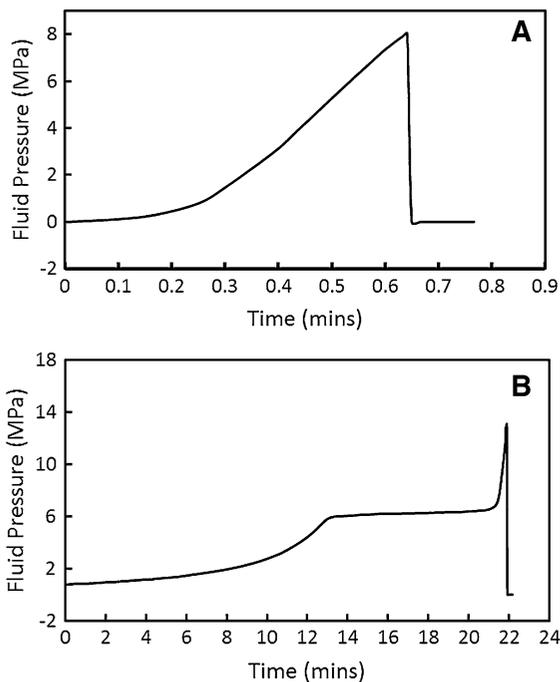
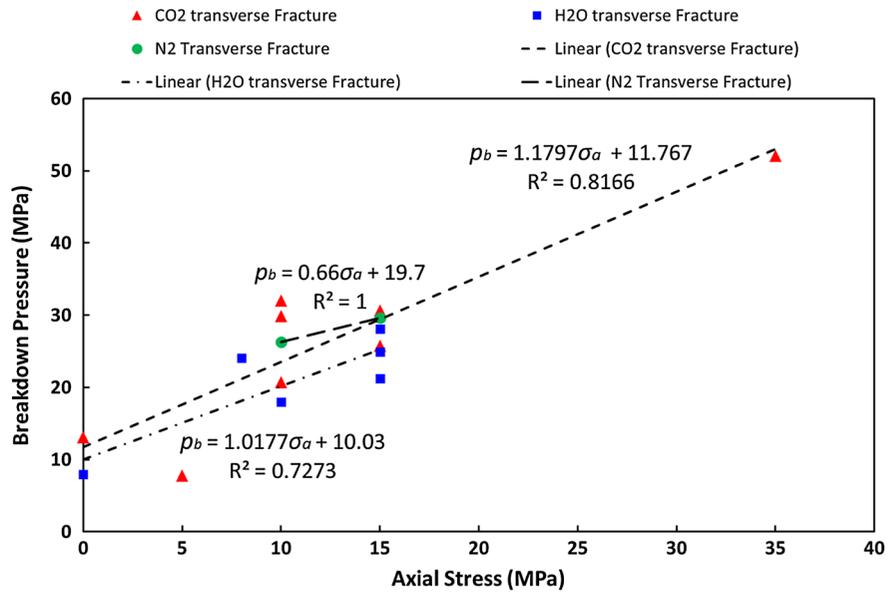
$$H_2O: p_b = 1.02 \sigma_{\min} + 10.0 \text{ MPa} \tag{11}$$

A straightforward interpretation of these breakdown pressure estimates is that the stress offset is proportional or equal to the tensile strength. Further, the variation of the estimates with different confining

**Table 1** Geomechanical properties of Green River shale

| Rock type                    | GRS   |
|------------------------------|---|
| Tensile strength, $\sigma_T$ | 9.3 MPa (load parallel to the bedding) (Li et al. 2015)<br>13.4 MPa (load perpendicular to the bedding)             |
| Young's Modulus, $E$         | 14.4 GPa  |
| Permeability, $k$            | $\sim 10^{-17} \text{ m}^2$ (Culp 2014)   |
| Porosity, $\phi$             | $\sim 10 \%$ (Morgan et al. 2002)   |
| Bulk Modulus, $K$            | 3.5–5 GPa (Bulk modulus of Kerogen) (Yan and Han 2013)<br>1.7–2.5 GPa (Shear modulus of Kerogen) (Yan and Han 2013) |
| Poisson ratio, $\nu$         | 0.2 (Aadnoy and Looyeh 2011)  |
| Elastic moduli ratio, $n$    | 0.84 (Aadnoy and Looyeh 2011)   |
| TOC                          | 17–20 %   |

**Fig. 8** Breakdown pressure as a function of axial stress (Case 1: transverse fracture)



**Fig. 9** Typical fluid pressure profiles for fracturing with **a**  $H_2O$  and **b**  $CO_2$ , which shows gas condensation behavior between  $\sim 5$  and 7 MPa

or axial stresses are due to the stress regime and the stress concentrations around the borehole. Since the borehole configuration remains the same in all

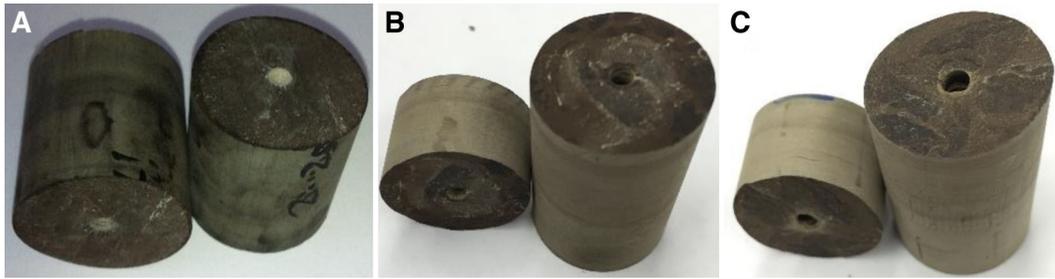
experiments, the results should therefore scale with confining stress and tensile strength.

### 3.4 Fracture surface morphology analysis

Fracture surfaces are measured using a Zygo New-View 7300 scanning white light interferometer with a scan speed up to 135  $\mu\text{m/s}$  and a sub-nanometer resolution. Three samples are fractured with either  $H_2O$ ,  $CO_2$  or  $N_2$ , under a confining stress of 25 MPa and an axial stress of 15 MPa and breakdown pressures measured (Fig. 10). Only one fracture surface of the two halves of each sample is profiled since the fracture surface of the two halves are complementary. Three random spots with a 1.6 mm  $\times$  1.6 mm window are captured from the surface of each sample for measurement (Fig. 11).

#### 3.4.1 Roughness

There are many different surface roughness parameters in use, although arithmetic average of the absolute values of the profile height deviations from the mean line, recorded within the evaluation length ( $S_a$ ; Eq. 12) is the most common. Other common parameters include root mean square (RMS)  $S_q$  and the average distance between the highest peak and lowest valley in each sampling length (Eq. 15)  $S_z$ . RMS is the root mean square average of the profile height deviations



**Fig. 10** Fracture patterns caused by **a:**  $H_2O$ ; **b:**  $CO_2$ ; **c:**  $N_2$ . (Sample: Green River shale; Confining stress: 25 MPa; Axial stress: 15 MPa)

from the mean line, recorded within the evaluation length (Eq. 13). Here fracture roughness is characterized by  $S_a$ ,  $S_q$  and  $S_z$

$$S_a = \frac{1}{n} \sum_{i=1}^n |y_i| \tag{12}$$

$$S_q = \sqrt{\frac{1}{n} \sum_{i=1}^n y_i^2} \tag{13}$$

$$S_t = S_p - S_v \tag{14}$$

$$S_z = \frac{1}{l} \sum_{i=1}^s S_{t_i} \tag{15}$$

where the roughness profile contains  $n$  ordered, equally spaced points along the trace;  $y_i$  is the vertical distance from the mean line to the  $i$ th data point;  $S_p$  is the maximum peak height;  $S_v$  is the maximum valley depth;  $l$  is the number of sampling lengths;  $S_{t_i}$  is  $S_t$  for the  $i$ th sampling length.

The average  $S_a$  value for samples fractured with  $CO_2$ ,  $H_2O$  and  $N_2$  are 18.73, 11.04 and 8.79 microns, respectively. The average  $S_q$  value for  $CO_2$ ,  $H_2O$  and  $N_2$  are 22.94, 13.83 and 11.06 microns, respectively. In Fig. 12 the errors bars indicate the uncertainty of the experiment and variability of the data. Figure 12a, b show that there is a significant difference between:  $CO_2$  versus  $H_2O$ ;  $CO_2$  versus  $N_2$  but  $S_a$  and  $S_q$  are indistinguishable within the uncertainty interval between  $H_2O$  versus  $N_2$ .

The average  $S_z$  value for fracturing with  $CO_2$ ,  $H_2O$  and  $N_2$  hydraulic fracturing (HF) samples are 141.23, 103.39 and 87.77 microns. Figure 12c shows that there is a significant difference between:  $CO_2$  versus  $N_2$  but  $S_z$  is indistinguishable within the uncertainty interval between  $H_2O$  versus  $N_2$ ;  $CO_2$  versus  $H_2O$ .

Overall, the 2 figures above show that the HF surfaces for  $CO_2$  have the highest roughness, followed by  $H_2O$ , and then  $N_2$ .

### 3.4.2 Complexity

Fracture complexity can be evaluated by considering the fractal dimension. The fractal characteristics of the artificial fractures has been investigated using the spectral method (Power and Durhum 1997), which describes the relation between the logarithms of power spectral density (PSD) and spatial frequency as linear for a fractal, with the slope of the line giving the fractal dimension (Fig. 13). When the PSD of the surface heights  $G(f)$  is given as a function of the spatial frequency  $f$  by

$$G(f) = Af^{-\alpha} \tag{16}$$

the fractal dimension of the surface ( $1 < D < 2$ ) is determined by

$$D = \frac{5 - \alpha}{2} \tag{17}$$

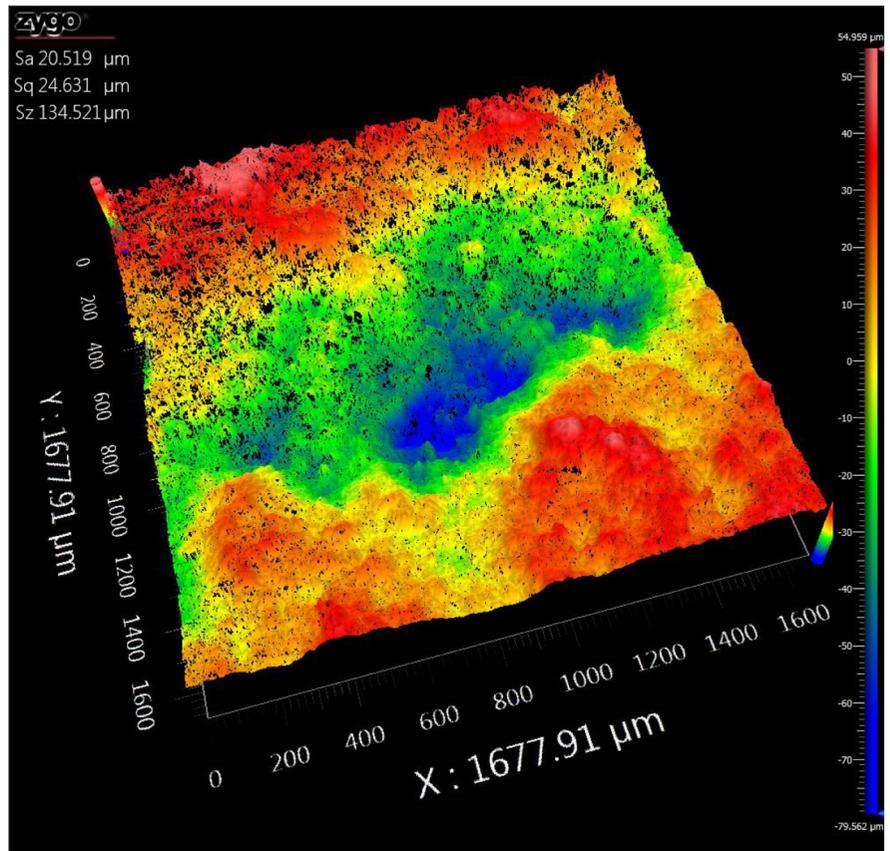
where  $\alpha$  is the power in Eq. 16, determined from the slope of the log–log plot of  $G(f)$ . Therefore the fractal dimension of the fracture surface in Fig. 11 is determined to be 1.84, since  $\alpha$  is 1.323.

The 1-D fractal dimension along the  $x$  and  $y$  direction of each measurement are shown in Table 2.

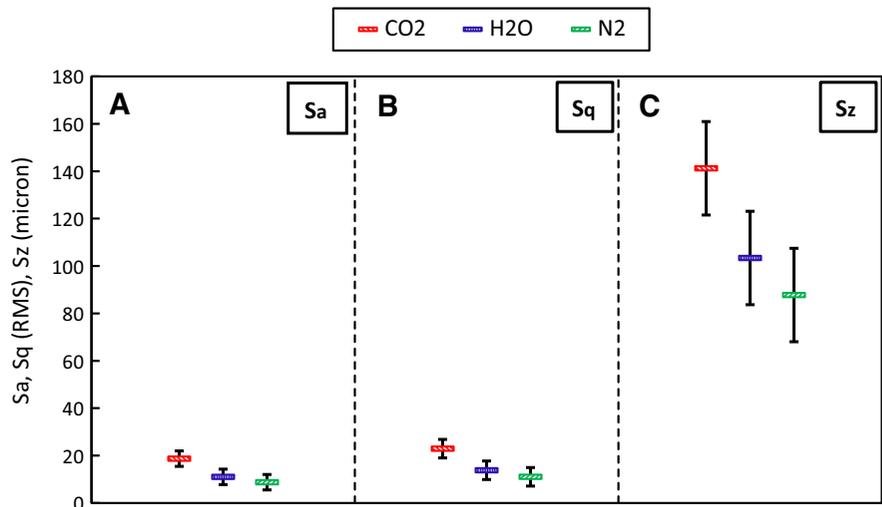
### 3.4.3 Other statistics

The mean and standard deviation of the distance away from the measuring mean plane are also calculated for statistic purpose. All of the fractures resulting from the three fracturing fluids have very similar mean values which is approximately zero (Table 3), and showing a

**Fig. 11** The view of fracture surface on sample fractured with  $CO_2$



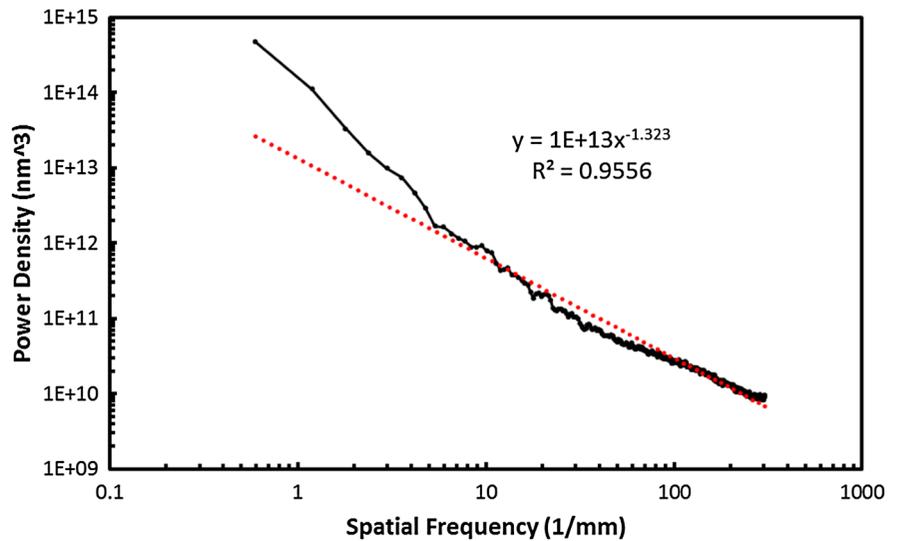
**Fig. 12**  $S_a$ ,  $S_q$  (RMS) and  $S_z$  of fracture surface fractured with  $H_2O$ ,  $CO_2$  and  $N_2$



normal distribution. However  $CO_2$  has the highest standard deviation, indicating that the data points are spread out over a wider range of values, compared to

$H_2O$  and  $N_2$  whose data points tend to be closer to the mean. These data also support that  $CO_2$  HF surface is the most complex and roughest.

**Fig. 13** Relationship between the logarithm of power spectral density and spatial frequency. ( $CO_2$  HF sample; #1 measurement; x direction)



**Table 2** 1-D fractal dimension along the x and y direction for each measurement

|                               | $CO_2$    | $H_2O$    | $N_2$     |
|-------------------------------|-----------|-----------|-----------|
| Fractal dimension, $D_{1(x)}$ | 1.84–1.91 | 1.75–1.83 | 1.71–1.8  |
| Fractal dimension, $D_{1(y)}$ | 1.77–1.85 | 1.8–1.84  | 1.73–1.81 |

Overall,  $CO_2$  HF surfaces have the highest fractal dimension, followed by  $H_2O$ , and then  $N_2$ . Hence,  $CO_2$  HF surfaces are the most complex ones, followed by  $H_2O$ , and then  $N_2$

#### 4 Discussion

A large number of experiments completed in Green River shale indicate the following:

1. Under the same applied stress conditions,  $CO_2$  returns the highest breakdown pressure, followed by  $N_2$ , and then  $H_2O$ . The distribution in breakdown pressures is of the order of ~25–~30 % of the maximum breakdown pressure for this progression of fluids from highest (with  $CO_2$ ) to lowest (with  $H_2O$ ). Under the same conditions of in situ rock stress and flow rate,  $CO_2$  has the higher breakdown pressure

compared to  $N_2$ , possibly in part due to its higher viscosity (Ishida et al. 2012) and higher molecular weight (Alpern et al. 2012). In our study another reason for  $CO_2$  having higher breakdown pressure compared to  $H_2O$  could be attributed to higher flow rate (5 ml/min for  $CO_2$ ; 1 ml/min for  $H_2O$ ) of  $CO_2$  injection (Schmitt and Zoback 1993) (Garagash and Detournay 1997). For  $CO_2$  fracturing, the pore pressures cannot be recharged during the short time of the rapid pressurization with infiltration, placing the sample at a higher effective confining stress and making it both stiffer and more difficult to break. Since the initial pore pressure within the sample is zero in our experiments and the flow of fluid exerts an equivalent body force on the medium, the higher pore pressure gradient from the borehole wall to the outer boundary of the sample may result in larger induced compressive infiltration stresses at the borehole wall which must be overcome in order to initiate fracture. Lubinski (1954) suggests that the magnitude of the compressive stress produced by fluid

**Table 3** The mean and standard deviation of the distance away from the measuring mean plane

|                    | $CO_2$                | $H_2O$               | $N_2$                 |
|--------------------|-----------------------|----------------------|-----------------------|
| Mean (mm)          | –0.00781 to –0.028342 | –0.00752 to –0.01226 | –0.03353 to 0.0033136 |
| Standard Deviation | 0.4784 to 0.79343     | 0.375587 to 0.52368  | 0.300645 to 0.505261  |

Detailed statistics of each measurement can be found in Table 4

**Table 4** Statistics of each measurement

| Parameter (unit)                     | CO <sub>2</sub> |          |          | H <sub>2</sub> O |          |          | N <sub>2</sub> |           |           |
|--------------------------------------|-----------------|----------|----------|------------------|----------|----------|----------------|-----------|-----------|
|                                      | #1              | #2       | #3       | #1               | #2       | #3       | #1             | #2        | #3        |
|                                      | Sa (μm)         | 20.519   | 12.405   | 23.251           | 10.085   | 13.796   | 9.233          | 9.168     | 8.214     |
| Sq (RMS) (μm)                        | 24.631          | 15.488   | 28.705   | 12.369           | 17.611   | 11.496   | 11.310         | 10.586    | 11.294    |
| Sz (μm)                              | 134.521         | 110.942  | 178.226  | 96.894           | 125.042  | 88.225   | 72.992         | 75.678    | 114.626   |
| Mode (mm)                            | 12.249          | 3.410720 | 3.1729   | -4.6531          | 4.6502   | -0.9059  | 1.7671         | -1.7399   | 1.1852    |
| Median (mm)                          | 2.83253         | 1.19189  | 0.4995   | -0.2928          | 0.2737   | 0.41743  | 0.30731        | -0.2264   | 0.038935  |
| Mean (mm)                            | -0.028342       | -0.00781 | -0.02127 | -0.01226         | -0.00752 | -0.01055 | -0.03353       | 0.0033136 | -0.026538 |
| SD                                   | 0.596503        | 0.4784   | 0.79343  | 0.4371           | 0.52368  | 0.375587 | 0.300645       | 0.313302  | 0.505261  |
| Fractal dimension, D <sub>1(x)</sub> | 1.84            | 1.86     | 1.91     | 1.79             | 1.83     | 1.75     | 1.77           | 1.8       | 1.71      |
| Fractal dimension, D <sub>1(y)</sub> | 1.85            | 1.84     | 1.77     | 1.8              | 1.84     | 1.80     | 1.73           | 1.81      | 1.73      |

infiltration is directly in proportion to the difference between injection fluid pressure and far-field pore pressure (Eqs. 18, 19). The effect is similar to the temperature gradient on an elastic medium, as

$$\sigma_{fr} = (n - 1)\beta \left( \frac{1 - 2\nu}{1 - \nu} \right) \frac{1}{r^n} \int_{r_w}^r (p - \beta p_0) r^{n-1} dr \tag{18}$$

$$\beta = 1 - \frac{K}{K_m} \tag{19}$$

where  $\sigma_{fr}$  is the induced compressive infiltration stresses (also known as seepage stress);  $n = 2$  (cylindrical flow) or 3 (spherical flow);  $\beta$  describes the compressibility of the material at some level;  $K$  is the bulk modulus of the porous material;  $K_m$  is the bulk modulus of the inter-pore material;  $\nu$  is the Poisson’s ratio;  $r$  is the current radial location;  $r_w$  is the wellbore radius;  $p$  is the pore pressure at a distance  $r$  away from the wellbore;  $p_0$  is the far-field pore pressure. Therefore diminished pore pressures within the rock matrix results in higher compressive infiltration stresses which can act against, and therefore further postpone, fracture as observed for the CO<sub>2</sub> tests with fast injection rate.

A very subtle pore pressure drop (usually around 0.2–0.3 MPa) just before failure is observed in most of the CO<sub>2</sub> experiments, providing another piece of evidence for diminished pore pressure. This drop in pore pressure is known as “dilatancy hardening” in compressive failure tests (Brace and Martin III 1968) where it is defined as a consequence of new porosity produced in the irreversible damage of the rocks prior to failure. These slight drops in pore pressure before failure might indicate dilatancy of the rock occurred immediately prior to failure as well as increased permeability which possibly results from the production of dilatant porosity such that conductive paths within the rock structure are enhanced and fluid would be expected to flow into highly porous regions. Another possible reason for CO<sub>2</sub> having the highest breakdown pressure followed by N<sub>2</sub> and then H<sub>2</sub>O could be stress corrosion (Anderson and Grew 1977). In this case, time-

dependent chemical reactions aid in bond breaking near the initial crack tip. In our experiments, tensile failure of the samples typically take  $\sim 20$  min for  $CO_2$ ,  $\sim 12$  min for  $N_2$ , and  $\sim 2$  min for  $H_2O$

2. Fracturing with  $CO_2$ , compared to other fracturing fluids, creates marginally more complex fracturing patterns (characterized by fractal dimension) as well as the roughest fracture surface (characterized by  $S_a$ ,  $S_q$  and  $S_z$ ) and with the greatest apparent local damage, followed by  $H_2O$  and then  $N_2$ . Study shows that low viscosity fluid tends to generate cracks extending more three dimensionally with a larger fractal dimension Ishida et al. (2004, 2012). This viscosity dependent behavior can be explained through the fluid loss equation, which indicates that a fracturing fluid with high viscosity results in a low rate of fluid-loss. Carter (1957) assumed that, for a fracture of uniform width that the fluid-loss velocity normal to the fracturing faces,  $v_l$ , takes the following form

$$v_l = \frac{K_l}{\sqrt{t - \tau}} \quad (20)$$

where  $v_l$  is the fluid-loss velocity;  $K_l$  is the overall fluid-loss coefficient and  $t$  is the current time;  $\tau$  is the time when filtration starts.  $K_l$  includes three effects: (1) viscosity and relative-permeability effects of the fracturing fluid, (2) reservoir- fluid viscosity-compressibility effects, (3) wall-buildup effects. Howard and Fast (1957) proposed that the relation between  $K_l$  and viscosity is as following

$$K_l \propto \frac{1}{\sqrt{\mu}} \quad (21)$$

Thus fracturing fluid with high viscosity results in low fluid-loss rates and hence less leak-off from the generated fracture plane. Therefore the pressure in the produced fracture can readily increase with the viscous fluid. Thus, the extension of a less complex fracture develops as a consequence. Among these three fluids,  $N_2$  has the smallest viscosity, followed by  $CO_2$  and then  $H_2O$ . These considerations support well the observation of  $CO_2$  HF surfaces being more complex than  $H_2O$  HF surfaces, but does not necessarily explain

why  $N_2$  HF surfaces are the least complex. Other literature also provides some other explanations that the geometry and dimensionality of some fractures may be a function of the fracture initiation point/borehole termination, failure pressure, and the physical characteristics of the testing material (Culp 2014).

3. Under a constant injection rate, the  $CO_2$  pressure response shows a long plateau of constant pressure due to condensation between  $\sim 5$  and 7 MPa. This condensation period implies the transit of  $CO_2$  from gas to liquid through a mixed-phase region. Due to the features of the other fracturing fluid, this is not observed for  $H_2O$  and  $N_2$ .
4. There is a positive correlation between minimum principal stress and breakdown pressure for failure in both transverse fracturing ( $\sigma_3$  axial) and longitudinal fracturing ( $\sigma_3$  radial).  $CO_2$  has the highest correlation coefficient/slope and  $H_2O$  has the lowest. This observation can be explained with the specific properties of the stimulating fluids and experiment conditions as illustrated in notation 1.

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