

# The effect of CO<sub>2</sub>-related diagenesis on geomechanical failure parameters: fracture testing of CO<sub>2</sub>-altered reservoir and seal rocks from a natural analog at Crystal Geyser, Utah

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## ABSTRACT:

Geomechanical testing results on samples collected from the Crystal Geyser field site, Utah indicate that sandstones naturally bleached by CO<sub>2</sub>-rich fluids have significantly lower fracture toughness values than adjacent, unaltered sandstones whereas altered and unaltered caprock siltstones collected from the site yield similar values. Both the short rod (SR) and double torsion (DT) test methods give comparable results for multiple test specimens from the same samples. CO<sub>2</sub>-related alteration at the Crystal Geyser field site is reflected in the bulk mineralogy of siltstone samples collected near fossil CO<sub>2</sub> seeps in fault-perpendicular transects and primarily characterized by elevated calcite near the fault. Dissolution of Fe-cements, along with the remobilization and precipitation of secondary carbonates and clays noted by petrography, are likely responsible for the geomechanical weakening effect in sandstones. Subcritical crack index, another fracture parameter obtained from the DT test, is also affected by CO<sub>2</sub>-related alteration. The effect of chemical environment (i.e. CO<sub>2</sub>-rich) on fracture toughness is not significant for the sandstone samples tested. These results have implications for the CO<sub>2</sub>-sequestration and are an example of both coupled chemical-geomechanical processes and time-dependent geomechanical behavior.

## 1. INTRODUCTION

The injection of supercritical CO<sub>2</sub> into brine aquifers for carbon sequestration, and the subsequent dissolution of CO<sub>2</sub> into the brine will significantly shift the subsurface geochemical environment in and around target reservoirs away from chemical equilibrium [1]. This will lead to chemical reactions between CO<sub>2</sub>-ladden brine and reservoir and seal rocks [1]. It is well known that fracture processes can be aided by chemical fluid-rock interactions in chemically reactive environments [2, 3]. It is thus reasonable to expect that fracture processes in reservoir and seal rocks could be influenced by the dissolution of CO<sub>2</sub> into the reservoir brine, which then affects fracture growth and thus the flow of CO<sub>2</sub> in the reservoir. Perhaps more importantly, chemically aided fracture growth could result in top seal failure thus controlling leakage behavior. Yet, many current models for CO<sub>2</sub> sequestration consider chemical and mechanical processes in CO<sub>2</sub> reservoirs as separate rather than coupled.

In this study, we investigate the effect of chemical alteration or diagenesis on reservoir and seal rocks

through geomechanical testing. Samples tested in this study were obtained from the Crystal Geyser site in Utah where natural CO<sub>2</sub> leakage resulted in alteration of a suite of lithologies exposed there, allowing geomechanical testing and comparison in rock mechanical properties of CO<sub>2</sub>-altered against unaltered rock. We consider rock altered under natural conditions and time scales as possibly more representative of the end-products of CO<sub>2</sub>-related alteration in CO<sub>2</sub> reservoirs than samples reacted over short laboratory time scales.

We measured two rock fracture mechanical parameters, fracture toughness  $K_{IC}$  and the velocity exponent  $n$  for subcritical fracture growth as defined by

$$V = A \left( \frac{K_I}{K_{IC}} \right)^n \quad \text{Eq 1.}$$

where  $V$  designates the subcritical fracture propagation velocity,  $K_I$  is the mode-I stress intensity factor,  $K_{IC}$  is the mode-I critical stress intensity factor or fracture toughness, and  $A$  is a pre-exponential constant. The subcritical index  $n$ , describes the velocity dependence of fractures propagating under subcritical conditions [4, 5]. Numerical models have shown that the subcritical fracture index impacts the geometry of evolving fracture

patterns, with increasing subcritical index resulting in transitions from irregularly spaced fractures, to regularly spaced fractures, and finally to clustered sets, thus controlling fracture network geometry, connectivity, and flow properties [6, 7]. Subcritical crack growth, which is favored under chemically reactive conditions [4] could thus be significant in controlling leakage from CO<sub>2</sub> reservoirs. Alternatively, it may be inhibited in certain chemical environments.

This paper presents results on fracture toughness and subcritical fracture testing on reservoir and seal rocks that were altered by CO<sub>2</sub>-rich brine in a natural system, comparing results to unaltered rocks of the same rock formation. Samples of altered and unaltered mineralogical composition were also analyzed using x-ray diffraction (XRD) for reference. Fracture testing was performed using two techniques, double torsion and short rod fracture testing, to evaluate the influence of test geometry on fracture parameters.

## 2. METHODS

### 2.1. X-ray diffraction

Over 50 samples from the Crystal Geyser field site were analyzed using powder X-ray diffractometry (XRD) across a range of lithologies using a Bruker D8 Advance X-ray at UT-Austin, but only those data relevant to this study are reported here. A 1:6 internal corundum standard was added to aid peak correction and to verify quantitative data. Samples were crushed and prepared into powders for quantitative analysis using the spray dry technique [8]. Peaks were first identified utilizing Bruker EVA software followed by quantitative Rietveld analysis refinement for quantitative mineralogy using TOPAS software.

### 2.2. Double torsion fracture test method

A double torsion test apparatus in the Center for Petroleum and Geosystems Engineering at UT-Austin was used for calculating fracture toughness and subcritical index values for sandstone and siltstone samples. The double torsion test apparatus and methodologies [9, 10] were first developed for calculating fracture toughness and subcritical index of brittle materials. Methods have been further adapted for use in sedimentary rocks [11]. Multiple test specimens (thin wafers: ~1.2" x 3" x 0.07") were sliced from each sample and a ~0.02" groove was cut lengthwise along the center of each specimen. Individual test specimens typically allow for multiple (2-5) runs consisting first of a "pre-crack" loading run followed by several subsequent load-relaxation runs for measuring subcritical crack growth until failure. Data reduction consisted of calculating fracture toughness from the maximum stress intensity factor at peak loads and subcritical index from the load decay curves using MATLAB scripts.

### 2.3. Short rod method

Over 40 test specimens from three samples (siltstone, sandstone, bleached sandstone) were prepared and run following Level 1 testing [12] with several modifications. The chevron notch was cut using a diamond wire saw and metal grips were attached to the top of the sample with epoxy which was then cured overnight for maximum strength and stiffness. Samples were tested in room-dry conditions using an ADMET mini-load frame at Sandia National Laboratories which applied a constant displacement with both load and displacement measurement. Fracture toughness was calculated from the maximum load and data corrections for sample geometry variations were applied [12]. Given the weakness of the rock, the torque effect of fixed sample grip was deemed negligible.

#### 2.3.1. Testing under controlled environmental conditions

Short rod specimens were suspended within the mini load frame during testing, which allowed it to be immersed in a variety of fluids (Figure 1). A plastic cup was placed under the load frame and metal tubing was bent into the cup in order to bubble CO<sub>2</sub> gas during testing. The gas was bubbled in for at least 5 minutes prior to testing in order to equilibrate with the 10,000 ppm NaCl brine used (pH dropped from ~6 to ~4). A 10,000 ppm brine was chosen reflecting a typical minimum salinity of planned target reservoirs for CO<sub>2</sub> storage. Samples were immersed in the fluid and testing began within 2 minutes.

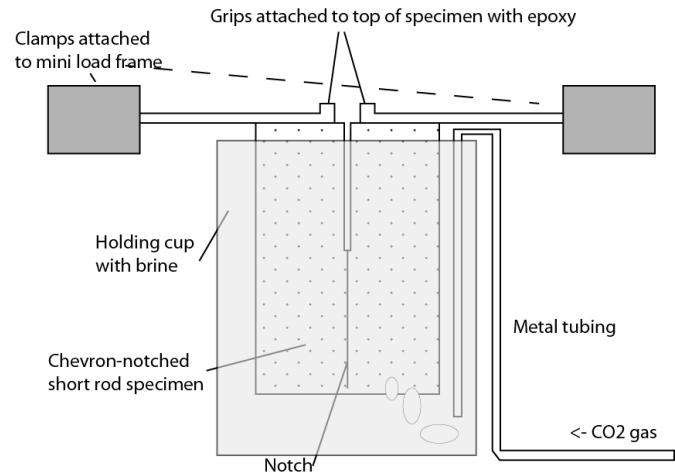


Figure 1. Schematic diagram of experimental setup utilized for testing the effect of chemical environment using the short rod method.

**Table 1.** Bulk mineralogy of samples collected in a fault-perpendicular transect away from a fossil CO<sub>2</sub> seep at the Crystal Geyser field site, Utah, analyzed by XRD. Samples for geomechanics were taken along this transect.

Distance from fault (m)	Illite-smec					other
	Cal	Qtz	Orth	Arag		
0	38	11	34	7	5	5
2	32	8	36	11	8	6
10	33	12	28	10	8	9
20	40	17	31	4	7	2
32	14	19	36	14	9	9
50	10	32	34	16	3	5

### 3. RESULTS

#### 3.1. Bulk mineralogy

The Summerville formation consists primarily of siltstone with minor shale and thin beds of limestone [13]. The siltstone is primarily composed of quartz, illite-smectite, and feldspar plus other minor phases. The mineralogy of siltstone samples taken in a cross-fault transect (Table 1) shows that CO<sub>2</sub>-related alteration has significantly changed the bulk composition of the rocks near a fossil CO<sub>2</sub> seep. Calcite varies by a factor of 4, comprising 30-40 wt% in a 20 m wide zone nearest the fault, but dropping to only 10 wt% tens of meters away from the fault. No clear trend is evident in aragonite, which is another mineral typically predicted to be associated with CO<sub>2</sub>-rich fluids. Illite is relatively low in abundance near the fault but increases away from the fault, whereas the silicates quartz and orthoclase show no major trends.

#### 3.2. Fracture testing

The results of two different test methods across multiple sample lithologies are presented below that document the significant effect of CO<sub>2</sub>-related alteration on rock geomechanical properties.

##### 3.2.1. Comparison of results from the double torsion and short rod methods

Test results show general agreement of fracture toughness values obtained two lithologies, sandstone and siltstone, using both the short rod and double torsion test methods (Figure 2). Calculated average K<sub>IC</sub> values of one method are within the 1 $\sigma$  error of the other, meaning they are statistically indistinguishable based on test data obtained.

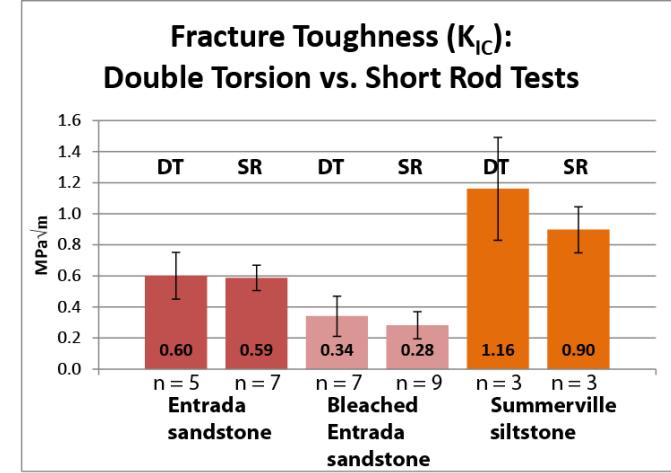


Figure 2. Comparison of fracture toughness test data obtained from samples collected at the Crystal Geyser field site using both the double torsion (DT) and short rod (SR) test method. Error bars are 1 $\sigma$  calculated from test data on multiple specimens taken from the same samples and n is the number of valid tests.

##### 3.2.2. Effect of CO<sub>2</sub>-related alteration on fracture toughness

As shown by the XRD data, CO<sub>2</sub>-related alteration has a significant effect on bulk mineralogy, and this may explain the difference in fracture toughness values measured on pairs of naturally altered and unaltered samples collected from the field site (Figure 3). CO<sub>2</sub>-related altered has lowered the fracture toughness of Entrada sandstone by nearly 50%. In contrast, the effect on siltstone is much less significant and within error of the test data collected.

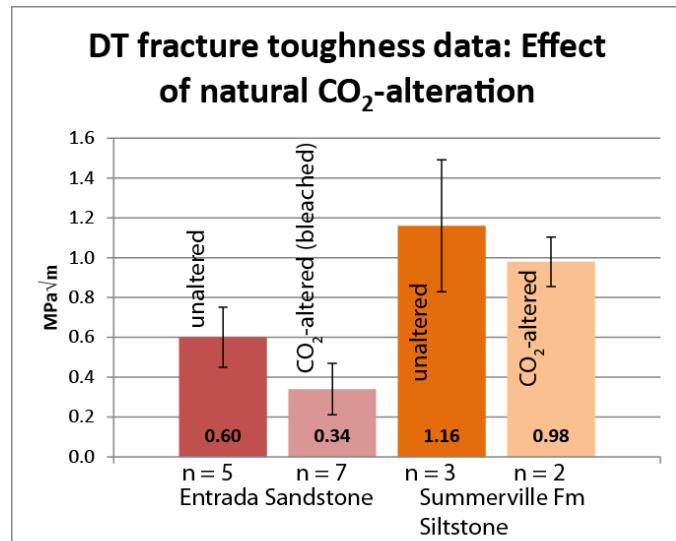


Figure 3. Fracture toughness test data obtained on unaltered and naturally CO<sub>2</sub>-altered pairs of sandstone and siltstone samples from the Crystal Geyser field site. Error bars are 1 $\sigma$  calculated from test data on multiple specimens taken from the same samples and n is the number of valid tests.

### 3.2.3. Effect of $\text{CO}_2$ -related alteration on subcritical index

These data show significantly different subcritical index values between 1 altered and 2 unaltered siltstone samples, but sandstones have comparable values (Figure 4). This indicates that siltstone  $\text{CO}_2$ -alteration has an effect on the subcritical index, whereas sandstone bleaching does not affect subcritical index. This is the opposite of what was observed in the fracture toughness values.

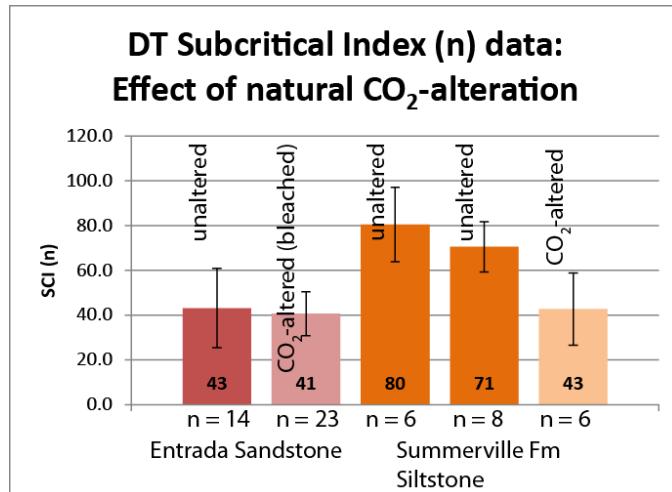


Figure 4. Subcritical index calculated using the double torsion method from pairs of unaltered and naturally  $\text{CO}_2$ -altered sandstone and siltstone from the Crystal Geyser field site. Multiple specimens from two different unaltered siltstone samples were tested due to a lack of enough sample material. Error bars are  $1\sigma$  calculated from test data on multiple runs from multiple specimens taken from the same samples and  $n$  is the number of valid tests.

### 3.2.4. Effect of $\text{CO}_2$ -rich environment on fracture toughness

The short immersion time is more akin to a hydraulic fracturing scenario, rather than a long-term injection and was designed primarily as a test of the experimental setup. The short immersion time was designed to isolate the effect of the immediate fluid environment at the fracture tip (i.e. at the top of the chevron notch) and to minimize the weakening of the entire sample by saturation of fluids and the bond with the epoxy.

Using this test protocol, fracture toughness values from bleached sandstone specimens tested under different controlled environments (ambient, brine, and  $\text{CO}_2$ +brine) show no significant differences (Figure 5), and additional tests on unbleached sandstone yielded the same result, based on a limited number of tests. This result was not completely unexpected and could indicate that the quartz-rich sandstone sample used is relatively insensitive to fluid chemistry on short time scales. A chemically reactive rock, such as limestone, shale, or calcite-cemented sandstone would more likely show a

measurable effect so this test on relatively geochemically stable quartz-rich sandstone could represent an opposite extreme chemical sensitivity.

### Effect of chemical environment on fracture toughness of bleached sandstone

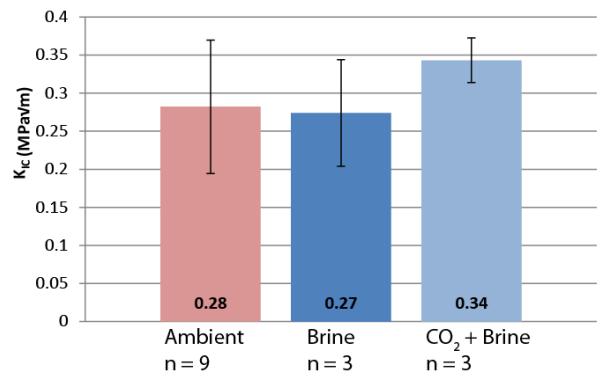


Figure 5. Short rod fracture toughness data obtained on multiple specimens of the same naturally altered sandstone but varying environmental conditions and using at least 3 specimens each.  $1\sigma$  error bars calculated from test data on multiple specimens taken from the same sample and  $n$  is the number of valid tests.

## 4. DISCUSSION

$\text{CO}_2$ -related alteration has significantly altered the bulk mineralogy of rocks at the Crystal Geyser field site which plays a role in their geomechanical properties. Degassing of  $\text{CO}_2$  is the primary driver of carbonate precipitation at Crystal Geyser [14] and has affected rocks up to tens of meters away from fossil springs along the Little Grand Wash Fault. Petrographic observations of the bleached, naturally  $\text{CO}_2$ -altered sandstones indicate dissolution of Fe-cements, along with the remobilization and precipitation of secondary carbonates and clays [15]. Similar alteration is likely affecting the siltstones sampled. Short rod and double torsion fracture testing results show that geomechanical failure parameters, such as fracture toughness and subcritical index of reservoir and seal lithologies, are significantly affected by this alteration. These fracture parameters play a role in fracture spacing, length, aperture, and clustering [16] and may be significant for the development of new fractures during and after  $\text{CO}_2$  injection. These results are also an example of time-dependent cracking described in the literature [2].

Subcritical crack growth is a product of stress corrosion, or the chemical processes occurring at the fracture tip, so it was somewhat unanticipated that immersion in  $\text{CO}_2$ -rich fluids had no significant effect on the fracture toughness. Most experimental data in the literature shows that water typically has a weakening effect leading to fracturing [2]. However, there are many complexities involved in fluid-rock interaction such as

the abundance and chemistry of dissolved ions and the mineralogy of the rock itself. We hypothesize that CO<sub>2</sub>-saturated, relatively low pH fluid may have a greater effect on calcite-cemented or calcite-rich rocks such as limestone and plan to evaluate this in future experiments.

The differences in measured values between naturally altered and unaltered samples are greater than the effect of CO<sub>2</sub>-rich environments, meaning that the end-product of CO<sub>2</sub>-alteration is more significant on fracture parameters than the present chemical environment, at least for the sandstones and siltstones found at Crystal Geyser. In CO<sub>2</sub> sequestration sites, however, CO<sub>2</sub> will be in a supercritical state, so room-temperature, CO<sub>2</sub>-in brine conditions used in this study may not be relevant. It likely possible that similar testing on shales or evaporite seal rocks may show a more significant effect.

## 5. CONCLUSIONS

This study of samples taken from a natural CO<sub>2</sub> system in mixed siliciclastic rocks similar to those likely to be encountered in CO<sub>2</sub> sequestration projects shows that CO<sub>2</sub>-rich fluids can significantly alter the bulk mineralogy and geomechanical properties of reservoir and seal rocks. Interaction with CO<sub>2</sub>-rich fluids is characterized by increased abundance of calcite near the fault.

Test data show that both the short rod and double torsion test methods yield comparable and complementary data for the same samples. Test data also show that CO<sub>2</sub>-related alteration can affect the geomechanical properties, such as fracture toughness and subcritical index of sandstones and siltstones. However, the presence of CO<sub>2</sub> does not appear to have a significant effect on fracturing on short time scales for a relatively geochemically “inert” rock such as quartz-rich sandstone.

Fracturing is the most likely mechanism for leakage of stored CO<sub>2</sub>, so the effect of CO<sub>2</sub> on reservoir and seal rocks should be included in time-dependent, coupled chemical-mechanical models.

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## REFERENCES

- [1] Kharaka YK, Thordesen JJ, Hovorka SD, Seay Nance H, Cole DR, Phelps TJ, et al. Potential environmental issues of CO<sub>2</sub> storage in deep saline aquifers: Geochemical results from the Frio-I Brine Pilot test, Texas, USA. *Applied Geochemistry*. 2009;24:1106-12.
- [2] Brantut N, Heap MJ, Meredith PG, Baud P. Time-dependent cracking and brittle creep in crustal rocks: A review. *Journal of Structural Geology*. 2013;52:17-43.
- [3] Atkinson BK. Subcritical crack propagation in rocks: theory, experimental results and applications. *Journal of Structural Geology*. 1982;4:41-56.
- [4] Atkinson BK. Subcritical crack growth in geological materials. *Journal of Geophysical Research*. 1984;89:4077-114.
- [5] Swanson PL. Subcritical crack growth and other time- and environment-dependent behavior in crustal rocks. *Journal of Geophysical Research*. 1984;89:4137-52.
- [6] Olson JE. Joint Pattern Development: Effects of Subcritical Crack Growth and Mechanical Crack Interaction. *J Geophys Res*. 1993;98:12251-65.
- [7] Olson JE. Predicting fracture swarms — the influence of subcritical crack growth and the crack-tip process zone on joint spacing in rock. *Geological Society, London, Special Publications*. 2004;231:73-88.
- [8] Hillier S. Use of an air brush to spray dry samples for X-ray powder diffraction. *Clay Minerals*. 1999;34:127-.
- [9] Evans AG. A method for evaluating the time-dependent failure characteristics of brittle materials — and its application to polycrystalline alumina. *J Mater Sci*. 1972;7:1137-46.
- [10] Williams DP. A simple method for studying slow crack growth. *J Test Eval*. 1973;1:264-70.
- [11] Holder J, Olson JE, Philip Z. Experimental determination of subcritical crack growth parameters in sedimentary rock. *Geophysical Research Letters*. 2001;28:599-602.
- [12] Suggested methods for determining the fracture-toughness of rock. *International Journal of Rock Mechanics and Mining Sciences & Geomechanics Abstracts*. 1988;25:71-96.

- [13] Doelling HH. Interim Geologic Map of the San Rafael Desert 30' x 60 ' Quadrangle, Emery and Grand Counties, Utah. Open-file Report 404. Salt Lake City: Utah Geological Survey; 2002.
- [14] Baer J, Rigby J. Geology of the Crystal Geyser and the environmental implications of its effluent, Grand County, Utah. Utah Geology. 1978;5.
- [15] Wigley M, Kampman N, Dubacq B, Bickle M. Fluid-mineral reactions and trace metal mobilization in an exhumed natural CO<sub>2</sub> reservoir, Green River, Utah. Geology. 2012.
- [16] Olson JE, Laubach SE, Lander RH. Combining diagenesis and mechanics to quantify fracture aperture distributions and fracture pattern permeability. Geol Soc Spec Publ. 2007;270:101-16.