Assessment of mechanical rock alteration caused by CO₂–water mixtures using indentation and scratch experiments

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Abstract

CO₂ injection into geological formations disturbs the geochemical equilibrium between water and minerals. Thus, some mineral phases are prone to dissolution and precipitation with ensuing changes of petrophysical and geomechanical properties of the host formations. Chemically-assisted degradation of mechanical properties can endanger the structural integrity of the storage formation and must be carefully studied and considered to guarantee safe long-term trapping. Few experimental data sets involving CO₂ alteration and mechanical testing of rock samples are available as these experiments are lengthy, expensive, and require specialized equipment and personnel. Autoclave experiments are easier to perform and control but result in a limited “skin depth” of chemically-altered zone near the surface of the sample. This article presents the validation of micro-indentation and micro-scratch tests as efficient tools to assess the alteration of mechanical properties of rocks geochemically altered by CO₂–water mixtures. Results from tests on sandstone and siltstone from Crystal Geyser, Utah naturally altered by CO₂-acidified water show that mechanical parameters measured with indentation (indentation hardness, Young’s modulus and contact creep compliance rate) and scratching (scratch hardness and fracture toughness) consistently indicated weakening of the rock after CO₂-induced alteration. Decreases of measured parameters vary from 14% to 87%. Experimental results and analyses show that micromechanical tests are potentially quick and reliable tools to determine the change of mechanical properties of rocks subject to exposure to CO₂-acidified water, particularly in well-controlled autoclave experiments. Measured parameters are not intended to provide inputs for coupled reservoir simulation with geomechanics but rather to inform the execution of larger scale tests investigating the susceptibility of rock facies to chemical alteration by CO₂-water mixtures. Recognizing this susceptibility of rock facies of CO₂ geological storage target formations is critical to controlling undesired emergent behavior associated with CO₂ sequestration.

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1. Introduction

The injection of carbon dioxide underground for geological CO₂ storage projects exposes host formations to reactive CO₂-water mixtures. Both field and laboratory experiments have shown that the injected CO₂ dissolves into brine, lowers the pH of the formation water and changes the species composition [Hovorka et al., 2006, 2013; Carroll et al., 2011; Lu et al., 2012]. Mineral-CO₂-water interaction varies according to the mineralogy of the host reservoir [Kaszuba et al., 2004; Assayag et al., 2009]. The reaction rate between rock and CO₂-rich brine depends on the mineralogy of the rock. Both siliceous and carbonate minerals are susceptible to mineral dissolution although carbonates tend to react faster than quartz and clays [Stumm & Morgan, 1996; Gunter et al., 2000; Pokrovsky et al., 2005; Le Guen et al., 2007, Bachu et al., 2007]. Long-term exposure may significantly alter the transport and mechanical properties of reservoir and caprock formations at CO₂ storage sites [Rutqvist & Tsang, 2002; Min et al., 2009; Espinoza et al., 2011; Rutqvist, 2012; Wojtacki et al., 2015].

In reference to mechanical properties, laboratory experiments show that CO₂-brine mixtures can dissolve carbonate minerals in carbonate and clastic rocks, change porosity [Yasuhara et al., 2006; Canal et al., 2013; Carroll et al., 2013], increase rock compaction [Le Guen et al., 2007; Liteanu et al., 2012], lower fracture toughness [Major et al., 2015], and modify yield stress locus [Rinehart, 2015]. The dissolution of rock minerals by CO₂-rich brine can change the matrix properties of the rock with a detriment to structural integrity of the formation [Schneider et al., 1996; Liu et al., 2006; Taron et al., 2010]. The degradation of mechanical properties by mineral dissolution can facilitate caprock straining, fracture propagation, and compaction-driven shear failure of the reservoir which may negatively affect storage effectiveness [Kim & Santamarina, 2014].

Core-flooding experiments documenting chemo-mechanical coupling with CO₂-water mixtures are limited because of the complexity of putting together such a system, instrumentation and personnel availability, and extended experiment durations [Carroll et al., 2011, 2013; Mason et al., 2013; Kutchko et al., 2008; Bachu et al., 2009]. Autoclave experiments on the other hand are easy to perform but result in a limited skin depth of chemical alteration in samples [Rimmele et al., 2010]. The impact of a reacted skin on the scale of one millimeter is negligible in mechanical testing of cores sized in several centimeters. Thus, a micromechanical test that can probe the reacted skin rather than the entire core seems a

reasonable diagnostic tool for rocks reacted in autoclave experiments. Indentation and scratching tests have been widely used in the industry to test various mechanical properties of different materials, such as ceramics, metals and polymers [Oliver & Pharr, 2004; Atkins, 2004; Kaupp, 2006; Bellemare et al., 2007; Wredenberg & Larsson, 2009; Akono et al., 2011; Pintaude, 2013]. In the oil and gas industry, the scratch test has been used to measure the strength, hardness and fracture toughness of sedimentary rocks [Adachi et al., 1996; Richard et al., 1998, 2012; Schei et al., 2000; Suarez-Rivera et al., 2002; Akono & Ulm, 2011, Krakowiak et al., 2011].

This article presents a quick and simple screening tool to assess the alteration of mechanical properties of rocks subjected to CO₂–water mixtures. Experiments are conducted on sandstone and siltstone naturally altered by a CO₂ seepage at Crystal Geyser, Utah. We use micro-scratching and indentation to reduce the size of the probed volume and expedite the duration of testing. These relatively simple techniques provide indices of elastic and failure parameters that can inform about the chemo-mechanical susceptibility of the rock facies to CO₂-water exposure.

2. Experimental methodology and data analysis

Micro-indentation and scratching tests target a probed volume smaller than ~1 mm³. Indentation tests involve applying a normal load on a flat surface with an indenter tip of known geometry. The penetration of the indenter tip causes both elastic and plastic deformation of the sample. When reaching a preset maximum load value, the normal load is reduced until partial or complete relaxation occurs. The resulting load/displacement curve provides data related to the mechanical nature of the material [Cripps, 2011].

The scratch test measures the resistance of a rock to surface damage induced from a scratch imposed with a stylus of known geometry moved at constant speed and normal force [ASTM G171]. A measure of the scratch hardness and fracture toughness can be calculated from the normal load, tangential load, and residual shape of the scratch [ASTM G171, Akono & Ulm, 2011]. Experiments targeted a contact area several times larger than the grain size such that the measured mechanical properties reflect the bulk properties of the cemented rock matrix rather than the properties of a single grain.

2.1. Specimen and sample preparation

We tested Entrada sandstone and Summerville siltstone from Crystal Geyser, Utah. The grain sizes vary from 50-100 μm for Entrada sandstone and 5-20 μm for Summerville siltstone. The outcrops in some parts have been altered (bleached) by natural leakage of CO_2 -charged brine. The alteration was characterized by dissolution and reprecipitation of carbonates, clays and iron cements (further details available in Wigley et al., 2012 and Major et al., 2015). We tested both CO_2 -altered and unaltered samples provided by J. Major and P. Eichhubl. Therefore, four samples are considered: unaltered Entrada sandstone, CO_2 -altered Entrada sandstone, unaltered Summerville siltstone and CO_2 -altered Summerville siltstone.

The samples were cut into 1 in cubes using a diamond saw for the indentation test. For the scratch test, the samples were drilled perpendicular to their bedding planes, producing cylindrical cores of 1 cm diameter. In both cases, one surface parallel to the bedding plane was polished with Allied Tech diamond lapping film on a lapping wheel to a desired surface roughness suitable to obtain reliable experimental results (the root-mean-squared roughness of the sample should be less than five times of the average penetration depth [Miller et al., 2008]).

2.2. Experimental Devices and Procedure

Indentation. A Nanovea PB1000 device was used to perform the indentation test. A standard Vicker's indenter was driven into the polished sample surface at a loading rate of 10 N/min. After the pre-set maximum load of 5 N was reached, the applied load was held and paused for 5 seconds. The non-contact optical sensor keeps recording any time-dependent creep response of the sample at this stage. After 5 seconds, the indenter tip was lifted with an unloading rate of 10 N/min. Indentation depth on the sample surface was recorded throughout the entire loading/unloading process. The recorded signal is plotted against the corresponding applied load for analysis.

Scratching. A Nanovea PB1000 device was also used to perform scratch test on the samples. Scratches were made on the rock samples with a sphero-conical stylus with radius of 200 μm applying a constant vertical load of 20 N drawn at a constant speed of 5 mm/min across the polished surfaces. A load cell records the lateral frictional resistance force on the stylus along the

scratch pathway. The width of the scratch was measured after testing separately with a Leica DFC420 C microscope and a Phillips/FEI XL30 Environmental SEM microscope.

2.3. Data Analysis

Indentation. The loading/unloading curves of the indentation test help assess three important material properties: indentation hardness H_I , Young's modulus E , and contact creep compliance rate $\dot{L}$ [ASTM E2546; Oliver & Pharr 2004; Cripps 2011; Vandamme et al., 2012]. A representative indentation loading curve is shown in Fig. 1.

Fig. 1. Representative loading and unloading curve for an indentation test on unaltered Entrada sandstone. Direct measurable outputs include maximum indentation depth h_{max} , unloading stiffness S and creep rate dh/dt at constant load.

The simplest direct output from the example curve in Fig. 1 is the maximum indentation depth h_{max} at the preset maximum applied load P_{max} (~5 N in our experiments). The stiffness S is calculated from a fit of the upper one third of the unloading curve. These three parameters enable an estimation of the contact depth h_c ,

$$h_c = h_{max} - \frac{3P_{max}}{4S} \quad (1).$$

For a perfect Vickers indenter, an estimate of the projected contact area A_c is provided by the indenter shape function,

$$A_c = 24.5h_c^2 \quad (2).$$

An experimentally determined estimation of the indenter shape function would be recommended to ensure the properties measured during the indentation test are indeed characteristic of the material. However, Eq. (2) provides a rough estimate for an ideal Vicker's indenter sufficient for comparison of properties measured with the same apparatus.

ASTM E2546 provides the equations for computing indentation hardness H_I [GPa], the reduced modulus E_r [GPa], and Young's modulus E [GPa],

$$H_I = \frac{P_{max}}{A_c} \quad (3),$$

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} \quad (4),$$

$$E = (1 - \nu^2) \left(\frac{1}{E_r} - \frac{1 - \nu_i^2}{E_i} \right)^{-1} \quad (5),$$

where E_i and ν_i are the Young's modulus and Poisson's ratio of the indenter tip (standard diamond $E_i = 1140$ GPa and $\nu_i = 0.07$ [NIST Ceramics]) and ν is the Poisson ratio of the tested sample. We assumed $\nu = 0.2$ for the tested clastic rock samples.

The contact creep compliance rate $\dot{L}$ [(GPa·s)⁻¹], a measure of the time-dependent response of the material to a step load, is given by

$$\dot{L} = \frac{2a_U \frac{dh}{dt}}{P_{max}} \quad (7),$$

where a_U is the contact radius at the beginning of the unloading phase and dh/dt is the change in indentation depth with respect to time. The derivation of this equation, detailed by Vandamme et al. (2012), takes into account plasticity under the indentation tip encountered during the loading phase. The measure is independent of loading profile and indenter geometry. Fig. 1 shows an inset with a zoom of the creep region from where dh/dt is obtained using a linear fit.

Scratching. The scratch test permits recovering the scratch hardness $H_{scratch}$ and fracture toughness K_c if conditions are appropriate. The result of the scratch test is a signal of lateral force F_T plotted as a function of lateral stylus displacement along the scratch path under a constant normal force F_v as shown in Fig. 2. The scratch width is measured independently. The scratch hardness $H_{scratch}$ [GPa] is calculated according to ASTM G171 as

$$H_{scratch} = \frac{kF_v}{w^2} \quad (7),$$

where k is a geometric constant (24.98 for the scratch tip used in this experiment), F_v is the applied load normal to the surface and w is the scratch width.

Fracture toughness K_c [MPa·m^{1/2}] is calculated as follows [Akono et al., 2011; Akono & Ulm 2012],

$$K_c = \frac{F_T}{\sqrt{2pA}} \quad (8),$$

where F_T is the horizontal force, p is the scratch perimeter and A is the horizontal projected load bearing contact area. Parameters p and A are a function of the scratch width and stylus shape (see detailed description in [Akono et al., 2011; Akono & Ulm 2012]). This formula is valid for depths of scratch sufficient for the $F_T/\sqrt{2pA}$ relationship to reach a horizontal asymptote, which occurs at a depth dependent on the material's internal length scale [Akono et al., 2012].

Fig. 2. Representative loading curve for a scratch test on unaltered Summerville siltstone. (a) Measured lateral and normal forces with constant vertical load scratching. (b) Light microscope image of respective scratch imprint, where the residual groove of the scratch appears as a lighter shade than the surrounding surface. White arrows indicate direction of travel.

3. Results

3.1. Indentation

Five indentation tests were performed on each of the four rock samples. Fig. 3 shows the results for the 20 indentation tests performed. The maximum indentation depth ranges from ~ 15 to $30 \mu\text{m}$ in Entrada Sandstone and from ~ 15 to $35 \mu\text{m}$ in Summerville siltstone. Hence, the contact area varies from about $5500 \mu\text{m}^2$ to $30,000 \mu\text{m}^2$, which in the case of Entrada sandstone is equivalent to approximately 1 to 10 times the cross sectional area of a sand grain. CO_2 -altered samples tend to show higher maximum indentation depths and less steep elastic rebound than the unaltered samples. Differences in hardness are discernible in Fig. 3 through corresponding differences in mean indentation depths between samples. Fig. 4 shows the average and 95% confidence intervals (as error bars) of indentation hardness, Young's Modulus, and creep compliance rate computed with Eqs. (3), (5), and (6).

Fig. 3. Indentation loading/unloading curves for (a) unaltered Summerville siltstone, (b) unaltered Entrada sandstone, (c) CO₂-altered Summerville siltstone, and (d) CO₂-altered Entrada sandstone.

Fig. 4. Indentation results for (a) indentation hardness, (b) Young's Modulus, and (c) contact creep compliance rate. Error bars represent 95% confidence intervals. Legend applies to all subfigures.

3.2. *Scratching*

Testing consisted of three scratches on each sample. Fig. 5 shows the results of the 12 scratch tests performed. The scratch width is measured from digital image analysis of microscope images using ImageJ. The averaged scratch width for unaltered sandstone is 384 μm , while the average scratch width for CO_2 -altered sandstone is 443 μm . The CO_2 -altered sandstone has potentially damaged areas along the scratches (indicated by red circles in Fig. 5c,d). Those areas are not included in calculating fracture toughness. The average scratch width for unaltered siltstone is 214 μm , while the average scratch width for CO_2 -altered siltstone is 610 μm . Scratches on the CO_2 -altered siltstone tended to carve in during scratching with a significant increment of scratch width towards the end (Fig. 5c). Scratches on the CO_2 -altered sandstone are generally wider than those on unaltered sandstone under constant normal force. Scratch hardness and fracture toughness were computed for five sections along the scratch path for each scratch with Eqs. (7) and (8). Fig. 6 shows the average and 95% confidence intervals (as error bars) of scratch hardness and fracture toughness.

Fig. 5. Lateral force F_T during scratching under 20 N constant vertical force for (a) unaltered Summerville siltstone, (b) unaltered Entrada sandstone, (c) CO₂-altered Summerville siltstone, and (d) CO₂-altered Entrada sandstone. Insets show the corresponding light microscopic images of scratched rock specimen. Colored numbers by plot lines indicate the scratches, identified in the insets, which data corresponds to. Direction of all scratches is from top to bottom. Red circles indicate potentially damaged areas, for which scratch damage will be higher if counted.

Fig. 6. Indentation results for (a) scratch hardness and (b) fracture toughness. Error bars represent 95% confidence intervals. Legend applies to all subfigures.

4. Discussion

4.1. Micromechanical tests and material internal length scales

Micromechanical experiments must consider the internal length scales of the tested geological samples. The objective is to test a representative volume that can inform about the effect of CO₂-acidified brine on the rock matrix including the impact of cementation at contacts if any. Let us use the ratio between the probed area A_c and the average cross sectional area of a grain $\sim(d_{50})^2$ as an indicator of relative length scales.

In indentation tests the ratio $A_c/(d_{50})^2$ varies from 1 to 10 in the tested sandstone and from 18 to 100 in siltstone. Hence, it is likely that the indentation test measurements may fluctuate due to grain scale heterogeneity. Indentation loading curves in sandstone (Fig. 3b,d) were more irregular than in siltstone. Higher preset load would have been desirable to test a larger volume in sandstone. Indentation in siltstone did not suffer from fluctuations due to lower internal length scales. However, it seems that two indentation tests, characterized by relatively large maximum indentation depths in Fig. 3c, landed on soft spots. It is uncertain whether these soft spots are the result of CO₂ alteration or not. The exclusion of these measurements reduces the change in contact creep compliance rate from unaltered to CO₂-altered Summerville siltstone from 160% to 70%. Table 1 summarizes the parameters measured from the two types of tests including mean values, 95% confidence intervals, and percentage changes after alteration.

Table 1.

Summary of average results and 95% confidence intervals from micromechanical tests and relative change of mechanical properties after being altered by CO₂-acidified water.

Material		Indentation Hardness (GPa)	Young's Modulus (GPa)	Contact Creep Compliance Rate (1/(GPa·s))	Scratch Hardness (GPa)	Fracture Toughness (MPa·m ^{1/2})
Entrada Sandstone	Unaltered	0.53 ± 0.23	16.25 ± 3.07	0.0013 ± 0.0005	0.35 ± 0.04	2.60 ± 0.07
	CO ₂ -Altered	0.33 ± 0.09	12.56 ± 1.71	0.0023 ± 0.0008	0.26 ± 0.05	2.24 ± 0.39
	Change	-38%	-23%	72%	-24%	-14%
Summerville Siltstone	Unaltered	0.63 ± 0.15	18.28 ± 2.37	0.0013 ± 0.0003	1.11 ± 0.05	7.82 ± 2.89
	CO ₂ -Altered	0.47 ± 0.21	8.90 ± 4.91	0.0035 ± 0.0022	0.14 ± 0.04	1.47 ± 0.52
	Change	-25%	-51%	160%	-87%	-81%

In the scratch tests the ratio between scratched area and average cross sectional area of a grain $A_s/(d_{50})^2$ varies from 175 to 300 in the tested sandstone and from 3000 to 5000 in siltstone. Significant variability did arise in acquired data of lateral force F_T as a function of length of the scratch path. The variability could be explained by a combination of heterogeneity of rock cementation, fracture propagation, grain size, and stick-slip behavior. Insets in Fig. 5 show evidence of chipping and fracturing behavior (demarcated by red circles) far away from the path of the stylus. Scratches are seen to intercept cemented grains. Backscattered electron images in Fig. 7 show a magnified view of this region.

Fig. 7. (a) Representative frontal view schematic of wide damage zone induced by stylus translation. Damaged areas are indicated in black. Stylus travels in z-direction (out of plane). Backscattered electron images taken with a Phillips/FEI XL30 Environmental SEM microscope without interpretations (a) and with interpretations (b). Red arrows over residual damage zones indicate direction of interpreted brittle fracturing processes.

Parameters from indentation and scratching experiments are not representative of core-scale measurements and much less of reservoir scale parameters. However, micromechanical experiments inform about mechanical properties of the rock matrix that may eventually upscale to characterize reservoir-scale phenomena.

4.2. Effect of CO_2 alteration on mechanical parameters

Indentation tests show that average indentation hardness of Entrada sandstone decreased by 38% while that of Summerville siltstone decreased by 25% after CO_2 alteration as shown in Table 1. Young's modulus of the Entrada sandstone decreased by 23% while that of siltstone decreased by 51% by CO_2 alteration. Both sandstone and siltstone became more susceptible to time-dependent deformation under influence of stress, as the contact creep compliance rate increased by 72% for sandstone and 160% for siltstone. A larger measurement volume is needed to reduce variance in the measurement of the contact creep compliance rate for CO_2 -altered Summerville siltstone and to better determine where the measured parameter is indicative of

CO₂-related weakening and/or a time-dependent response to stress. In general, the indentations on altered siltstone all have less steep unloading curves than the unaltered siltstone, indicating a less stiff material response.

Average scratch hardness of Summerville siltstone decreased by 87% after CO₂ alteration over geological time, while average scratch hardness of Entrada sandstone decreased by 24% as summarized in Table 1. Scratch hardness measurements are not intended to provide direct quantitative insight into the strengths of the materials, as values would be expected to vary across different loading conditions and indenter geometries. Their use is restricted to comparative purposes. Scratch tests show that averaged fracture toughness of Summerville siltstone decreased by 81% after CO₂ alteration over geological time, while averaged fracture toughness of Entrada sandstone decreased by 14%.

Major et al. (2014) used double torsion and short rod tests to report that average fracture toughness for Entrada sandstone decreased from 0.60 to 0.34 MPa·m^{1/2} and that for Summerville siltstone decreased from 1.16 to 0.98 MPa·m^{1/2} after CO₂ alteration. The values of fracture toughness obtained from scratch tests are systematically higher than these values. Differences in reported values between these two studies may be partially accounted for by the difficulty of comparing testing methods that induce varying degrees of in-plane shear (Mode II) and opening (Mode I) fracture. Fractures may propagate with less resistance in Mode I or Mode II regimes depending on the material [Liu et al., 2005; Xia & Hutchinson, 1994]. Characterization of pure fracture toughness values for separate modes is very difficult as a degree of Mode I behavior will always exist at the front of the fracture tip [Liu et al., 2005]. Differences in reported values may also be partially accounted for by an effect of ductility on scratch measurements made at a depth low with respect to internal material length scales and indenter tip size. Akono et al. (2012) indicates that $F_t/\sqrt{2pA}$, which is equivalent to apparent fracture toughness, approaches a horizontal asymptote representing the fracture toughness after reaching a ratio of $d/R > 0.3$, where d is scratch depth and R is the indenter tip radius. This criterion was reported as valid for samples including ceramics, soft and hard metals over a penetration depths ranging from 8 to 500 μm. An extension of these criteria to sedimentary rocks as a qualitative indication of brittle fracture may be plausible and will be validated in future work. In the case of the scratches conducted on unaltered and altered Entrada sandstone and altered Summerville siltstone, the $d/R > 0.3$ ratio is exceeded by a large margin, though we suspect that fracture toughness values

still may have been measured in a transition zone between ductile and brittle behavior. In the case of unaltered Summerville siltstone, the depth ratio only reached 0.3, raising concerns about the interpretation of this value as a measure of fracture toughness. Qualitatively, the presence of small fractures indicative of brittle behavior is noted in a representative topographical backscattered electron image of altered Summerville siltstone in Fig. 7.

The general trends from both testing methods agree well, indicating significant mechanical weakening of rock samples by CO₂-alteration. Petrographical analysis shows that the CO₂ altered rocks at Crystal Geyser underwent changes in carbonate content, illite formation, and iron cement dissolution [Wigley et al., 2012; Major et al., 2014] and may explain the apparent weakening indicated by scratch and indentation test results.

4.3. Application to autoclave laboratory experiments

Rocks reacted in an autoclave in the laboratory generally affect a limited outer skin in contact with the reactive fluid. The indentation and scratch techniques introduced here can take advantage of this limitation and probe the reacted rock skin. Autoclave experiments are relatively easy to perform. Thus, micromechanical tests could be performed on samples altered with CO₂-water mixtures over various time scales to give insight into the kinetics of chemical reactions through their effect on mechanical testing.

The rock samples tested in this article come from a site naturally altered by a CO₂ seepage. Alteration is visible over tens of meters of length and occurred over a geological period of time. Thus, tested rock samples were either entirely altered or unaltered. Ongoing work involves the testing of several rock facies exposed to CO₂-water mixtures in autoclave experiments in the laboratory. The objective is to efficiently test the susceptibility of rocks to chemo-mechanical alteration by CO₂-water mixtures and investigate associated time scales of alteration.

5. Conclusions

CO₂ injection into geological formations disturbs the geochemical equilibrium such that some mineral phases may dissolve with an ensuing change of geomechanical properties of the host formations.

We tested the suitability of micromechanical tests to give insight into changes of mechanical properties with and without CO₂-induced geochemical alteration. These kinds of tests are cost-efficient, repeatable, and are well-suited to testing rocks reacted in autoclave experiments due to the limited depth of CO₂-water mixture alteration.

Micromechanical tests ought to account for the internal length scales of heterogeneous geological materials. Conducted tests targeted a tested volume that was large enough to capture the response of the cemented rock matrix rather than the individual properties of mineral grains. Thus, the volume affected by the indentation and scratch tests was several times larger than the average grain volume.

Results from indentation and scratch tests show that long-term exposure of Crystal Geyser rocks to reactive CO₂-water mixtures resulted in measurable mechanical degradation. All micromechanical measurements showed consistent change, indicating that the CO₂-altered rocks from Crystal Geyser demonstrated significant weakening in comparison to the unaltered samples. Comparing the CO₂-altered samples with respect to the unaltered samples, indentation hardness, Young's modulus, scratch hardness, and fracture toughness decreased in a range from 12% to 87%. The interpretation of fracture toughness values from scratch tests is not straightforward because of the effect of a materials ductile response on the fracture toughness at sufficiently low scratching depths. Future work will investigate, with respect to sedimentary rocks, the variation of apparent fracture toughness with scratch depth as well as the linearity of the relationship between vertical force and indenter shape function.

Experimental results and analyses show that the combination of micromechanical indentation and scratch tests are potentially efficient and useful tools in determining the change of mechanical properties of rock samples subject to exposure to CO₂-acidified water. Parameters measured from micromechanical tests are not intended to provide direct inputs for coupled reservoir simulation with geomechanics. However, they may inform about the susceptibility of certain rock facies to CO₂-related degradation to perform more carefully conceived tests at a larger scale on pre-selected rock facies.

Acknowledgements

This work was supported as part of the Center for Frontier of Subsurface Energy Security II, an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Basic Energy Sciences under Award DE-SC0001114. The research was conducted at Center of Petroleum and Geosystems Engineering (www.cpge.utexas.edu) at The University of Texas at Austin (UT). We would like to thank Jon Major and Peter Eichhubl from the Bureau of Economic Geology UT for providing the rock samples for testing, Karalee Jarvis from Texas Material Institute UT for assistance in preparing polished samples suitable for indentation and scratch tests, Shaina Kelly from UT for assistance in rock sample imaging, and Brent Jackson from the Jackson School of Geosciences for assistance with acquisition of backscattered electron images. We are also thankful to anonymous reviewers for suggestions that helped significantly improve this manuscript.

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