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Shale creep as leakage healing mechanism in CO_2 sequestration

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Abstract

Experiments on shale specimens targeted creep deformation in order to investigate whether this mechanism could be relied upon to close fractures and stop leakage near injection wells. Uniaxial compression tests were conducted with stress ramp up paused near expected failure stress of the tested shale and additional deformation, interpreted as initial creep deformation, was logged. The tests were repeated on shale samples exposed to HCl acid solutions, simulating expected pH conditions near the well upon exposure to injected CO_2. The purpose of the tests was to compare the creep propensity of the material after possible dissolution of load-bearing minerals, to the virgin case. The results show slight enhancement of creep deformation. Exposure to super-critical CO_2 on the same shale was carried out on hollow cylinder plugs, with radial closure of the borehole measured under radial stress conditions. Results show a doubling in radial deformation when compared to control tests with brine exposure. Finally, analyses of synchrotron beam tomography images were performed to quantify volume change between shale specimens preserved in oil and specimens exposed to HCl, showing that total volume increased by 9 %.

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Keywords: Creep; super-critical CO_2; shale; fracture healing.

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1. Introduction

CO_2 storage reservoirs will be accessed by purpose-built injection and production wells, in the case of saline aquifer storage, or older wells retrofitted for the purpose, in the case of depleted oil and gas fields. This entails strict requirements to well cementing to the surrounding rock formations, so as to eliminate or at least considerably lower probability for leakage in the near-well area. Of special importance is the state of the near-well area in shale formations, as these are, most of the time, the original sealing barriers through geological times; a leakage path through one of these barriers must imperatively be stopped to avoid losing large plume volumes in the next high permeability block [1]. One mechanism by which such a leakage path might cease to be a threat to storage containment is creep deformation of the shale itself, thus closing in on the well cement and healing the fracture.

Shale formations are known, depending on lithology and in-situ stress conditions, to be capable of creep (deform continuously as a function of time, for constant applied stress) so as to tightly press on a well, to the extent of eliminating previously recorded hydraulic conductivity between the well and the surrounding formation [2]. Such a mechanism would certainly be most welcome in the case of a leakage path around a well penetrating a CO_2 storage site (so-called micro-annulus). One could envisage that healing of micro-cracks would be taken care of naturally, by long-lasting creep deformation, requiring no active stress change, as long as prevailing compressive stress conditions remain valid over time.

One of the effects of CO_2 exposure is pH reduction of the fluid diffusing into the shale pores. A relevant study related to this subject was published by Hangx et al. [3], where uniaxial compaction creep experiments on granular aggregates of quartz and Ca-bearing feldspar were presented. The objective of the tests was to examine the effects on creep due to CO_2 injection. Creep effects generally depend on several factors, and the effect of pore fluid pH was only one element studied in detail in this paper. The tests were run under both wet and dry conditions, and the pore fluid pH was altered by addition of acidic and alkaline additives and by CO_2 injection. For the quartz experiments, the pH-value varied from 2.8-12.4. The results showed that the pH of the pore fluid had a significant impact on the amount of creep in the samples, as well as on creep rate. Reduced pH showed less creep and lower creep rates compared to near-neutral pH environments. In contrast, alkaline pore fluids ($pH > 7$) enhanced creep and creep rates compared to near-neutral fluids. Also, increased creep was seen for wet conditions, as compared to laboratory-dried samples. For the feldspar experiments, at room temperature, creep strain decreased with increasing pH in the pH range of 2.8-5.5, and remained more or less constant between pH 5.5 and 11. At reservoir conditions (80 °C, 36 MPa effective stress) creep strain and creep rates generally increased with greater solution pH and with enhanced salinity. The acceleration of creep with increasing pH and salinity might be connected to enhanced stress corrosion cracking. In comparison to near-neutral solutions, addition of CO_2 inhibited creep or decelerated it strongly, both at room temperature and reservoir conditions. Also for the feldspar samples, creep was enhanced by adding aqueous pore fluid compared to dry conditions [3].

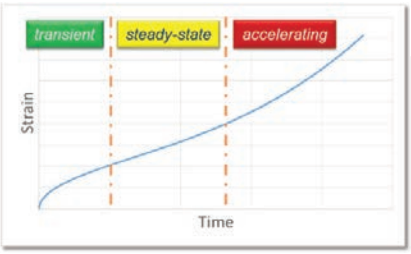
Olabode and Radonjic [4] investigated geochemical interaction of shale cap rock with aqueous CO_2 flooding, with focus on risk to cap rock integrity. Crushed shale samples of known mineralogy from the Pottsville Formation in Alabama (USA) were used in a flooding experiment of approximately 3-month duration. The mineralogical composition of shale cap rock can impact its ability to seal effectively, and the samples used in these tests consisted of mainly quartz, feldspar and (bulk) clay. The results from these experiments highlighted that petrophysical properties can be affected by shale/CO_2-brine interaction under dynamic conditions. It was shown that rock properties of shale can be significantly altered on a nano-scale. For instance, the naturally low permeability in shale may be altered, thus integrity with respect to permeability can be impaired [4]. Similarly, in 2015, Balashov et al. [5] carried out a comprehensive study to understand how shale interacts with CO_2 under conditions relevant for sequestration sites. The mineralogy of the tested shale resembled black shale from the Marcellus formation mainly consisting of quartz, illite and chlorite. A reactive diffusion model was used to simulate the transport of CO_2 in a subsurface sandstone reservoir capped by the shale. From the results, the authors concluded that shale with higher content of Mg, Ca and Fe ions (e.g. more chlorite or smectite) will more likely self-seal after injection of CO_2 than other shale formations.

Shale swelling and shrinkage have been studied at length, in connection with well drilling operations [6]; swelling may cause stuck pipe incidents and shrinkage can lead to formation fracturing and lost mud circulation. These borehole stability risks are mitigated with appropriate loading of drilling mud with salts at optimal concentration [7], influencing chemical activity, osmosis and ion exchange mechanisms. Since in sequestration operations, super-critical CO_2 will probably interact with shale resident pore brine, it is of interest to determine whether CO_2-brine mixtures affect differently the shale, as compared to pure brine with a given salt concentration. Thus, experiments described in this paper attempted to quantify creep deformation of outcrop shale samples, comparing between brine-saturated specimens, CO_2 in brine exposed specimens and specimens exposed to HCl acid solution in brine. Both uniaxial compression tests and hollow cylinder isotropic stress tests were conducted, uniaxial testing at SINTEF in Norway and isotropic testing at GEUS in Denmark.

2. Creep vs. consolidation

Creep is defined as a time-dependent deformation occurring in minerals under stress. Due to viscoelastic effects in the solid framework creep can occur in both dry and saturated rocks [8]. Viscoelastic behavior can be seen as a superposition of elastic, reversible deformation and strain flow proportional to applied stress. Contrary to brittle material failure, materials exhibiting creep deformation fail in a ductile manner, as strain accumulates with time once a threshold stress level is passed. Deformation rate depends in such materials on applied stress, temperature and elapsed time [9]. Three stages of creep are identified: initial transient stage, with deformation rate declining with time; steady-state creep, linear with time; and accelerating creep where deformation increases with time (Fig. 1a). If the applied stress is removed while still in the transient stage, the deformation will be reversed and the material will return to its original state. Reducing the stress to zero after the rock has reached steady-state or the accelerating stage will result in a permanent deformation of the rock. All stages of creep will only fully develop when applying moderate stress levels to the material (Fig. 1b). Depending on the level of applied stress, the transition between the three phases of creep can occur more or less rapidly: for high enough stress, accelerating creep sets in early on and may lead to failure of the specimen. This is due to the weakening effect associated with accelerating deformation. At low stress levels, the material may on the contrary stabilize after a period of transient creep [8].

a



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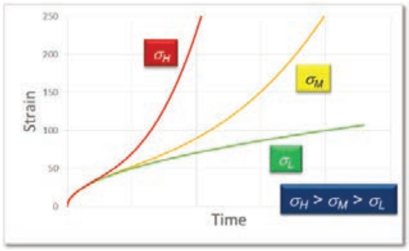

Fig. 1. (a) The three stages of creep; (b) influence of applied stress level on development of creep severity (after [8]).

While creep can occur in both dry and saturated rocks, consolidation is a time-dependent effect that can only appear for the latter condition. Consolidation develops when pore fluid pressure delays the deformation of the rock and is well known from soil mechanics where it is of common concern in the building branch, where settlement of structures and constructions must be taken into account [10]. When a stress increase is applied on a porous, deformable material such as soil or rock, if the pore fluid is much less compressible than the framework skeleton, time-dependent deformation occurs (applied stress being kept constant). This is due to the framework deforming instantaneously, while a low permeability will result in increase in pore fluid pressure until dissipation upon fluid flow away from the deformed region. Consolidation is accompanied by varying shear strength increase as the pore pressure evolves with time, hence the name [10,11].

Determining the consolidation time in the rock is of interest when trying to distinguish between creep and consolidation. An estimate of the time it takes before the pore pressure has reached equilibrium can be made from:

$$\tau_D = \frac{l_D{}^2}{C_D} \tag{1}$$

where τ_D is the consolidation time, l_D is the pore pressure diffusion length (for the shale experiments below, the radius of the plugs) and C_D is the pore pressure diffusion constant [8]. For competent rocks, C_D can be evaluated from:

$$C_D = \frac{k}{\eta_f}\left(K_{fr} + \frac{4}{3}G_{fr}\right) \tag{2}$$

where k is the intrinsic permeability of the rock, η_f is the dynamic viscosity of the fluid, K_{fr} is the bulk modulus of the rock frame and G_{fr} is the shear modulus of the rock frame [8].

3. Uniaxial creep experiments

Two series of uniaxial creep tests were carried out on cylindrical specimens of outcrop Pierre shale I (from a Colorado quarry). One series had the specimens saturated with 3.5 wt% NaCl brine, while the second series had the specimens saturated with a HCl acid solution in 3.5 wt% NaCl brine, yielding a pH value of approximately 3. All the core plugs were drilled with the core axis perpendicular to the bedding plane, with dimensions 25 mm (1 inch) in diameter and 52 mm (2 inch) in length. The mineralogical composition of the Pierre shale consists mainly of chlorite, illite, smectite and quartz. Details of the mineralogy are given in Table 1, with reactivity to HCl.

Table 1. Mineralogical composition of Pierre shale.

Mineral	%	Reactivity
Quartz	16.00	None
Plagioclase	8.55	Slight
Chlorite	29.50	Dissolves
Illite	19.90	May dissolve
Smectite	19.80	Swells in fresh water
Calcite	1.54	Dissolves
Siderite	1.44	Slight
Dolomite/Ankerite	2.63	Slight
Pyrite	0.64	Slight

An estimation of the consolidation time for the shale plugs gave 40 minutes; this, based on equations (1) and (2), with the characteristic diffusion length being given by the plug radius, a bulk modulus of 1 GPa, a Poisson's ratio of 0.36 and a permeability of 40 nD. The specimens were saturated in brine or acid solution by immersion in a container (see Fig. 2), assuming diffusion of ions was enough to penetrate the plug and saturate it. Since shales act as semi-permeable membranes, it is important to choose a brine that does not lead to swelling or shrinkage; therefore, a NaCl solution at 3.5 wt% was used. The acid solution was diluted so as not to alter significantly the chemical activity, as compared with only brine. This resulted in an acid solution with pH 3.14 (measured with an electronic pH-meter). Again, an estimation was made of the time needed for diffusion of ions over a distance corresponding to a plug's radius; this time, the diffusion coefficient was taken to be 100 times lower than the pressure diffusion coefficient used to calculate consolidation time, based on fluid exposure tests previously run at SINTEF [12]. The value found for the ion equilibration time was 2.8 days. On the other hand, experiments were performed whereby shale plug deformation was monitored upon immersion in brine. The time needed to reach stable deformation was interpreted as completion of ion diffusion into the plug; this time was 15 h. For the creep tests, a minimum period of 24 h exposure to brine or brine-acid mixture was chosen.

The creep tests were carried out in an MTS load frame, instrumented with deformation gauges and ultrasonic transducers (Fig. 2). An aluminium cylindrical container was used to expose the plugs to NaCl brine, while a copper one was used for the acid exposure (to avoid potential reaction with aluminium); fluid exposure was run at 0.5 MPa axial stress. Four thermocouples were inserted in the container to monitor temperature at different heights in the cell. Acoustic transducers were inserted in the axial pistons, transmitting pulses from the top to the bottom piston; the signals were collected with the Aptrans software and analysed with an in-house software.

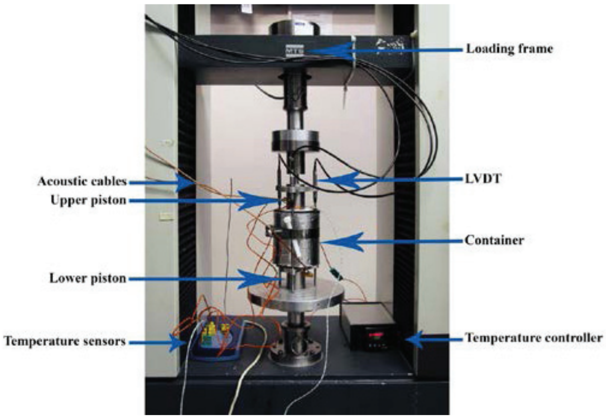

Fig. 2. Uniaxial 10 kN MTS electromechanical load frame. Axial strain is measured with 3 Linear Variable Differential Transducers (LVDT). The shale is exposed to brine or acid-brine in the container, then submitted to uniaxial stress.

A constant temperature of 30 °C was kept during the tests, with the aid of a heating element and insulation foil wound round the container.

Initial tests were run to failure so as to measure the unconfined compressive strength (UCS) of the samples exposed to the two different fluids; this was done to calibrate the stress steps in the creep tests in such a way to come close to failure in the final step, and hopefully transition rapidly to accelerating creep. UCS values found (Table 2) were 9.2 MPa when exposed to brine and 7.7 MPa when exposed to acid-brine, if one excludes the outlier at 3.6 MPa. This value is believed to be due to the fact that exposure to brine was not done under stress, thus encouraging swelling of the shale and reduction in its shear strength [13]. In addition, the plug is believed to have contained small fractures before testing, increasing dramatically exposure – see Fig. 3.

Table 2. UCS values for Pierre shale exposed with brine or acid-brine.

Test #	Exposure fluid	UCS [MPa]
1	NaCl brine	9.2
2	NaCl brine	9.2
3	HCl in NaCl brine	7.7
4	NaCl brine	3.6

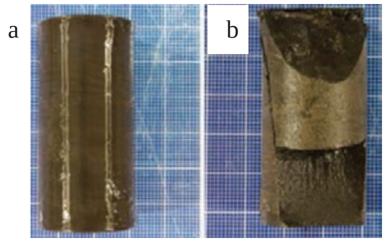

Fig. 3. (a) Pierre shale plug before UCS test (test #4 in Table 2); (b) same plug after failure at unexpected low value of 3.6 MPa axial stress, showing multiple fracture planes, suggesting pre-existing fractures.

The creep tests were run using the following procedure:

- Load up to 0.5 MPa and hold stress for 24 hours to complete ion diffusion
- Start creep test by loading up to 1.0 MPa over a time period of 5 minutes (0.1 MPa/min)
- Hold stress, let consolidate and creep for 4 hours
- Continue to increase load stepwise with 0.5 MPa (approximate running speed 0.1MPa/min) until failure.
- Hold stress, let consolidate and creep for 4 hours at each step

The transition from consolidation to creep was verified at each stress hold period by plotting strain as a function of the square root of time; departure from linearity indicates onset of creep deformation. In total, seven tests were run; however, 3 tests experienced early failure of the plug, which led to revision of the stress loading procedure, from one creep hold near failure to the one presented above. Table 3 sums up the uniaxial creep tests.

Table 3. Summary of uniaxial creep tests run at SINTEF.

Test #	Loading scheme	Fluid exposure	Comment
1	Direct to near-failure stress	Brine	Sample failed after 1.6 h at 7.8 MPa
2	Direct to near-failure stress	Brine	Sample failed on stress ramp at 6.3 MPa
3	Direct to near-failure stress	Brine	Sample failed after 1.9 h at 5.7 MPa
4	Stepwise stress increase	Brine	Successful test, failure on level 10 (5.5 MPa)
5	Stepwise stress increase	Brine and HCl Mixture	Successful test, failure on level 12 (6.5 MPa)
6	Stepwise stress increase	Brine and HCl Mixture	Successful test, failure on level 7 (4.0 MPa)
7	Stepwise stress increase	Brine	Successful test, failure on level 13 (7.0 MPa)

3.1. Creep tests in NaCl brine

In these uniaxial creep tests, the specimens were exposed to 3.5 wt% NaCl brine, with a pH of 7.3 and a chemical activity of 0.942. The first successful test, test #4 in Table 3, revealed that consolidation, as measured from adherence to a square root function of time, scales with absolute stress level (Fig. 4), exceeding at high stress the postulated 40 min (ranging from 1.5 h to 2.4 h). This is in agreement with previous work by Cogan [14].

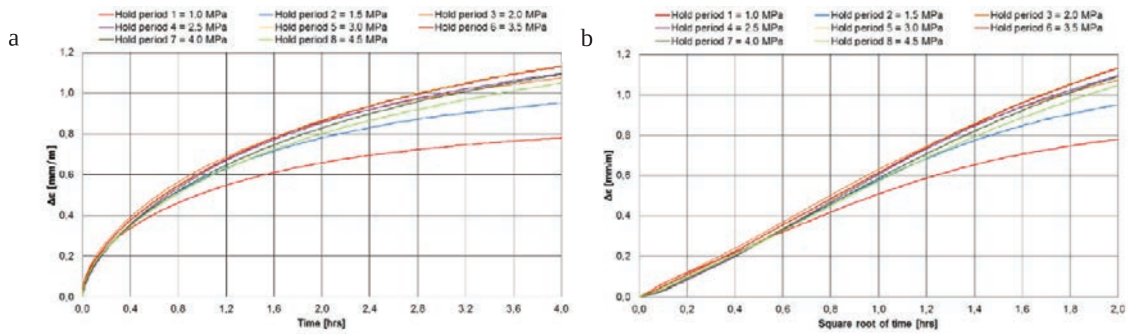

Fig. 4. (a) Axial strain timelines for test #4 in Table 3 for all stress hold periods; (b) Axial strain as a function of square root of time to determine extent of consolidation period.

The second successful test, test #7 in Table 3, also showed that consolidation, as measured from adherence to a square root function of time, scales with absolute stress level (Fig. 5), ranging from 1.5 h to 3.7 h. Both tests showed a slight increase in sound velocity V_p during the hold periods at constant stress, suggesting that continued straining affects closure of micro-cracks in a similar manner to stress increase (Fig. 6). Whether this small increase in velocity can be useful for CO_2-sequestration monitoring, when looking at micro-annulus healing, remains to be seen. Both tests also showed an increase in stiffness (Young's modulus E) with increasing stress, of 40 %. Fig. 7 shows the plugs after testing.

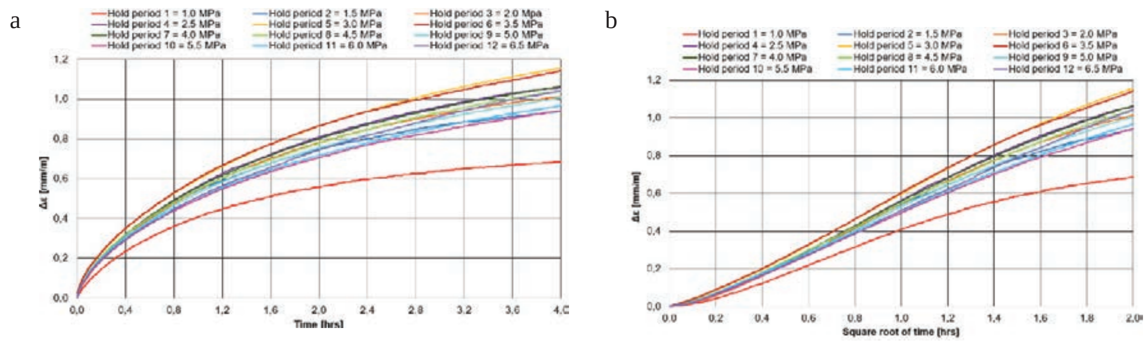

Fig. 5. (a) Axial strain timelines for test #7 in Table 3 for all stress hold periods; (b) Axial strain as a function of square root of time to determine extent of consolidation period.

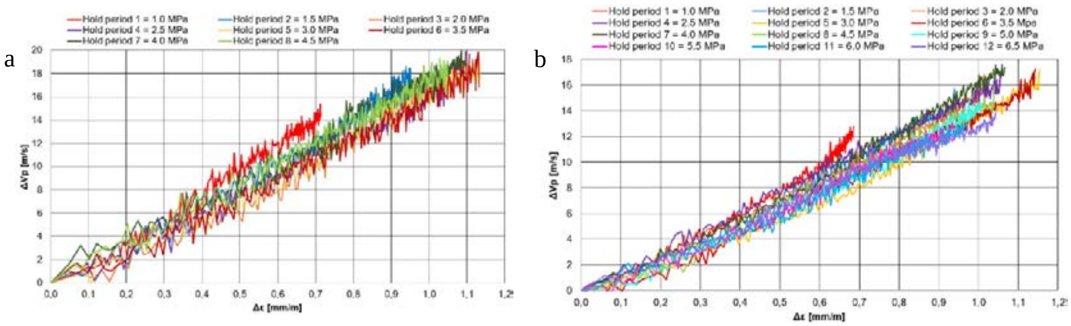

Fig. 6. (a) Increase in measured V_p for test #4 in Table 3 for all stress hold periods; (b) Increase in measured V_p for test #7 in Table 3 for all stress hold periods.

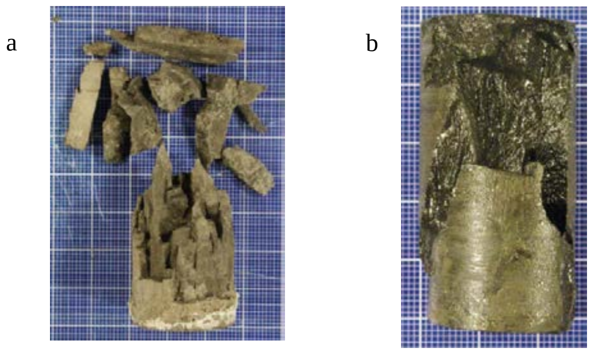

Fig. 7. (a) Pierre shale plug after creep test in brine (test #4 in Table 3); (b) Pierre shale plug after creep test in brine (test #7 in Table 3).

3.2. Creep tests in HCl acid brine

In these uniaxial creep tests simulating the pH effect of exposure to super critical CO_2, a mixture of 3.5 wt% NaCl and HCl was used as the fluid surrounding the core samples. The acid was added to the brine until the resulting fluid itself had a pH value of 3.14 and a chemical activity of 0.938. The obtained pH is in accordance with published values for super critical CO_2 [15,16]. The same type of deformation curves and consolidation content are obtained as before (Fig. 8). Consolidation time ranges from 1.2 h to 2.3 h. The second low pH test, test #6 in Table 3, had the shale fail at a lower stress of 4 MPa. This may be due to a discovered leakage of the immersion fluid, leaving the shale dry and thus possibly developing fractures from its surface. Consolidation time ranges from 0.8 h to 4 h (Fig. 9).

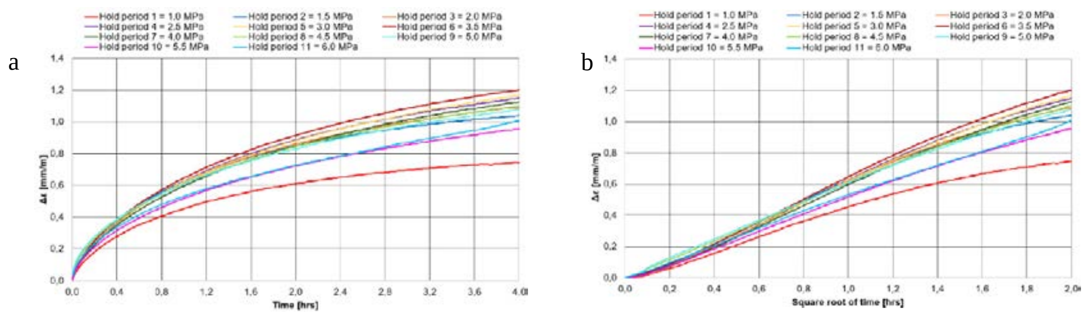

Fig. 8. (a) Axial strain timelines for test #5 in Table 3 for all stress hold periods; (b) Axial strain as a function of square root of time to determine extent of consolidation period.

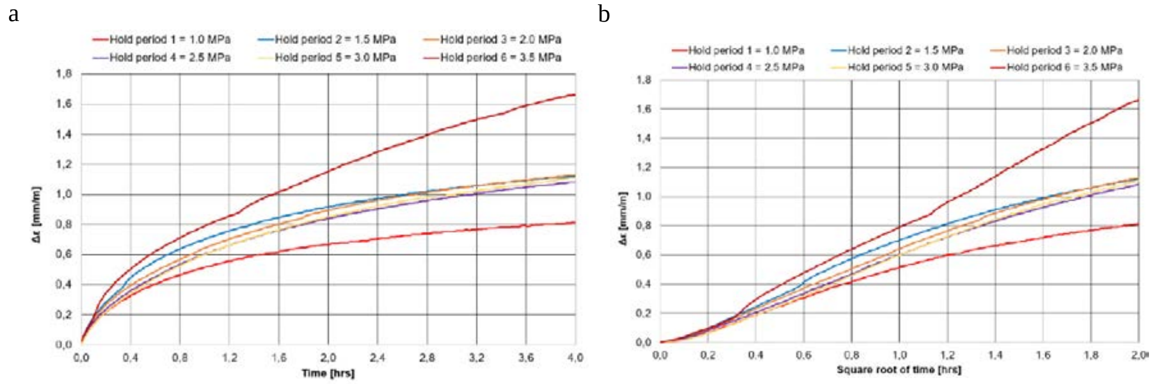

Fig. 9. (a) Axial strain timelines for test #6 in Table 3 for all stress hold periods; (b) Axial strain as a function of square root of time to determine extent of consolidation period.

Both tests again showed a slight increase in sound velocity *Vp* during the hold periods at constant stress. Both tests also showed an increase in stiffness with increasing stress, of 44 % and 66 %, respectively for test #5 and test #6. Photographs of the plugs after testing are shown in Fig. 10.

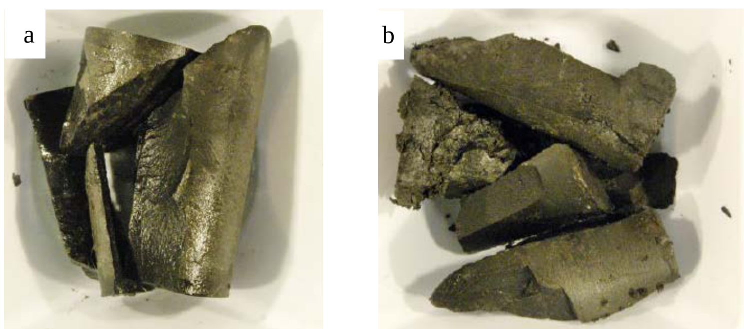

Fig. 10. (a) Pierre shale plug after creep test in acid (test #5 in Table 3); (b) Pierre shale plug after creep test in acid (test #6 in Table 3).

3.3. Comparison of creep in NaCl brine and HCl acid brine

In order to highlight potential differences in time-dependent mechanisms as pore fluid pH is altered, creep test #5 was compared to creep test #7 (Table 3) since both tests had approximately the same number of stress hold periods before failure. The same applies for creep test #4 and creep test #6.

Fig. 11 shows a comparative plot of the tests, with timelines of stress steps and accompanying axial deformation. The blue line in the plots represents the creep tests performed in brine (pH=7.31) and the red line is used for the experiments conducted in a mixture of brine and acid (pH=3.14).

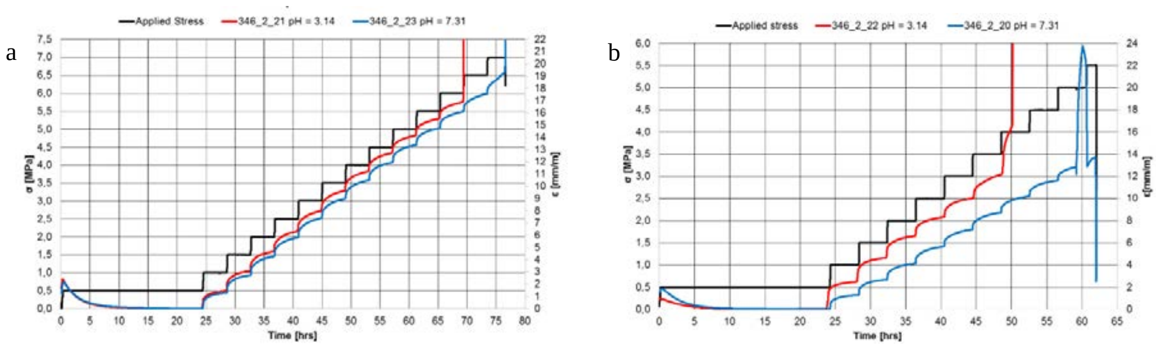

Fig. 11. Comparison of creep tests in brine with creep tests in acid (a) comparison of test #5 and test #7 from Table 3; (b) comparison of test #4 and test #6 from Table 3.

It is apparent from Fig. 11 that lowering the pH has an accelerating effect on creep, the more so the higher the stress level is, due to accumulation of strain. Focusing on the hold periods, the acid-immersed samples clearly show overall higher creep (Fig. 12 and Fig. 13). The lower failure stress of the low-pH samples may be due to either more creep deformation than for high-pH (with inherent weakening) or as evidenced in the standard UCS tests, chemical weathering weakening the core before creep testing even started.

Looking at creep rates and averaging for brine tests and for acid tests, it is also apparent that lower pH seems to accelerate deformation (Fig. 14). Likewise, consolidation time seems to be longer for brine-exposed shale than for acid-exposed (Fig. 15 and 16). This can be interpreted in two ways: either there is more creep occurring faster in low pH conditions, which is desirable for micro-annulus healing, or simply the permeability of the acid-immersed plugs was higher, which may somewhat compromise the sealing ability of the shale, reducing the healing mechanism.

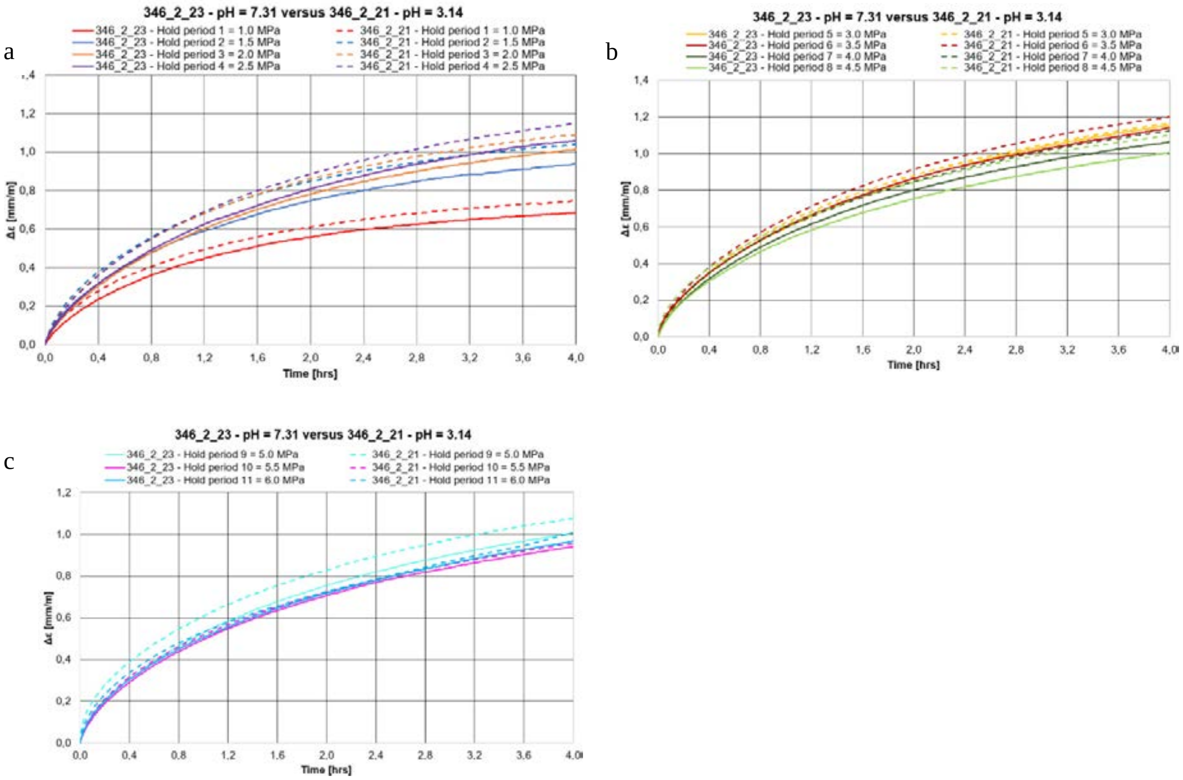

Fig. 12. Comparison of creep tests in brine with creep tests in acid for test #5 and test #7 from Table 3; (a-c) low to high stress hold values. Continuous lines: brine exposure; dashed lines: acid exposure.

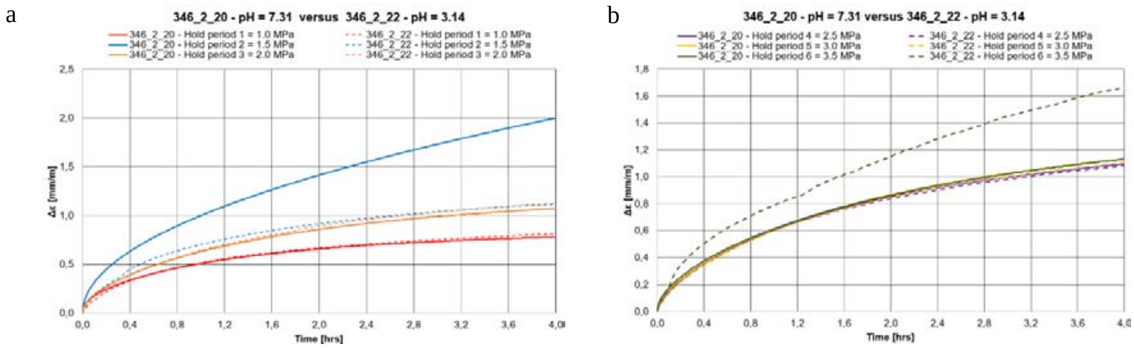

Fig. 13. Comparison of creep tests in brine with creep tests in acid for test #4 and test #6 from Table 3; (a) low stress hold values (b) high stress hold values. Continuous lines: brine exposure; dashed lines: acid exposure.

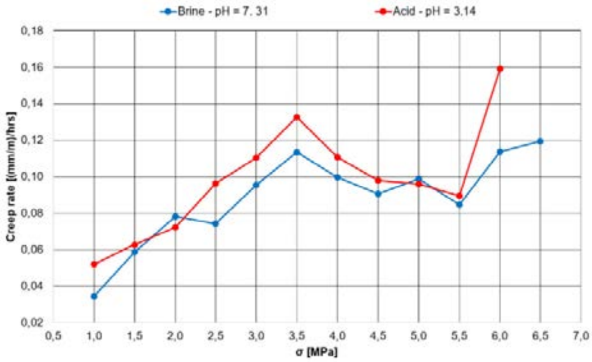

Fig. 14. Trend in average creep rates for the final 15 min of the hold periods. Average rates for brine and for acid exposed shale.

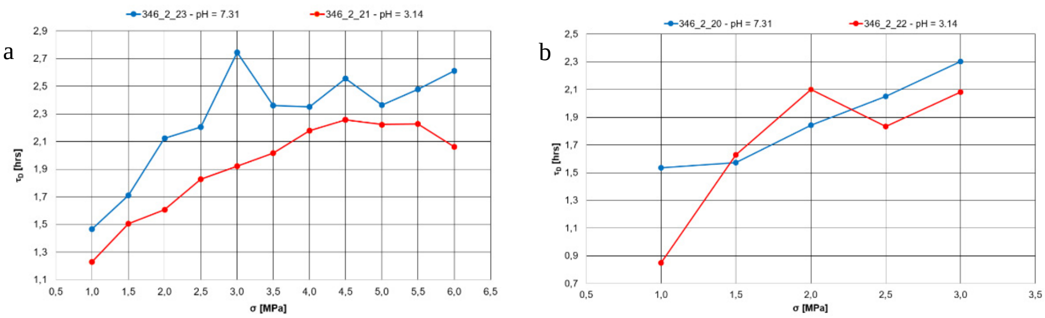

Fig. 15. Consolidation time as a function of stress. (a) comparison of test #5 and test #7 from Table 3; (b) comparison of test #4 and test #6 from Table 3.

4. Hollow cylinder creep experiments

A second series of creep experiments was conducted at GEUS in Denmark. This series explored creep in a more realistic radial symmetry, representing a wellbore in a shale section. The goal of the experiments was to assess differences in creep amount, here corresponding to a radial closure of the borehole, between brine and supercritical CO_2 fluid in the borehole. Ideally, the creep propensity of the shale exposed to CO_2 should help determine whether a typical micro-annulus between well cement and shale caprock could heal when in contact with supercritical CO_2.

Two tests were conducted, one where the shale was exposed only to 15 wt% NaCl, the other where the shale was exposed to both 15 wt% NaCl and supercritical CO_2 in a water-alternating-gas (WAG) experiment. The salt concentration was chosen to inhibit shale swelling, in order to capture specific effects related to supercritical CO_2 in addition to known effects in brine. Two hollow cylinder Pierre shale I specimens preserved in oil were sent to Denmark. The dimensions of the specimens were 3.81 cm (1.5") outer diameter, with 5 mm borehole diameter and 7.62 cm (3") length. The tests were run in the following manner:

- Clean plug in isopropanol at 40°C by solvent flush cleaning at a confining pressure of 1 MPa. Subsequently, saturate the plug with 15 wt% NaCl by flooding with backpressure at a differential pressure < 1 MPa. Both cleaning and saturation were carried out with a brass plug inserted in the inner borehole.

- Heat set-up to 40 °C (in order to allow for scCO_2 at manageable temperature)
- Insert plug in hydrostatic core holder and ramp up confining stress to 11 MPa and brine or scCO_2 pressure in the borehole to 10 MPa
- Exposure to brine or scCO_2 by flowing along the borehole axis. For the brine-only experiment, the plug was flooded with a volume of NaCl corresponding to 3 pore volumes of the inner borehole (PV). For the scCO_2 experiment, a WAG flooding scheme with 3 PV NaCl followed by 1 PV scCO_2 was performed. The WAG scheme was repeated three times before the creep experiment was performed with NaCl in the inner borehole.
- Consolidate and expose to 15 wt% NaCl for 1-2 weeks with no flow in the system
- Increase confining stress to next step (see Table 4 below) over 10 min
- Stop axial borehole flow and let shale creep for 90-130 h
- Increase confining stress to next step

Table 4. Creep test procedure at GEUS.

Step	Pore pressure [MPa]	Confining pressure [MPa]	Duration
Consolidation/CO_2/brine reaction	10	11	1-2 weeks
Loading	10	15	10 min
Creep phase I	10	15	~90 h
Loading	10	19	10 min
Creep phase II	10	19	~100-130 h
Loading	10	23	10 min
Creep phase III	10	23	~100-130 h
Unloading	0	0	2 h

In these tests, creep deformation was measured as a change in volume of the confining fluid, as the corresponding borehole fluid volume change was more difficult to measure accurately, since the pump maintaining borehole pressure was connected to a piston cylinder in addition, itself not temperature-insulated, making the changes in the borehole vanishing compared to the total fluid volume. Fig. 16 shows the measured volume changes in confining fluid, for each hold period. The creep estimate from the external deformation is probably conservative. Due to radial geometry, in the case where the shale plug is still deforming elastically, the radial displacement will be amplified near the borehole [8]. This will be even more pronounced if the wellbore is already deforming plastically [17], near the wellbore (Fig. 17).

It is clear from Fig. 16 that creep is much enhanced after exposure to CO_2 in the borehole as compared to the NaCl brine-only experiment. It seems that the difference is not only in total deformation, but also in type of creep: whereas with brine only transient creep seems to be explored, with CO_2, steady-state creep is attained. This difference is highlighted in Fig. 18, where the volume change or creep is plotted as a function of the square root of time; linear portions in this representation correspond to consolidation behavior.

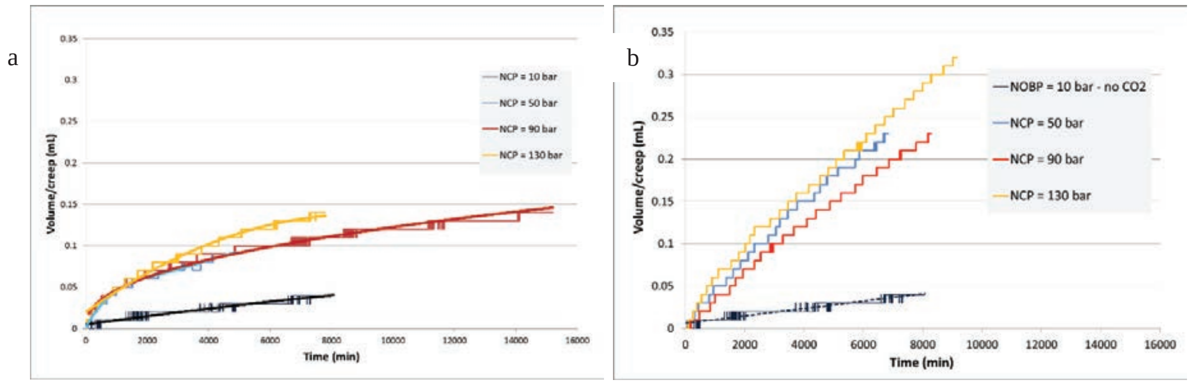

Fig. 16. Creep in hollow cylinder shale tests as estimated by the volume change of the confining fluid in the hold periods. (a) First test with 15 wt% NaCl brine in borehole; (b) test after exposure to $scCO_2$ in borehole.

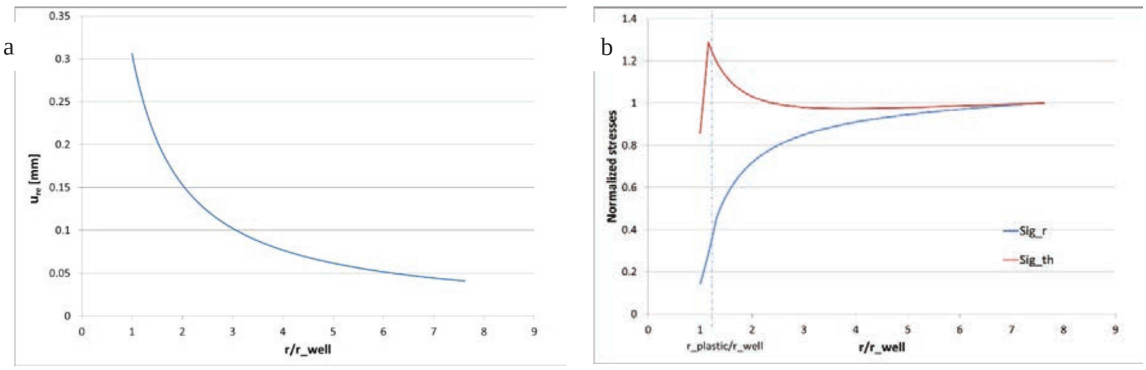

Fig. 17. (a) Elastic solution for the radial deformation for Pierre shale surrounding a well, with assumed outer radius much larger than well radius *r_well*; (b) Total radial and tangential stresses around the well, showing transition to plasticity in vicinity of well.

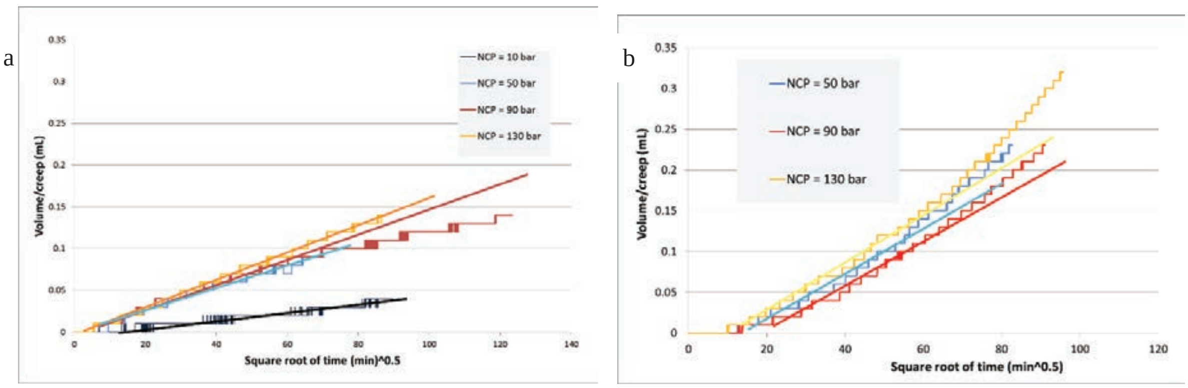

Fig. 18. Consolidation or transient creep in hollow cylinder shale tests as estimated by linear portion in square root of time plots in the hold periods. (a) First test with 15 wt% NaCl brine in borehole; (b) test after exposure to $scCO_2$ in borehole.

5. Synchrotron imagery of Pierre shale fragments

Available beam time at the Swiss Light Source (SLS) at the Paul Scherrer Institute was used to expose fragments of Pierre shale to synchrotron radiation. The SLS is located mid-way between Zürich and Basel, in northern Switzerland, with the Institute being part of the ETH Domain. Here follows further instructions for authors. The SLS offers 16 beam lines of third-generation synchrotron light source, with time available for researchers from around the world [18]. The photon have an energy of 2.4 GeV, permitting high resolution for research in materials science, biology and chemistry. The time resolution is especially important for our purpose here, since in the course of the available beam time, enough scans can be obtained such that an image of the rock fragments can be reconstructed at numerous time intervals, capturing possible evolution of the pore space, principally, under the influence of exposure to the pore fluids.

2000 slices were obtained from two fragments, one conserved in oil, the other exposed to HCl brine for 3 hours (Fig. 19). The software Avizo [19] was used to identify pore space and accordingly segment the images [20]. Porosity and porosity change from image to image could thus be evaluated, as measured in number of pixels belonging to each segmented category. The results show that the shale fragment exposed to acid increased its total volume by 9 %, as compared to initial volume, itself identical to the oil-preserved fragment. Since the HCl acid solution was identical to the one used in the uniaxial creep experiments, i.e. diluted in 3.5 wt% NaCl brine, it is not known whether this swelling would have been completely eliminated in a solution with 15 wt% NaCl, nor if the same expansion could be confirmed with CO_2-exposure. However, since downhole conditions correspond more to a mixture with low salinity, it is still possible that the observations made here are relevant to expected swelling in a micro-annulus environment.

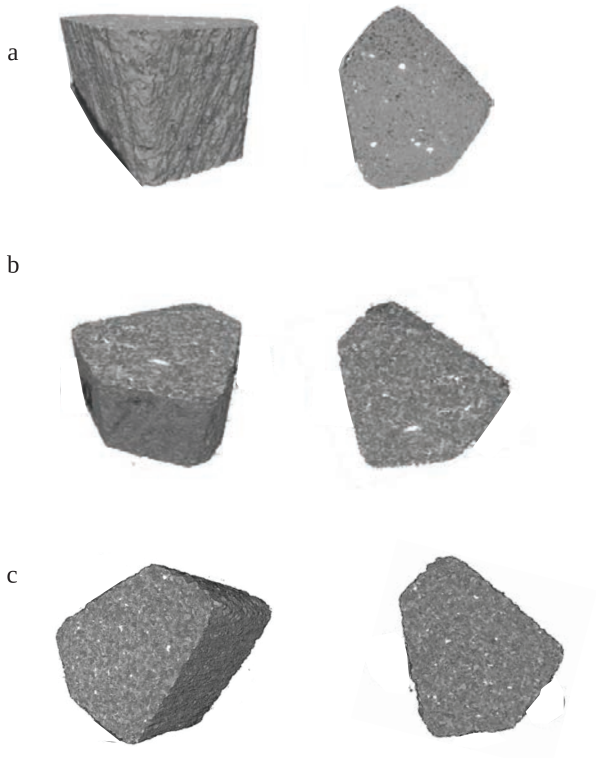

Fig. 19. Reconstructed images from synchrotron beam experiments. (a) Fragment preserved in oil; (b) fragment immersed in HCl brine solution for 3 h prior to synchrotron test, after 1':07" in synchrotron beam and (c) same fragment after 3':47" in synchrotron beam.

6. Conclusion and further work

The creep experiments related above show that Pierre shale experiences increased creep behavior when exposed to low pH brine solutions, as compared to neutral pH brine. This creep increase is further strengthened in radial geometry, as compared to uniaxial compression. The tests carried out with $scCO_2$ also confirm that creep behavior occurs at higher pore pressure conditions. This is encouraging for CO_2 sequestration, where one can thus expect that small micro-annuli forming between injection wells and surrounding shale caprock can heal relatively quickly through creeping of the shale formation towards the well.

Further work should address other types of shale material expected to be encountered above CO_2 storage reservoirs, to confirm that the range of creep deformation observed still permits healing of cracks. Systematic swelling and creep tests (perhaps at even longer hold times) should be performed with different brine systems and $scCO_2$. Finally, creep tests on cement / shale assemblies with the presence of realistic micro-annuli should be performed under exposure to CO_2-brine solutions at different pore pressure values, so as to allow gaseous CO_2 to come out of solution in contact with the shale. This, to evaluate potential creep compromise upon drying of the shale face towards the wellbore.

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