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An experimental study of the influence of stress history on fault slip during injection of supercritical CO₂

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7 *The injection of super-critical CO*² *into a depleted reservoir will alter the pore pressure of the* basin, which if sufficiently perturbed could result in fault slip. Therefore, knowledge of the 8 9 acceptable pressure limits is required in order to maintain fault stability. A two-part laboratory study was conducted on fully saturated kaolinite fault gouge to investigate this issue. 10 Previously, we showed that fault slip occurred once pore-pressure within the gouge was 11 12 sufficient to overcome the normal stress acting on the fault. For kaolinite, this behaviour occurred at a pressure similar to the yield stress. The current study shows that following a 13 slow-reduction in the maximum principal stress, as would be expected through changes in 14 effective stress, the reactivation pressure shows a stress memory. Consequently, the pressure 15 necessary to initiate fault slip is similar to that required at the maximum stress encountered. 16 Therefore, fault slip is at least partially controlled by the previous maximum stress and not the 17 current stress state. During the slow reduction in normal stress, the flow characteristics of the 18 fault remain unchanged until pore-pressure exceeds shear stress and does not increase 19 20 significantly until it exceeds normal stress. This results in fault slip, which slows the rate of flow increase as shear is an effective self-sealing mechanism. These observations lead to the 21 conclusion that stress history is a vital parameter when considering fault stability. 22

23 Keywords

24 Fault slip; Carbon Capture and Storage; kaolinite; shear testing; stress memory.

25 **1.0 Introduction**

26 The capture of CO₂ from large point source emitters and storage in the form of a super-critical fluid within geological formations has been identified as a key technology in tackling 27 anthropogenic climate change (Haszeldine, 2009; Bickle, 2009). To achieve a reduction in 28 29 emissions, significant quantities of CO₂ need to be injected into suitable geological formations capable of containing the fluid for thousands of years, such as active or depleted hydrocarbon 30 reservoirs, and saline aquifers. Several Carbon Capture and Storage (CCS) demonstration 31 32 projects have been conducted injecting megatonne scale CO₂ into geological reservoirs, such as Sleipner (saline aquifer; Norwegian North Sea; Arts et al., 2008), Weyburn (enhanced oil 33 recovery; Saskatchewan Province, Canada; Wilson and Monea, 2004) and In Salah (enhanced 34 gas recovery; Algeria; Mathieson et al., 2010). Faults are usually present in potential CCS sites 35 and this paper aims to investigate how CCS operations may impact fault-slip potential. 36 37 Perturbations of the reservoir pore fluid pressures are necessary to utilise storage capacity. These changes in pore pressure, and as a result the stress state, may result in undesired 38 geomechanical deformation that could affect the integrity of the overlying seal and any faults 39 present. 40

An existing fault will remain locked as long as the applied shear stress is less than the frictional 41 and cohesive strength of the contact. Karl Terzaghi first showed in 1923 that pore-fluid under 42 pressure has a profound effect on the physical properties of porous solids (Terzaghi, 1943). In 43 a saturated porous system, the fluid supports some proportion of the applied load, and lowers 44 the overall stress exerted through the grains. Strength is therefore determined not by confining 45 pressure alone, but by the difference between confining and pore-pressures. Hubbert & Rubey 46 (1959) showed that this behaviour applies to faults, so that a pore pressure of P_f reduces the 47 frictional strength of faults (τ_{f}), which can be represented by a criterion of Coulomb form: 48

49
$$\tau_f = C + \mu \sigma'_n = C + \mu (\sigma_n - P_f)$$
[1]

where C is the cohesive strength of the fault, μ is the coefficient of friction, σ_n is the normal 50 stress on the fault, and 'denotes effective stress. Byerlee (1978) showed that μ ranges between 51 0.6 and 1.0 for most rocks, but can be approximated as 0.75 ± 0.15 (Sibson, 1994). However, 52 phyllosilicate minerals can have a significantly lower coefficient of friction, e.g. 0.1 - 0.32 for 53 smectite (Morrow et al., 1992; Saffer & Marone, 2003), 0.35 - 0.68 for illite (Morrow et al., 54 1982, 1992; Saffer & Marone, 2003), ~0.3 for wet kaolinite (Crawford et al., 2008) and up to 55 0.8 for dry kaolinte (Morrow et al., 2000). Fault reactivation can occur when shear stress along 56 57 the fault (τ) equals τ_{f} . This condition can occur through an increase in shear stress, decrease in 58 normal stress, or an increase in fluid pressure. Additionally, chemical interactions between 59 fault gouge and CO₂ saturated brine can result in clay alteration to weaker minerals, which will result in a change in frictional behaviour that could result in fault movement. Further, the 60 variation in frictional properties with saturation state for kaolinite could result in fault 61 weakening if water migrates into an under-saturated fault core where the mineral is present. 62

Hydraulic and mechanical interactions therefore play a critical role in reactivating faults at 63 various scales in the Earth's upper crust (Scholz, 1990). Injection of fluid and the resulting 64 65 changes in the stress-state can result in the reactivation of existing faults if the pore pressure variation is sufficient in magnitude (Cappa & Rutqvist, 2011; Segall & Rice, 1995), which may 66 result in seismic failure. This effect has occurred in geothermal projects (e.g. Bachmann et al., 67 68 2012; Gan & Elsworth, 2014), waste water injection during shale gas exploration (e.g. Ellsworth, 2013), slip of an existing fault at Preese Hall (England) as a result of hydraulic 69 70 stimulation (e.g. Clarke et al., 2014; Holland, 2013), and by natural gas injection at the Castor storage site in Spain (Cesca et al., 2014). However, only micro-seismicity has been observed 71 during Carbon Capture and Storage (Verdon et al., 2013). 72

Experimental work related to slip and/or fault reactivation has tended to look at mechanical controls using analogue sand-box experiments (Krantz, 1991; Richard & Krantz, 1991; Dubois *et al.*, 2002; Bellahsen & Daniel, 2005; Del Ventisette *et al.*, 2006) or examining the flow properties of fault gouge and inferring fault weakness on geomechanical response (Crawford *et al.*, 2008; Faulkner & Rutter, 2000; Faulkner & Rutter, 2001). Modelling studies of fault reactivation potential, or slip tendency, have been conducted by several workers; as summarised by Rutqvist (2012).

80 **1.1 Objectives and previous work**

This paper presents results from an experimental study aimed at evaluating the role of stress 81 history on fault-slip potential within the laboratory. The current study represents the third stage 82 83 of a three-part investigation of the potential for fault slip during the sequestration of carbon 84 dioxide. The three parts of the study were; 1) the role of stress history on fault-flow properties, as reported in Cuss *et al.* (2016); 2) quantification of fault-slip potential as a result of elevated 85 86 pore pressure, as reported in Cuss & Harrington (2016); and 3) the role of stress history on fault slip (the current study). The scenario being investigated is for a fault that has undergone a 87 reduction in the maximum principal stress (vertical stress) through a change in effective stress, 88 with an increase in pore pressure initiating fault slip. Therefore, an increase in pore pressure is 89 directly simulated in response to the injection of CO₂. The objectives of the study are: 90

• Investigate the change in fault transmissivity during shear and/or reduction in stress;

Investigate the pore-pressure necessary to initiate fault slip following a reduction in the
maximum principal stress.

To simulate a critically stressed fault, gouge material was sheared to a stress representative of the residual shear strength, following which, vertical stress was reduced and finally pore pressure was elevated. The shearing of the fault to residual stress conditions ensured that the

97 fault plane was actively stressed. The primary aim of the study was to establish the maximum
98 pore pressure perturbations that could be employed during carbon sequestration on faults that
99 have had a complex geological history.

100 Previous experimental work at the British Geological Survey (BGS) on fracture transmissivity in Opalinus clay (Cuss et al., 2011; 2014^{a,b}), Callovo-Oxfordian claystone (Cuss et al., 2017) 101 and kaolinite gouge (Sathar et al., 2012) showed that hydraulic flow is a complex, focused, 102 transient property that is dependent upon stress history, normal stress, shear displacement, 103 fracture topology, fluid composition, and clay swelling characteristics. Cuss et al. (2016) 104 showed that fault transmissivity has clear hysteresis during unloading, demonstrating that stress 105 106 history is a vital component in dictating the flow properties of faults. Cuss & Harrington (2016) showed that fault slip occurred at a pressure dependent on the physical properties of the fault 107 gouge, with different gouges showing variations in fault slip potential. The current 108 109 experimental program aimed to extend this knowledge by investigating the influence of stresshistory on the potential for fault slip. 110

111 **2** Experimental setup

All experiments were performed using the bespoke Angled Shear Rig (ASR, Fig. 1) designed and built at the British Geological Survey. Previous experiments conducted on Opalinus Clay (Cuss *et al.*, 2009; 2011; 2014^b) showed that fracture topology is a key parameter in controlling fluid flow along fractures. To reduce the number of variables required to fully understand flow and slip potential, an analogue discontinuity with smooth fracture surfaces was investigated. The surfaces of the discontinuity were machined from steel and therefore flow could only occur through the fault gouge within the discontinuity.

119 The ASR (Fig. 1) has five key components:

Rigid steel body that had been designed to have a bulk modulus of compressibility and
 shear modulus approximately two orders of magnitude greater than the clay gouge tested,
 resulting in minimal deformation of the apparatus compared to the test sample;

 Vertical load system comprising an Enerpac hydraulic ram that was controlled using a Teledyne/ISCO 260D syringe pump, a rigid loading frame and an upper thrust block (up to 20 MPa vertical stress, 72 kN force). The Enerpac ram had a stroke of 105 mm, which means that it could easily accommodate the vertical displacement of the top block as it rides up the fault surface at constant vertical load. Note: The vertical stress created by the ram is not equal to the normal stress perpendicular to the fault plane and represents the maximum principal (vertical) stress within a reservoir;

3. Shear force actuator comprised of a modified and horizontally mounted Teledyne/ISCO
500D syringe pump designed to drive shear as slow as 14 microns a day at a constant rate
(equivalent to 1 mm in 69 days), or as fast as 0.5 mm per second, along a low friction
bearing. This capability gives a range of shear strain rate of more than 7 orders of
magnitude;

4. Pore pressure system comprising a Teledyne/ISCO 500D syringe pump that could deliver
either water or gas up to a pressure of 25.8 MPa. The syringe pump delivered fluid through
the centre of the top block directly to the fault surface;

138 5. A state-of-the-art custom designed data acquisition system using National Instruments
 139 LabVIEW[™] software facilitating the remote monitoring and control of all experimental
 140 parameters.

The experimental fault assembly consisted of precision machined 316 stainless steel top and bottom blocks (thrust blocks) with a dip of 30 degrees with respect to horizontal (the shearing direction). Previous workers (e.g. Crawford *et al.*, 2008; Tembe *et al.*, 2010) employed

144 roughened surfaces to ensure slip was not simply between the clay gouge and thrust blocks. However, the thrust blocks used in the current study were polished so as not to introduce 145 preferential pathways for flow. The top block was connected to the vertical loading 146 arrangement by means of a swivel mechanism, which was engaged to the shoulders on either 147 side of the top block. Care was taken in the design of the swivel mechanism so as to negate 148 rotation and tilting of the top blocks and shear mechanism. Two pore pressure transducers, 149 150 attached to ports that were positioned orthogonally to each other at 15 mm from the central pore fluid inlet allowed measurement of pore pressures within the fault gouge (Fig. 1a). The 151 152 thrust blocks of the apparatus were made with a contact area of $60 \text{ mm} \times 60 \text{ mm}$. The lower thrust block was longer and wider than the top one so that the contact area of the experimental 153 discontinuity could be maintained constant throughout the test. The movement of the top block 154 155 relative to the bottom block was minimised to 1 mm (< 1.5 % shear strain) so that no adverse effects were caused by new clay gouge entering the fault at the leading edge of the top block 156 and sheared clay gouge exiting at the trailing end of the thrust block. 157







160 c

Figure 1a, the shear-force actuator acted upon the angled bottom-block of the apparatus. The movement of the bottom-block was measured using a linear variable differential transducer (LVDT), which had a full range of ± 25 mm and an accuracy of 0.5 µm. Vertical travel of the thrust block was measured by a high precision non-contact capacitance displacement transducer, which had a full range of ± 0.5 mm and an accuracy of 0.06 µm. Horizontal load was measured using a load cell fitted laterally to the top-block. This measured the force resultant from lateral movement of the bottom block transmitted through the clay gouge.

168 Kaolinite was selected as the gouge material for a number of reasons. Pure mechanical tests 169 could not be conducted without gouge as the stainless steel thrust blocks would have coldwelded together. A synthetic lubricant could have been used, although this would not be 170 representative of natural faults. Therefore, it was necessary to include a realistic gouge, with a 171 clay-based material representing a faulted caprock. However, swelling clays such as illite and 172 smectite add a significant complication to the interpretation of experimental results. The 173 swelling can take several weeks to equilibrate and the injection of water risks further swelling 174 if the clay gouge is not fully saturated. Therefore, kaolinite was selected as it has a minimal 175 tendency to swell, yet displays many of the properties associated with clay gouge. The water 176

content of the kaolinite gouge was such that it was fully saturated in order to minimise swelling
resultant from water injection. Kaolinite is also a fault gouge observed in nature and has been
used by a number of previous investigators (e.g. Crawford *et al.*, 2008).

Gouge material for the experiments was prepared from powdered kaolinite, supplied by Imerys 180 from St Austell (UK). Highley (1984) report that kaolinite is of well-ordered form with coarse 181 hexagonal platelelets. The equivalent spherical diameter of the clay grains was 100 % (by 182 183 weight) less than 10 μ m, 95 % less than 2 μ m, and 85 % less than 1 μ m. Water (16 \pm 0.1 g of de-ionised water) and clay powder $(20 \pm 0.1 \text{ g})$ were stirred for five minutes giving a fully 184 saturated paste. The mixed paste was smeared uniformly onto the surface of the top block, 185 186 which was carefully lowered onto the bottom block thus forming a paste gouge. The initial thickness of the gouge was about 1 mm. However, as no lateral confinement was made of the 187 clay gouge, thickness decreased to approximately $180 \pm 10 \,\mu\text{m}$ with loading up to 10 MPa. The 188 189 excess clay squeezed from between the thrust blocks acted as a buffer preventing water from the shear bath entering the fault gouge or causing sloughing. No lateral gouge confinement was 190 191 included because sealing elements with a high frictional component would have been required along the fault surface, which would have experimentally overshadowed the low frictional 192 193 properties of the clay. However, the thickness of the gouge was seen to be sufficient as the 194 minimum thickness experienced (180 µm) is much greater than the grain size of the kaolinite used (95 % less than 2 µm). 195

Eight experiments were conducted that looked at aspects of the role of stress history on flow properties and/or fault reactivation pressure (Table 1). These tests examined the effects of instantaneous reduction in vertical stress before reactivation pressure was determined, the change in fracture transmissivity during the slow reduction of vertical stress to almost zero vertical stress, and a gradual reduction in vertical stress before reactivation pressure was determined. In addition, envelopes for starting, yield, and peak shear stress were determined for all 28 tests conducted as part of the current study and additional tests reported in Cuss & Harrington (2016). The experimental programme was designed to examine aspects of changes of effective stress during injection of carbon dioxide and the period following injection, as well as the role stress history during geological time-scales prior to injection would have on the mechanical strength of faults.

207 All tests were conducted in multiple stages, the first of which was identical. Once the apparatus 208 had been assembled, vertical stress was increased in steps up to the desired magnitude and a 209 constant pore pressure was established in the clay gouge. Vertical stress was kept constant by the Teledyne/ISCO syringe pump for the remainder of the stage. The shear actuator was 210 211 initiated to give 1 mm of strain over a 24-hour period, which equated to a strain-rate of $1.93 \times$ 10⁻⁷ s⁻¹. Data were logged every minute throughout the experiment. During the 24-hour shear 212 stage, the gouge had achieved stable-peak stress sliding. After approximately 24 hours, the 213 214 shear actuator was turned off and the next stage was conducted.

215 For tests ASR_BigCCS_21Kfl and ASR_BigCCS_34Kfl (Table 1) the Teledyne/ISCO syringe 216 pump controlling vertical stress was switched to constant refill mode, which resulted in a slow and gradual reduction of vertical stress acting on the fracture. Pore-fluid flow was monitored 217 until a condition of very low vertical stress after approximately 48 hours. For tests 218 ASR_BigCCS29Ksh - ASR_BigCCS_33Ksh (Table 1) the second stage of testing was a slow 219 reduction in vertical stress over a 24-hour period followed by a stage of fault reactivation. For 220 test ASR_BigCCS_20Kst (Table 1) an instantaneous stepped reduction in vertical stress was 221 performed, followed by a stage of fault reactivation. 222

The fault reactivation stage was performed by injecting de-ionised water into the central port of the top thrust block at a constant flow-rate of 0.25 ml h^{-1} , which was sufficient to raise pore fluid pressure within the fault gouge to 10 MPa over a 24-hour period. Fault reactivation was

observed as an instantaneous reduction in horizontal stress and simultaneous change in vertical
displacement of the load frame. Some tests showed single movements, others showed multiple
slip events, whilst some tests (see Table 1) showed no evidence of reactivation. Still minimal
reactivation was possible, causing changes in shear stress and vertical displacement that were
too small to measure.

231 Once the time of fault reactivation was known, it was possible to determine the vertical and 232 horizontal stress at reactivation. Pore pressure was calculated as the average pore pressure within the fault gouge, which was more representative of the force acting to oppose normal 233 stress over the complete fracture surface as opposed to the pump injection pore pressure that 234 235 represented a localised increase over the area of the injection filter. As shown in Figure 1a, radial flow was assumed from the central injection filter, giving an average pore pressure within 236 the gouge of 0.35 Pp, where Pp is the injection pressure. The recorded vertical and horizontal 237 stress components were rotated to represent normal and shear stress, as represented in Figure 238 1b. Throughout this paper, vertical and horizontal stresses are referred to when discussing far-239 240 field stresses, whereas normal and shear stress are used to discuss the local stress on the fault.

241 **3** Experimental results

The initial stages of all experiments were performed in a similar manner (Fig 2a). Tests were mostly conducted at 4.9 MPa normal stress for a constant vertical stress of 6.0 MPa, although test ASR_BigCCS_33Ksh was conducted at a reduced 2.0 MPa normal stress for a 2.5 MPa vertical stress. As shown in Figure 2a, good repeatability was seen during repeat testing at given normal stresses. Test ASR_BigCCS_29Ksh had a slightly reduced shear modulus, but achieved similar peak shear strength conditions (Figs. 2a, c).

The coefficient of friction (μ) for all tests was calculated as the ratio between normal stress and differential shear stress, where the differential shear stress is equal to the difference between 250 the peak stress and the starting shear stress (Figs 2b, 3). Figure 2b shows that μ ranges between 0.26 and 0.28 for the kaolinite tested, which yields a friction angle of 15.1° and a fault angle of 251 252 37.5°. As seen, the test conducted at a lower normal stress (ASR_BigCCS_33Ksh) gave a 253 similar coefficient of friction. This outcome compares well with that reported by Crawford et al. (2008) of 0.3 for 100 % kaolinite given the limited strain conducted in the current study. 254 Therefore, we infer that our shear box apparatus gives similar results to the more traditional 255 256 triaxial method of investigating fault behaviour. It should be noted that μ could be estimated as the slope of the peak stress (Fig. 3), which would give a result of 0.85, which corresponds 257 258 well with the result published by Morrow et al. (2000) for dry kaolinite. However, the kaolinite tested was fully saturated and wet. 259

Figure 2c shows an example result for test ASR_BigCCS_31Ksh and the four parameters that 260 can be calculated for each test. The starting shear stress is simply the magnitude of stress 261 262 observed before shear was initiated. The initial stress-strain response was linear, and the slope is used to calculate the shear modulus. In most tests, the deviation from this linear response 263 occurred at the yield shear stress as the departure from the linear region by 0.02 MPa. All tests 264 were checked to determine that this criterion was appropriate and that a similar result would 265 266 have been achieved by manual identification. The final shear stress parameter identified was 267 peak shear stress. As shown in Figure 2a, all tests showed classic elasto-plastic behaviour. Therefore, the peak stress condition also describes the residual strength of the gouge, although 268 a complete peak was not fully achieved by the end of the limit of 700 µm displacement (1.3 % 269 270 shear strain). Table 1 states the directly measured horizontal and vertical stress for the start, yield, and peak shear stress conditions, along with the corresponding normal and shear stress. 271

Figure 3 shows the results for starting, yield, and peak shear stresses for all experiments in the current study and those reported in Cuss & Harrington (2016). As can be seen, the data describe linear relationships with few outliers. Linear regression is shown in Figure 3 with the intercept set to zero. An example shear test is also displayed showing the variation of both normal andshear stress during the active shearing of the gouge.

Figure 4 summarises the observations made of fracture transmissivity during shearing. For test 277 ASR_BigCCS_30Ksh, a marked reduction in fracture transmissivity (T) was noted with shear. 278 Initially, T was 6.6×10^{-15} m² s⁻¹ and reduced to a steady value of 2.1×10^{-17} m² s⁻¹. A slight 279 increase in flow can be inferred at yield when the clay gouge would have undergone dilatancy, 280 281 although the noise in the data does not provide conclusive evidence. This test suggests that shearing reduces fracture transmissivity by two orders of magnitude. In contrast, quite 282 dissimilar results were seen in test ASR_BigCCS_34Kfl. From the onset of the shear stage, 283 flow within the fracture was low, averaging a steady transmissivity of 1.9×10^{-17} m² s⁻¹. Yield 284 of the fault gouge is expected to include a degree of dilatancy on the fault plane, which is likely 285 to enhance fluid flow. However, this behaviour was not observed even though the fluid 286 287 injection pressure was greater than the shear stress acting on the fault plane. Therefore, it can be concluded that in this test shear had no effect on fracture transmissivity. However, the flow 288 rate throughout the shear stage was similar to that achieved after 200 µm of shear in test 289 290 ASR_BigCCS_30Ksh.

Following the shear stage, the syringe pump controlling the vertical stress was changed to refill 291 mode to slowly lower the vertical stress. Figure 5 summarises the mechanical response 292 recorded by the data. As can be seen, all tests followed a stress path parallel with the envelope 293 294 defined for peak shear stress of kaolinite. For test ASR_BigCCS_34Kfl, which was unloaded to almost zero vertical stress, a minor curvature of the unload path was seen (Fig. 5c). Here, 295 296 the difference between the observed shear stress during unload and the envelope for peak stress is shown. This non-linear response may be derived from hysteresis in the gouge or may suggest 297 that the envelops for starting, yield and peak shear stress are not perfectly linear. 298

299 Figure 6 shows the fracture transmissivity response of the fault gouge during slow unloading over a 2-day period. Both normal and shear stress showed some hysteresis during unload (Fig. 300 6a), with pore fluid pressure exceeding normal stress within 1.5 days of unload. Initially, 301 fracture transmissivity was low (Fig. 6b), with an average around 2.0×10^{-17} m² s⁻¹, and no 302 discernible change until day 4.34, when a small variation in normal displacement was observed 303 (Fig. 6c) along with a minor change in shear stress (Fig. 6c). This variation is approximately 304 305 when the average pore fluid pressure in the gouge equalled the shear stress. Flow accelerated at day 4.6 and 4.64 when marked changes in normal displacement and shear stress were 306 307 observed. This behaviour shows a marked change in the ratio of shear stress to normal stress (Fig. 6b). The coincidental change in normal displacement and shear stress indicates that fault 308 slip was initiated. By day 4.6, the average pore pressure in the fault gouge had exceeded the 309 310 normal stress acting on the fault plane. A second event occurred at day 4.75 with a similar 311 change in normal displacement, shear stress, and shear stress to normal stress ratio. However, this event coincides with a reduction in the rate of increase of fracture transmissivity. This 312 change suggests that the second fault slip, or reactivation, resulted in a degree of self-sealing 313 of the gouge, whereas the first enhanced flow. Fracture transmissivity continued to increase 314 until day 5.07 when at a fracture transmissivity of approximately $690 \times 10^{-17} \text{ m}^2 \text{ s}^{-1}$, the gouge 315 underwent breakthrough and could no longer support fluid pressure. This event corresponded 316 to a change in normal displacement and shear stress of greater magnitude than the previous 317 318 events, which can be described as a catastrophic slip.

Figure 7 shows an example result from fault reactivation test ASR_BigCCS_31Ksh using deionised water as the injection fluid. As shown (Fig. 7a), the injection of fluid at a constant rate increased the pore fluid pressure in the fault from the starting average pore pressure of 0.1 MPa up to 4 MPa over a 7-hour period. As pore pressure rose, a single slip event was initiated, as shown by a reduction in shear stress (Fig. 7b) and change in vertical displacement (Fig. 7c). This occurred at an average pore pressure within the gouge of 3.05 MPa. No further slip events were detected in this test, whereas some tests showed multiple slip events.

Figure 8 summarises the fault-slip data following vertical stress reduction, as would occur 326 during uplift and erosion of a sedimentary basin over geological timescales prior to injection. 327 As shown in Figure 8 and Table 1, three of the five tests showed fault slip. These events 328 occurred at an average pore pressure between 2.73 and 3.82 MPa. They are clearly at a pressure 329 330 much greater than the peak shear-stress condition for kaolinite. Comparing these data following stress reduction, with the fault-slip data reported in Cuss & Harrington (2016) shows an 331 increase in fault slip pressure. In Cuss & Harrington (2016), it was concluded that fault slip of 332 333 a critically stressed fault would occur at a pressure close to the yield strength of the gouge. The current data show that following stress reduction, the fault can sustain a much greater pore 334 pressure, similar in magnitude to that expected at the maximum stress condition experienced 335 (Table 1). 336

337 Test ASR_BigCCS_20Kst (Fig. 8, open blue circle) with its vertical stress reduced in an
338 instantaneous step following shearing has a contrasting result. The stress state for fault slip is
339 similar to that expected had the reduced vertical stress been the maximum stress condition.

340 4 Discussion

Figure 4 shows that shear may influence the flow properties of faults. For test ASR_BigCCS_30Ksh, a clear reduction in flow was observed as the fault started to shear, with up two orders of magnitude reduction in transmissivity during active shearing of the kaolinite fault gouge. However, the story is not clear-cut, as shown in Figure 4b. Test ASR_BigCCS_34Kfl showed little or no response to shearing. To determine the true behaviour, it is useful to compare the current studies using the analogue fault arrangement with those conducted on competent rocks, such as reported for Opalinus clay, a Jurassic shale from

Switzerland (Cuss et al., 2011; 2014^{a,b}). Cuss et al. (2014^a) showed that shear had a profound 348 influence on fracture transmissivity. During the initial stages of shearing, one order of 349 magnitude reduction in flow was seen, while attaining a transmissivity similar to the host rock. 350 Opalinus clay has a composition of 40 - 80 % clay (9 - 29 % illite, 3 - 10 % chlorite, 6 - 20 351 % kaolinite, and 4 - 12 % illite/smectite mixed layers in the ratio 70/30), other minerals (15 -352 30 % quartz, 6 - 40 % calcite, 2 - 3 % siderite, 0 - 3 % ankerite, 1 - 7 % feldspars, 1 - 3 % 353 pyrite, <1 % organic carbon) and a total water content ranging from 4 - 19 % (Gautschi, 2001). 354 Microstructural analysis attributed the reduction of flow to clay smearing along the fracture 355 356 reducing the overall fracture aperture. Unfortunately, the use of saturated clay gouge in the current tests do not allow for the recovery of the test material at the end of the test, and therefore 357 it is not possible to make any microstructural observations. However, it is suggested that the 358 359 reduction seen during testing can only be attributed to shearing. The transmissivity seen in the tests that did not show any change in flow (ASR_BigCCS_34Kfl; 1.9×10^{-17} m² s⁻¹) is of 360 similar magnitude to that seen in the test that showed flow reduction (ASR_BigCCS_30Ksh; 361 2.1×10^{-17} m² s⁻¹). Therefore, it is suggested that during the setup of the tests, a small shear 362 occurred during the increase in vertical stress prior to initiating shear. During setup, a small 363 vertical stress is imposed on the gouge and checks are made that the top thrust block is not 364 touching the side of the apparatus, as this would severely affect results. At this time, it is 365 common to adjust the position of the top thrust block and this action results in the gouge 366 367 undergoing small amounts of shear prior to the start of the test. Further evidence of the selfsealing capacity of shear in kaolinite is the observed change in transmissivity during the 368 reduction in maximum principal stress test (ASR_BigCCS_34Kfl). In this test, at low vertical 369 370 stresses, the fracture transmissivity started to increase. At a stress state where fault slip was initiated, the rate of change of flow reduced. Therefore, shear reduced the transmissivity of the 371 fault gouge. 372

The vertical stress reduction test (ASR_BigCCS_34Kfl, Fig. 6) shows that the slow change in 373 normal stress did not result in recovery of transmissivity between day 3.1 and day 4.3, with 374 transmissivity remaining less than 10×10^{-17} m² s⁻¹. This behaviour matches the observation 375 376 reported in Cuss *et al.* (2016^a), where flow did not recover in the stepped approach undertaken until vertical stress was very low. The current data shows that transmissivity did not start to 377 increase until the average pore pressure in the fault was greater than the normal stress on the 378 379 fault. At this condition, small changes in the shear stress and vertical displacement were observed as fluid started to flow. Therefore, shear stress acting on the fault dictates when flow 380 381 started to recover. Fluid is not able to increase its ability to flow until the pressure exerted exceeds the minimum principal stress component, i.e. the shear stress. This behaviour helps to 382 explain the hysteresis seen in the system during stress reduction. 383

During test ASR_BigCCS_34Kfl, fault slip was not initiated until the average pore pressure 384 within the fault gouge exceeded the normal stress acting on the fault. Therefore, slip occurs 385 once the normal component of stress, created by the pore fluid pressure, exceeds the opposing 386 normal stress. Consequently, stress-history on fault slip could be inferred to have no influence 387 and that the magnitude of transmissivity and slip is simply dictated by the timing of when pore 388 389 fluid pressure exceeds shear or normal stresses. However, the tests conducted following a 390 reduction in normal stress showed a response that was not simply based on pore pressure overcoming normal stress, but rather the occurrence of clear stress memory (Fig. 8). The pore 391 pressure required to cause slip following normal stress reduction is of a similar magnitude to 392 393 that expected at the maximum stress encountered. For example, as shown in Table 1, test ASR_BigCCS_31Ksh was initially loaded to a normal stress of 5.84 MPa, which as shown by 394 Cuss & Harrington (2016) would predict a slip pressure of 3.93 MPa. During unloading the 395 normal stress was reduced to 2.67 MPa, which would predict a slip pressure of 1.68 MPa. The 396 slip pressure was recorded to be 3.05 MPa, which is nearly twice that expected had the fault 397

398 only been stressed to a maximum of 2.67 MPa and is less than that expected had the slip test been conducted at the maximum normal stress of 5.84 MPa. Although a small reduction did 399 occur, it is clear that slip occurs at a pore pressure significantly greater than that seen at the 400 401 current stress state. This behaviour can in part be explained by the non-recovery of flow seen in the gouge. The hysteresis seen in the transport properties is mirrored in the non-recovery of 402 the mechanical properties. However, test ASR_BigCCS_34Kfl showed that normal and shear 403 404 stresses are recovered during vertical stress reduction and it would be expected that this shows a recovery in mechanical properties. Therefore, it can be suggested that the maximum stress 405 406 encountered results in a repacking of the kaolinite grains. The reduction of vertical stress results in the elastic recovery of stress, but the pore throats of the re-packed kaolinite do not recover. 407 This textural change gives the gouge a memory of the maximum stress encountered. This 408 409 outcome is not overly surprising as many factors of clay-rich rocks, such as mechanical strength 410 and flow properties, are related to the over-consolidation ratio, which is directly related to the maximum stress-state that the rock has encountered during geological history (Burland, 1990; 411 Skempton, 1970; Horseman et al., 1987). 412

413 One test was performed with an instantaneously stepped reduction in normal stress (ASR BigCCS 20Kst). The resultant slip pressure for this test corresponded with what was 414 415 expected had the reduced normal stress of 2.5 MPa been the maximum stress encountered. Therefore, the instantaneous, stepped, reduction in vertical stress appears to have "re-set" the 416 stress-memory of the fault. It should be noted that only one test was conducted with a stepped 417 418 reduction in vertical stress. This observation requires further exploration and is outside of the scope of the current study. It is unlikely that this phenomena is derived from drainage of the 419 kaolinite gouge, as even at the low fracture transmissivity recorded, the pore pressure in the 420 gouge is quick to equilibrate. 421

422 Figure 9 shows the results from the current study plotted in the Mohr space for stress history ASR_BigCCS_33Ksh (Fig. 9a) and vertical stress reduction flow 423 test test 424 ASR_BigCCS_34Kfl (Fig. 9b). The stress history test shows that at the maximum stress state, 425 the fault was far from the condition necessary to result in slip. The slow reduction in vertical stress did not bring the stress state to a condition of slippage and hence no movement was 426 observed. Fault slip was initiated by raising pore fluid pressure, but as can be seen, movement 427 428 occurred at a pressure much greater than the Mohr approach would have predicted. This error in the Mohr approach is created by the analysis not taking into account the stress history of the 429 430 fault. The flow test conducted until the fault was fully exhumed (test ASR_BigCCS_34Kfl), shows that the high pore pressure used in this test (5 MPa) still resulted in an initial condition 431 of stability. The reduction in vertical stress resulted in failure of the fault at a stress condition 432 433 greatly below that predicted by the Mohr analysis. Again, this outcome is a result of the simple analysis not including stress history. Therefore, it can be concluded that the widely accepted 434 Mohr analysis of fault reactivation becomes problematic when the fault under consideration 435 has undergone a complex stress-history. When such a history is the case, the stress history has 436 to be incorporated. 437

438 Careful consideration needs to be taken with respect to the time component when interpreting 439 the current experiments, which were conducted over a short duration. The initial shearing stage lasted approximately 24-hours, the slow reduction of stress took a further 24 hours, while the 440 final pore-pressurisation stage took up to 24 hours. All three stages were undertaken 441 442 considerably faster than would occur in the natural or man-made systems of interest. Considering unloading during exhumation and erosion over geological history prior to 443 injection, the event could take millions of years, eight orders of magnitude slower than the 444 current experiments. Considering a stress change based on an engineering time scale of 10 445 years would be four orders of magnitude slower. During pressurisation, the time-scale of 446

pressure increase is likely to range between a month to a few years, therefore representing a 447 pressure rise 2 to 3 orders of magnitude slower. It should also be noted that the magnitude of 448 the pore pressure perturbation is likely to be much less than encountered during the current 449 450 experiments. In designing the experiments, a 24-hour period was determined as a trade-off between the time necessary for the clay gouge to drain and the total time available for all 451 experiments. Despite the low fracture transmissivity of $< 5 \times 10^{-17}$ m² s⁻¹, the small surface area 452 of the experimental apparatus (60×60 mm) drained relatively quickly. For instance, the tests 453 operated at 0.25 MPa pore pressure would take approximately 1 - 2 hours for the pressure to 454 455 reduce and plateau once the injection syringe pump was turned off. Therefore, drainage is not a major limitation of the test methodology. However, over geological and engineering time-456 scales, processes may act that would alter this behaviour. For example, slow compaction and 457 458 consolidation of the gouge material may alter the cohesion of the fault plane, which will alter 459 the slip potential. However, this change is likely to strengthen the fault gouge and lead to an enhanced stress memory effect as slip pressure would increase. The current study clearly 460 461 identified a stress memory effect of slip potential common with the hysteresis seen in flow properties in a stress-reducing system (e.g. Cuss et al., 2016). Further work is required to 462 investigate this feature for realistic rocks and natural conditions to verify the observations. Such 463 experiments would aim to investigate the influence of other geological processes that may 464 occur, such as compaction and clay smearing, on the potential for fault slip. Accounting for all 465 466 the limitations of the current experimental investigation, stress memory in flow and slip potential still requires further attention. 467

The main implication of the current work is that a consideration only of the current stress-state will result in inaccurate predictions of flow and fault slip potential. For the flow properties, not accounting for stress history will result in the over-estimate of flow. Therefore, the faults will be over-estimated for their ability to transmit fluids. In terms of fault slip, not accounting for

stress-history will result in the under-prediction of the pore pressure necessary to initiate fault 472 reactivation. Consequently, faults that have a significant stress-history prior to injection will 473 474 be stronger than predicted and may sustain a greater pore pressure variation. It should be noted 475 that exhumation and erosion of overlying sediments prior to injection is not the only possible driver for stress variation in a reservoir used for carbon sequestration. Localised variations will 476 result from pore-pressure variations, thermally derived stresses resultant from injection 477 478 cooling, or from stresses resultant from reservoir compaction. These stress drivers are all likely to result in relatively minor changes in the stress-state acting on faults and may not result in 479 480 noticeable stress-memory. The inaccurate prediction in flow is not so straightforward. Whilst it will result in faults appearing to be better seals than predicted, it will result in the under-481 estimation of pore fluid pressure under a sealing fault, which could result in higher pore-fluid 482 483 pressures acting upon the fault and/or caprock. This latter observation suggests that stress-484 history should not be ignored if pore pressures are likely to get close to the magnitude that would either cause fault slip or new failure. Stress history is also important to understand the 485 flow properties of all faults to yield an understanding of the fluid distribution within a reservoir 486 during injection. 487

The hysteresis seen in flow and mechanical behaviour in clay-rich rocks is well established in 488 489 engineering geology and is one of the basic concepts of critical-state soil mechanics (Roscoe et al., 1958; Schofield & Wroth, 1968). However, such phenomena are not routinely 490 incorporated into structural geological studies that are based on rock mechanics, which in 491 492 general defines driving forces and not relaxing stress-states. Several studies have looked at geological history, and hence stress history, such as Rutter et al. (2012) who aimed to unravel 493 494 the stress history of the Carboneras fault zone. However, this study did not result in stress history predictions of the faults themselves. Aldrich et al. (1991) examined the stress history 495 of a geothermal site in Honduras. By unravelling the regional stress fields from the faults, they 496

497 were able to examine the stress field and potential for reactivation of faults during hydraulic fracturing for geothermal energy. Whilst the magnitude of stress change was not determined, 498 their work clearly showed the influence of stress history on the fault system. Several studies 499 500 have looked at slip potential or reactivation (e.g. Morris et al., 1996; Gartrell et al., 2002; Colletini et al., 2005; Moeck et al., 2009). However, none incorporate the hysteresis seen in 501 flow and mechanical behaviour observed during the current study. A future study could be 502 503 conducted for an existing, or future, reservoir identified for sequestration of carbon dioxide to reconstruct the full stress history that all faults have experienced. Flow modelling could then 504 505 be used to determine the full consequence of the observations from the current study and those of Cuss & Harrington (2016). This approach will establish whether stress history on faults plays 506 a significant role on fluid flow within the reservoir and the influence it has on slip potential. 507

508 5 Conclusions

This paper presents results from an experimental study of eight shear tests on a simulated fault angled 30° to the shear direction with a fault gouge of saturated kaolinite. The primary aim of the study was to investigate the influence of stress-history on fault slip by comparing the current data with that of Cuss & Harrington (2016). The main conclusions of the study were:

The injection of pore-fluid into a depleted reservoir could result in fault slip if the pore
pressure perturbation is sufficient.

Following a slow reduction in the maximum principal stress, the pore pressure necessary
 to initiate fault slip has a clear memory, with slip occurring at a pressure similar to that
 observed at the maximum stress condition.

Ignoring stress-history can result in an under-estimate of the pore pressure necessary to
 initiate fault reactivation.

The stress "memory" observed is suggested to be created by the re-packing of the clay
gouge, which occurs at the maximum stress condition.

- The instantaneous reduction in vertical stress results in fault slip and a resetting of the
 stress memory. This is an unlikely scenario during storage of CO₂.
- Shear has been observed to be an effective self-sealing mechanism during the initial stages
 of shearing and also following fault slip.
- Fault transmissivity remains unchanged during the slow reduction in the maximum
 principal stress until the average pore pressure exceeds the shear stress (minimum principal
 stress). Fault slip is not triggered until average pore pressure exceeds the normal stress on
 the fault.
- The Mohr analysis of fault reactivation is not sufficient to predict when fault reactivation
 is likely to occur because stress history is not included.
- Stress-history is a vital parameter in determining both fault transmissivity and fault slip
 potential. Not accounting for stress history will result in an under-estimate of the maximum
 pore pressure that can be achieved in a reservoir. However, ignoring stress-history will
 result in an under-estimate of fault transmissivity that may result in higher than predicted
 pore pressure in the vicinity of faults, which if sufficient, could result in slip. Therefore, it
 is concluded that stress-history should be considered fully when assessing reservoirs for
 the storage of CO₂.

539

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704 c

Figure 1 The Angled Shear Rig (ASR). a) Schematic of the apparatus; b) plan view of the slip-plane labelled in (a); c) Description of the stresses. Note that the apparatus directly measures vertical and horizontal stresses, whereas normal and shear stress were determined by stress rotation.





Figure 2 Mechanical strength data for shear tests conducted on kaolinite gouge material. a)
Shear stress versus displacement; b) Variation of coefficient of friction during shear strain; c)
Identification of starting shear stress, yield shear stress, peak shear stress, and shear modulus.



Figure 3 Strength parameters for shear tests conducted on kaolinite gouge. Clear linear trends are seen for the starting shear stress, the yield shear stress, and the peak shear stress. An example stress-strain result for test ASR_BigCCS_31Ksh. Note closed symbols are from the current study, open symbols are from Cuss & Harrington (2016).



Figure 4 Flow variation during shearing for (a) test ASR_BigCCS_30Ksh; and (b)
ASR_BigCCS_34Kfl. Fracture transmissivity is seen to reduce during test
ASR_BigCCS_30Ksh (a), whereas it is low throughout test ASR_BigCCS_34Kfl (b).



Figure 5 Mechanical response during fault unloading. All tests show that shear stress
reduces in a manner parallel with the defined peak shear stress envelope (a, b). Close

- examination suggests a minor non-linearity of the data when comparing the observed shear
- stress with the peak stress envelope for test ASR_BigCCS_34Kfl (c).



Figure 6 Data during vertical stress reduction stage of test ASR_BigCCS_34Kfl. a) normal
stress, shear stress and average pore pressure during stress reduction; b) fracture transmissivity
and ratio of shear to normal stress; c) normal displacement and adjusted shear stress (shear
stress minus a polynomial).





- 743 Figure 7 Example results from fault reactivation test ASR_BigCCS_31Ksh. a) The injection
- of de-ionised water creates a pore pressure increase. Fault reactivation is identified by a
- reduction in shear stress (b) and vertical displacement on the fault plane (c).



Figure 8 Results of fault slip following normal stress reduction. Results of the current study





Figure 9 Mohr analysis of results from the current study. a) Stress-history test
ASR_BigCCS_33Ksh, where stress has initially reduced as a result of the slow lowering of
vertical stress, followed by a further effective stress reduction as pore pressure was increased
in the fault gouge. b) Vertical stress reduction flow test ASR_BigCCS_34Kfl where effective
stress was lowered until very low stress conditions, which due to the high pore pressure in the
gouge greatly exceed the normal stress on the fault. greatly exceed the normal stress on the

Experiment	Type of test		Normal (or Vertical	stress (MI	Pa)	Shear (or Horizontal) stress (MPa)					Pore	Reactivat	ion pore-pressu		
		Average	Start	Yield	Peak	Reactivation	Shear modulus	Start	Yield	Peak	Reactivation	pressure during shear (MPa)	From maximum stress	From minimum stress	Observed	Observation
ASR_BigCCS_20Kst	#1	4.87 (5.34)	4.88 (5.92)	5.43 (6.01)	5.84 (6.26)	2.67 (2.64)	461	2.81 (1.53)	4.26 (3.19)	4.98 (3.94)	2.65 (1.98)	0.50	3.91	1.65	1.53	Close to reduced stress condition
ASR_BigCCS_21Kfl	#2	5.63 (6.17)	4.88 (5.93)	5.53 (6.04)	5.88 (6.31)	/	497	2.79 (1.51)	4.50 (3.46)	5.00 (3.96)	/	0.25	3.95	/	/	Reactivation not observ
ASR_BigCCS_34Kfl		5.66 (6.21)	4.90 (5.93)	5.45 (6.06)	5.85 (6.30)	0.54 (0.58)	455	2.84 (1.57)	4.23 (3.14)	4.96 (3.91)	0.47 (0.43)	5.00	3.94	0.36	1.75	Reactivation not observ
ASR_BigCCS_29Ksh	#3	5.59 (6.16)	4.93 (5.96)	5.51 (6.08)	5.82 (6.27)	/	341	2.88 (1.61)	4.38 (3.30)	4.93 (3.89)	/	0.25	3.92	1.67	/	Reactivation not observ
ASR_BigCCS_30Ksh		5.62 (6.19)	4.91 (5.96)	5.50 (6.06)	5.85 (6.31)	2.50 (2.67)	454	2.82 (1.53)	4.39 (3.32)	4.93 (3.88)	2.15 (1.98)	0.25	3.95	1.67	2.73	Closer to maximum stre condition
ASR_BigCCS_31Ksh		5.60 (6.17)	4.92 (5.95)	5.48 (6.07)	5.84 (6.29)	2.49 (2.68)	441	2.86 (1.58)	4.30 (3.21)	4.93 (3.87)	2.12 (1.94)	0.25	3.93	1.68	3.05	Closer to maximum stre condition
ASR_BigCCS_32Ksh		5.64 (6.19)	4.91 (5.95)	5.48 (6.07)	5.84 (6.27)	/	437	2.83 (1.56)	4.30 (3.21)	4.97 (3.93)	/	0.25	3.92	1.66	/	Reactivation not observ
ASR_BigCCS_33Ksh		2.40 (2.59)	2.04 (2.43)	2.28 (2.48)	2.49 (2.67)	2.45 (2.62)	324	1.23 (0.78)	1.84 (1.52)	2.11 (1.97)	2.10 (1.92)	0.25	2.21	1.64	3.82	Greater than maximum stress condition

Table 1 – List of all experiments undertaken as part of the current study and corresponding results. #1 = stress history test with a stepped reduction

in vertical stress; #2 = flow test during slow reduction in vertical stress to zero; #3 = stress history test with slow reduction in vertical stress;

760 parenthesis indicate measured horizontal and vertical stresses.