1	An experimental study of the potential for fault reactivation during changes in gas and
2	pore-water pressure.
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Abstract: The injection of CO_2 into a depleted reservoir will alter the pore pressure, which if 8 9 sufficiently perturbed could result in fault reactivation. This paper presents an experimental study of fault reactivation potential in fully saturated kaolinite and Ball Clay fault gouges. 10 11 Clear differences were observed in fault reactivation pressure when water was injected, with 12 the addition of mica/illite in Ball Clay seen to reduce the pressure necessary for reactivation. Slip occurred once pore-pressure within the gouge was sufficient to overcome the normal 13 14 stress acting on the fault. During gas injection localised dilatant pathways are formed with 15 approximately only 15 % of the fault observing an elevated gas pressure. This localisation is insufficient to overcome normal stress and so reactivation is not initiated. Therefore faults 16 are more likely to conduct gas than to reactivate. The Mohr approach of assessing fault 17 reactivity potential gave mixed results. Hydro-mechanical coupling, saturation state, 18 mineralogical composition and time-dependent features of the clay require inclusion in this 19 approach otherwise experiments that are predicted to be stable result in fault reactivation. 20

21 Highlights

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The shear apparatus allowed fault reactivation to be observed and investigated for variations in clay gouge mineralogy

24	•	Reactivation pressure related to yield strength and starting shear strength in kaoninte
25		and Ball Clay respectively
26	•	Gas not able to initiate fault reactivation with faults becoming conductive to gas as
27		opposed to creating slip
28	•	Mohr-circle approach to assessing safe pressure changes insufficient to predict
29		reactivation

30 Keywords

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31 *Fault reactivation; multiphase flow; kaolinite; Ball Clay; shear testing.*

32 **1.0 Introduction**

The capture of CO₂ from large point source emitters and storage in the form of a super-33 critical fluid within geological formations has been identified as a key technology in tackling 34 anthropogenic climate change (Haszeldine, 2009; Bickle, 2009). To achieve a reduction in 35 emissions, significant quantities of CO₂ need to be injected into suitable geological 36 formations capable of containing the fluid for thousands of years. It has been estimated that 37 approximately 30 billion barrels of CO₂ need to be injected annually (Zoback & Gorelick, 38 2012). Several demonstration projects have been conducted injecting megatonne scale CO₂ 39 40 into depleted hydrocarbons reservoirs, such as at Sleipner (Norwegian North Sea; Arts et al., 2008), Weyburn (Saskatchewan Province, Canada; Wilson et al., 2004) and In Salah 41 42 (Algeria; Mathieson et al., 2010). Storage of CO₂ in depleted reservoirs offers the security of storage with an effective top-seal that previously acted as a seal to hydrocarbons. 43

The use of a depleted reservoir will play a role in the performance of the storage facility. During depletion, pore pressure within the reservoir will have been lowered during hydrocarbon extraction and as a result the reservoir will have subsided. The injection of super-critical fluid into a depleted reservoir will result in the opposite, with pore pressure

increased and heave of the reservoir. The use of injection and extraction boreholes can 48 minimise this effect, with water injected at a rate similar to the extraction rate of the 49 hydrocarbon during drawdown, and extraction of aquifer water at a similar rate to CO₂ 50 injection during carbon sequestration. Local deformation will still occur though if the two 51 boreholes are well spaced, as seen during the In Salah CO₂ storage project in Algeria 52 (Mathieson et al., 2010). Perturbations of the reservoir pore fluid pressures are required in 53 54 order to initiate flow out of, or into the reservoir. These changes in pore pressure, and as a result the stress state, may result in undesired geomechanical deformation that could affect 55 56 the integrity of the overlying seal. Zoback & Gorelick (2012) identified the risk to security from a geomechanical point of view, while Economides & Ehlig-Economides (2009) showed 57 that an upper pressure limit exists for CCS, above which the seal is potentially compromised 58 59 due to the formation of fractures. However, Vilarrasa & Carrera (2015) state that large 60 earthquakes are unlikely to be triggered during CO₂ injection in sedimentary basins and therefore leakage is not likely to be induced. Verdon et al. (2013) examined the deformation 61 62 observed at injection sites and noted that the geomechanical response was complicated and non-intuitive at Weyburn, small at Sleipner due to the high permeability of the reservoir, and 63 uplift and microseimic activity was noted at In Salah. Therefore, reservoirs need to be 64 considered on an individual basis based on their geometry and the properties of the geology 65 present. 66

Hydraulic and mechanical interactions play a critical role in reactivating faults at various
scales in the Earth's upper crust (Scholz, 1990). Injection of fluid and the resulting changes in
the stress-state can result in the reactivation of existing faults (Cappa & Rutqvist, 2011;
Segall & Rice, 1995), which can result in felt seismicity. This has occurred in geothermal
projects (e.g. Bachmann *et al.*, 2012; Gan & Elsworth, 2014), waste water injection during
shale gas exploration (e.g. Ellsworth, 2013), during hydraulic fracturing (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 during Carbon Capture and
Storage (Verdon *et al.*, 2013).

Faults with high clay content within the fault core may have a permeability as low as 10^{-22} m² 76 (Faulkner & Rutter, 2000). Such flow barriers within a reservoir may increase overpressure 77 locally, which could result in fault reactivation (Rutqvist et al., 2007; Rinaldi et al., 2015). 78 This may create an open migration pathway for CO₂ to escape from the reservoir (Zoback & 79 80 Gorelick, 2012), although no correlation between seismicity and leakage was found in numerical modelling (Rinaldi et al., 2014^{a,b}). Experimental work related to fault reactivation 81 has tended to look at mechanical controls using analogue sand-box experiments (Krantz, 82 1991; Richard & Krantz, 1991; Dubois et al., 2002; Bellahsen & Daniela, 2005; Del 83 Ventisette et al., 2006) or examining the flow properties of fault gouge and inferring fault 84 85 weakness on geomechanical response (Crawford et al., 2008; Faulkner & Rutter, 2000; Faulkner & Rutter, 2001). 86

87 Modelling studies of fault reactivation potential, or slip tendency, have been conducted by several workers; some of which are summarised here, see Rutqvist (2012) for a more 88 89 comprehensive summary of numerical modelling. Streit & Hillis (2004) estimated fault stability for underground storage of CO₂ based on the Mohr-Coulomb approach of predicting 90 individual fault strength. A similar approach using slip tendency analysis using the 3-91 dimensional Mohr-space has been proposed by Leclère & Fabbri (2013). Williams et al. 92 (2015) calculated slip tendency based on the ratio of shear to normal stress for faults within 93 94 the Moray Firth, North Sea, to determine which were critically stressed. A critically stressed fault is one where the shear stresses acting upon the fault is at the limit of the frictional 95 strength of the fault, i.e. as soon as stress is increased on the fault it will result in slip. They 96 97 found that pore fluid increases as modest as several kPa were sufficient to cause reactivation

98 for certain fault segments, with a maximum pore pressure of 20 MPa. However, Zhang et al. (2015) used a coupled geomechanical-fluid flow modelling approach and demonstrated that 99 reactivation wasn't likely in the South West Hub of Western Australia. Coupled reservoir-100 101 geomechanical numerical modelling (Rutqvist, 2011) has been used to simulate fault/fracture zone reactivation induced by CO₂ injections (Cappa & Rutqvist, 2012; Rinaldi & Rutqvist, 102 2013) to assess the potential for fault instability and shear failure (Cappa & Rutqvist, 2011). 103 104 Gan & Elsworth (2014) modelled the role of both pore fluid change and temperature drawdown on fault reactivation in relation to geothermal projects and showed that 105 106 temperature variations needed to be considered when examining fault stability.

A fault will remain locked as long as the applied shear stress is less than the strength of the 107 contact. Karl Terzaghi first showed in 1923 that pore-fluid under pressure has a profound 108 effect on the physical properties of porous solids (Terzaghi, 1943). In a saturated porous 109 110 system, the fluid supports some proportion of the applied load lowering the overall stress exerted through grains. Strength is therefore determined not by confining pressure alone, but 111 112 by the difference between confining and pore-pressures. Hubbert & Rubey (1959) showed this applies to faults; a pore pressure of P_f reduces the frictional strength of faults (τ), which 113 can be represented by a criterion of Coulomb form: 114

115
$$\tau_f = C + \mu \sigma'_n = C + \mu (\sigma_n - P_f)$$
[1]

116 where *C* is the cohesive strength of the fault, μ is the coefficient of friction, σ_n , is the normal 117 stress on the fault, and ' denotes effective stress. Byerlee (1978) showed that μ ranges 118 between 0.6 and 1.0, but can be approximated as 0.75 ± 0.15 (Sibson, 1994). Fault 119 reactivation can therefore occur when shear stress along the fault (τ) equals τ_f . This condition 120 can occur through an increase in shear stress, decrease in normal stress, or an increase in fluid 121 pressure.

This paper presents results from an experimental study aimed at evaluating fault reactivation 122 potential within the laboratory in two fault gouges. The current study represents the second 123 stage of a three-part investigation of the potential for fault reactivation during the 124 125 sequestration of carbon 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 126 reactivation potential as a result of elevated pore pressure (the current study); and 3) the role 127 128 of stress history on fault reactivation. The scenario being investigated is for a static boundary condition for stress acting on a fault with an increase in pore pressure initiating fault 129 130 reactivation; therefore directly simulating an increase in pore pressure in response to the injection of CO₂ during sequestration. The objectives of the study were: 131 • Investigate whether fault reactivation could be detected using a shear apparatus with an 132 angled fault-plane within the laboratory; 133 • Investigate the mechanical properties of two clay gouges during shear; 134 • Variation in fault reactivation behaviour between two clay gouges; 135 • Variation in fault reactivation potential as a result in elevation of gas or water pressure. 136 In order to simulate a critically stressed fault, gouge material was sheared to a stress 137 representative of the residual shear strength before pore pressure was elevated. This ensured 138 that the fault plane was actively stressed. Equation (1) shows that the coefficient of friction 139 140 dictates the strength of a fault, although cohesion also contributes to fault strength. Two clay gouges were selected so as to determine whether different material properties would alter the 141 potential for fault reactivation, or whether a single parameter could be used to estimate the 142

stress state at failure for different gouge compositions. The primary aim of the study was to

144 establish maximum pore pressure perturbations that could be employed during carbon145 sequestration.

Previous experimental work at the British Geological Survey (BGS) on fracture transmissivity in Opalinus clay (Cuss *et al.*, 2011; 2014^{a,b}) and kaolinite gouge (Sathar *et al.*, 2012) showed that hydraulic flow is a complex, focused, transient property that is dependent upon stress history, normal stress, shear displacement, fracture topology, fluid composition, and clay swelling characteristics. The current experimental program aimed to extend this knowledge by investigating the potential for fault reactivation by elevating pore pressure within gouge filled discontinuities.

153 2 Experimental setup

All experiments were performed using the bespoke Angled Shear Rig (ASR, Figure 1) designed and built at the BGS. 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. In order to reduce the number of variables required to fully understand flow, 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.

161 The ASR (Figure 1) comprised of 5 key components:

Rigid body that had been designed to have a bulk modulus of compressibility and shear
 modulus approximately 2 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
meant that it could easily accommodate the vertical displacement of the top block as it
rode 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) along a low friction bearing;

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.

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

The experimental fault assembly consisted of precision machined 316 stainless steel top and 181 182 bottom blocks (thrust blocks) with a dip of 30 degrees with respect to horizontal (the shearing direction). The thrust blocks were polished so as not to introduce preferential pathways for 183 flow. The top block was connected to the vertical loading arrangement by means of a swivel 184 185 mechanism which was engaged to the shoulders on either side of the top block. Care was taken in the design of the swivel mechanism so as to negate rotation and tilting of the top 186 blocks and shear mechanism. Two pore pressure transducers, attached to ports which were 187 188 positioned orthogonally to each other at 15 mm from the central pore fluid inlet allowed measurement of pore pressures within the fault gouge (see Figure 1). The thrust blocks of the 189 apparatus were made with a contact area of $60 \text{ mm} \times 60 \text{ mm}$. The lower thrust block was 190

191 longer than the top one so that the contact area of the experimental discontinuity could be192 maintained constant throughout the test.

As shown in Figure 1, the shear force actuator acted upon the angled bottom-block of the 193 apparatus. The movement of the bottom-block was measured using a linear variable 194 195 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 196 capacitance displacement transducer, which had a full range of ± 0.5 mm and an accuracy of 197 198 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 199 200 the clay gouge.

201 Gouge material for the experiments was prepared from either powdered kaolinite or Ball Clay 202 (as described in Table 1); 16 ± 0.1 g of de-ionized water was added to 20 ± 0.1 g of oven dried clay powder. The water and clay were then stirred for five minutes giving a fully 203 saturated paste. The mixed paste was smeared uniformly onto the surface of the top block, 204 205 which was then carefully lowered onto the bottom block thus forming a paste gouge. The initial thickness of the gouge was in the order of 1 mm. However, as no lateral confinement 206 207 was made of the clay gouge, thickness decreased to approximately $70 \pm 10 \ \mu m$ with loading up to 10 MPa and clay was squeezed from between the thrust blocks; this excess material 208 acted as a buffer preventing water from the shear bath entering the fault gouge or causing 209 sloughing. No lateral gouge confinement was included as this would require sealing elements 210 that would have a high frictional component along the fault surface compared with the low 211 frictional properties of the clay. 212

Twenty-eight experiments are described in this paper (Table 2); of these, 13 were fault reactivation experiments conducted using water as the injected fluid, 7 were fault reactivation

experiments conducted with gas as the injection fluid, and the remaining 8 are reported only 215 for mechanical data. For all 28 experiments the first stage was to conduct a shear experiment. 216 Once the apparatus had been assembled, vertical stress was increased in steps up to the 217 218 desired magnitude. Vertical stress was kept constant by the Teledyne/ISCO syringe pump for the remainder of the experiment. The shear actuator was initiated to give 1 mm of strain over 219 a 24 hour period; this equated to a strain-rate of 1.93×10^{-7} s⁻¹. Data were logged every 220 minute throughout the experiment. Within the 24-hour long shear experiment, the gouge had 221 222 achieved stable peak stress sliding. After approximately 24 hours the shear actuator was 223 turned off and constant pressure was maintained in the vertical loading ram.

Fault reactivation experiments were performed by injecting fluid into the central port of the 224 top thrust block. For water injection, de-ionised water was injected at a constant pressure of 225 0.25 MPa throughout the shear experiment. Once stable pressure had been achieved, the 226 injection syringe pump was switched to a constant flow-rate of 0.25 ml h⁻¹, sufficient to raise 227 pore fluid pressure within the fault gouge to 10 MPa over a 24-hour period. For gas injection 228 229 experiments, an interface vessel was filled with 170 ml of helium at a pressure of 2 MPa. 230 Cuss et al. (2015) showed that the gas entry pressure of kaolinite gouge was in excess of 5 MPa, therefore a starting pressure of 2 MPa would not result in gas flow within the gouge. 231 The injection syringe pump was switched to constant flow rate operation and delivered 10 ml 232 h^{-1} of water into the base of the interface vessel, raising the pressure within the gas to 233 sufficient levels to allow gas entry within a 5 hour time-frame. Helium was selected as the 234 permeant as it is inert and to allow direct comparison with previous experiments (Sathar et 235 al., 2012; Cuss et al., 2015). Fault reactivation was observed as an instantaneous reduction in 236 shear stress and change in vertical displacement of the load frame. Some tests showed single 237 movements, others showed multiple slip events, whilst some tests showed no sign of 238 reactivation. 239

240 Once the time of fault reactivation was known, it was possible to determine the vertical and horizontal stress at reactivation. Pore pressure was calculated as the average pore pressure 241 within the fault gouge, this being more representative of the force acting to oppose normal 242 stress over the complete fracture surface as opposed to the maximum pore pressure, which 243 represented a localised increase. As shown in Figure 1, radial flow was assumed from the 244 central injection filter. This would result in a pore pressure gradient as shown in Figure 8a, 245 246 giving an average pore pressure within the gouge of 0.35 P_p , where P_p is the injection pressure. The recorded vertical and horizontal stress components were rotated to represent 247 248 normal and shear stress. Throughout this paper, vertical and horizontal stresses are referred to when discussing far-field stresses, whereas normal and shear stress are used to discuss the 249 local stress on the fault. 250

Gas entry-pressure was determined using the methodology described in Cuss *et al.* (2015), by comparing the pressure predicted from Boyle's law with the observed gas pressure. Using the ideal gas law it is possible to determine the mass flux into the clay gouge. A departure is seen between predicted and observed once gas starts to enter the clay; from this the gas entry pressure is then derived.

256 **3 Experimental results**

A total of 28 tests were conducted during the current study, as shown in Figure 2 and Table 2; of these, 22 were conducted on kaolinite and 6 were Ball Clay. All 28 tests are reported here for their mechanical shear content, the initial stage of each test was identical for all tests. Following shearing, a total of 20 of the tests were conducted as fault reactivation experiments; a total of 13 water-injection reactivation experiments were conducted, 7 gasinjection.

Figure 2 shows the results for the 24-hour long shear tests conducted, with all tests conducted 263 with the same protocols irrespective of whether they were fault reactivation tests or not, or 264 whether they were gas or water injection. Tests on kaolinite gouge ranged in vertical stress 265 266 from 1.1 to 6.4 MPa, while for Ball Clay the range was 2.6 to 6.3 MPa. As shown in Figure 2a and b, good repeatability was seen during repeat testing at given vertical stresses for both 267 kaolinite and Ball Clay gouges. Figure 2c shows an example result for test 268 269 ASR_BigCCS_11K and the four parameters that can be calculated for each test. The starting shear stress is simply the magnitude of stress observed before shear was initiated. The initial 270 271 stress-strain response was linear, the slope of which described the shear modulus. In most tests, this was observed as a well-defined linear response, the deviation from which describes 272 the yield shear stress. The yield stress was determined as the departure from the linear region 273 274 by 0.02 MPa; all tests were checked that this criterion was appropriate and that a similar 275 result was being achieved as would be by manual identification. The final shear stress parameter identified was peak shear stress. As shown in Figure 2, all tests showed classic 276 elasto-plastic behaviour. Therefore the peak stress condition also describes the residual 277 strength of the gouge. Table 2 outlines the vertical and shear stress for the start, yield, and 278 279 peak shear stress conditions.

Figure 3 and Table 3 show the results for starting, yield, and peak shear stresses for all 280 experiments in the current study. As can be seen, the data describe linear relationships with 281 few outliers. Linear regression is shown in Figure 3 with the intercept set to zero; as shown in 282 Table 3, this does not significantly reduce the R^2 achieved showing that it is a good 283 approximation. Comparing the trends for kaolinite and Ball Clay shows that Ball Clay has a 284 higher starting shear stress; therefore the starting condition is not simply the translation of 285 vertical stress into the horizontal direction with the difference being due to the mineralogical 286 difference of the two clays. Ball Clay, however, has lower yield strength with a much reduced 287

288 linear relationship observed between stress and strain. Ball Clay is also a weaker material and is not able to sustain as high a shear stress as kaolinite. Therefore the addition of illite, quartz, 289 and possibly water content are resulting in a reduced strength compared with pure kaolinite. 290 291 Figure 4 shows the data for shear modulus; as shown in Table 2 tests ASR BigCCS 19BC and ASR_BigCCS_25Kg gave anomalously low and high shear moduli respectively. Figure 4 292 shows that kaolinite is a more stiff material when stress is below 5.5 MPa, with Ball Clay 293 showing greater stiffness above this condition. However, considerable spread is seen in the 294 kaolinite data compared to Ball Clay, with R^2 of 0.37 and 0.95 respectively. The slope of 295 peak shear stress represents the coefficient of friction (μ), whilst the intercept represents the 296 cohesion (C) of the material, as shown in Figure 4b. From this parameter it is possible to 297 derive the angle of internal friction (ϕ) and fault angle (θ), as shown in Table 4, from the 298 299 relationships:

300
$$\mu = tan\phi$$
 and $\phi = 90^\circ - 2\theta$ [2]

Figure 5a-c shows an example result from fault reactivation test ASR_BigCCS_14BC using 301 water as the injection fluid. As shown (Figure 5a), the injection of fluid at a constant rate 302 303 increased the pore fluid pressure in the fault from the starting average pore pressure of 0.1 MPa up to 9 MPa over a 24-hour period. As pore pressure rose, a series of slip events were 304 initiated, as shown by a reduction in shear stress (Figure 5b) and change in vertical 305 306 displacement (Figure 5c). A total of nine slips occurred, with the first occurring at an average pore pressure in the gouge of 1.27 MPa. The time between slip events decreased with 307 subsequent slip events, this was not related to the increase in pore pressure gradient with time 308 309 as the pore pressure between slip events also decreased. Therefore the gouge was undergoing strain softening as a result of reactivation, with further slip events taking less energy to 310 initiate. 311

All 13 reactivation tests conducted resulted in slip of the critically stressed fault plane as a 312 result of elevated pore pressure, results are shown in Figure 6, Table 2, and Table 3. The 313 reactivation pressure is defined as the pore pressure that is sufficient to initiate fault 314 315 reactivation and slip. Kaolinite gouge showed good repeatability for the three tests conducted at 2.7 MPa vertical stress. A linear relationship is seen between reactivation pressure and 316 vertical stress, with a value of R^2 of 0.91 (Figure 6a, Table 3). This is reduced to 0.39 when 317 the intercept is set to zero, with this suggesting that reactivation in kaolinite gouge is 318 controlled by the yield strength of the clay. A less well defined linear relationship is observed 319 for Ball Clay, with a value of R^2 of 0.56 (Figure 6b, Table 3); note that tying the intercept to 320 zero does not significantly alter the statistics. The results suggest that the initial starting stress 321 controls the reactivation pressure. This indicates that Ball Clay has little strength and that the 322 323 first slip occurs once vertical stress has been overcome. Plotting reactivation pressure against 324 vertical stress (Figure 6c) shows that both clays form similar relationships with differences in the intercept, which may be related to the difference in relative strength of the two clays. 325 326 However, plotting the data in the differential stress versus effective mean stress space (Figure 6d) gives a single fault reactivation envelope for both clays. 327

During gas injection, the addition of water in the base of the interface vessel results in an 328 exponential increase in gas pressure dependent on the starting volume of the gas and the 329 change in volume, which is related to the rate at which the syringe pump delivers water into 330 the vessel. The form of the pressure response can be predicted from Boyle's law, as can the 331 STP (standard temperature pressure) flow of gas into the fault gouge. Initially the STP flow 332 rate is very small and rises gradually but then the rate of increase of the flow rate abruptly 333 increases. The pressure at which this occurs is identified as the gas entry pressure. Gas peak 334 pressure is simply the maximum gas pressure experienced. Gas breakthrough is the pressure 335 when gas was able to reach the outside of the top block, resulting in a reduction in gas 336

pressure. Table 5 shows the gas entry and maximum gas pressure for all gas injection
experiments. Note that test ASR_BigCCS_22Kg was started from 2.5 MPa, which was
greater than the gas entry pressure.

340 The results for the fault reactivation tests conducted on kaolinite using gas as the injection fluid markedly contrast with the results seen for water injection (Figure 5d-f, Table 2). Only 341 one test resulting in evidence of fault reactivation, as shown in Figure 5d-f. Assuming radial 342 flow, this occurred at an average pore pressure within the gouge of 1.65 MPa, which is lower 343 than that seen during water injection (average of 2.1 MPa). As shown in Figure 5d, fault 344 reactivation resulted in increased flow into the gouge, as seen by a marked change in slope of 345 346 pore pressure, this increased until gas pressure peaked at 5.58 MPa, when gas injection was stopped. This was followed by a reduction in pressure to approximately 1 MPa as gas escaped 347 along a conductive pathway between the injection filter and the outside of the gouge. The 348 349 reduction of gas pressure accelerated at Day 1.13, suggesting that a further gas pathway had managed to reach breakthrough. 350

351 Figure 7 shows the results from the fault reactivation experiments using gas as the permeant. No sensitivity to vertical stress was observed in gas entry pressure or the maximum gas 352 pressure achieved (Figure 7a). Only one experiment resulted in fault reactivation. As seen, 353 gas pressure was not able to achieve the level observed during water injection, except for one 354 test conducted at a low vertical stress of 1.13 MPa. However, this test did not show any signs 355 of fault reactivation. Figure 7b shows that no significant differences were apparent in shear 356 stress between tests conducted with gas or water injection. As plotted, the shear stress at gas 357 358 entry and that during reactivation with water entry perfectly correspond, clearly demonstrating that mechanically there were no differences between the two types of test. 359

360 4 Discussion

The current study successfully reproduced fault reactivation in the laboratory and allowed differences to be noted between water and gas injection, as well as variations related to clay gouge mineralogy.

364 The mechanical aspects of the current study produced well constrained data for two fault gouges. Very good repeatability was seen for repeat tests conducted at near identical 365 boundary conditions. Well constrained linear relationships were noted for starting, yield and 366 peak shear stress. Few outliers were seen in all tests and these occurred in the starting shear 367 stress. These tend to remain unexplained and are probably due to small shear movements 368 occurring during the setup of the experiment. It should be noted that the anomalous data 369 370 points did not result in anomalous yield or peak strength results; strengthening the assumed hypothesis of shear movement during setup. As starting shear stress is not the primary dataset 371 these are not viewed as problematic. The differences between the starting shear stress for the 372 373 two gouges is likely to represent variations in cohesion. Although zero cohesion has been assumed, a better fit to the Ball Clay data is achieved with cohesion of 0.33 MPa (Table 4), 374 375 whereas little change is seen in kaolinite. However, the addition of quartz and mica/illite 376 results in more vertical stress being translated into the horizontal direction, suggesting that Ball Clay is a weaker material with less frictional strength. This is also apparent in the peak 377 stress condition and lower coefficient of friction. This observation is in contrast with 378 Crawford et al. (2008), who showed that sheared gouge samples showed a continuous 379 reduction in frictional strength with increasing clay fraction. This suggests that either the 380 mica/illite content played a significant role in weakening the gouge, or that the nature (grain 381 size, roundness etc) differed between the two studies. It could also be a result in variations in 382 clay saturation, although in all tests the gouge was close to 100 % saturation. Figure 4 shows 383 that the results from this study correspond with Byerlee's law (Byerlee, 1978) and therefore 384 that the measured values are consistent with natural rocks. 385

386 The fault reactivation study was able to clearly identify reactivation. However, some hydraulic injection tests resulted in single reactivation, whereas others resulted in multiple 387 slip-events (see Figure 5b). The cause for this is uncertain. One hypothesis may be that a 388 larger single slip event releases more energy than a smaller one. However, no variation in 389 shear stress reduction or magnitude in dilation was observed. In general, all slip events using 390 water tended to have similar magnitudes in shear stress reduction and dilation. Variations in 391 392 the number of slip events were seen for the four tests conducted with a kaolinite gouge at a 393 vertical stress of about 2.6 MPa. Figure 8a shows the assumed pore pressure distribution 394 within the fault gouge. Cuss et al. (2011) reported that not all of a fracture surface in Opalinus Clay was conductive during hydraulic flow and that deformation along a sheared 395 fracture was localised into zones of differing texture. It is possible that the initial pore 396 pressure distribution is similar to that described by Figure 8a, but as slip occurs the gouge is 397 modified resulting in parts becoming conductive, whilst other parts are self-sealed by the 398 399 shear movement. In tests that showed limited slip events it is possible that the gouge contained conductive channels following shear that resulted in pore pressure dissipation and 400 pressure not increasing as expected. In tests that did show multiple slip-events, these channels 401 402 did not result in pore pressure dissipation and pressure continued to ramp, becoming sufficient to cause further slip events. Data is not available to fully determine the reasons for 403 these observations. 404

The results for hydraulic injection produced reliable data that showed a marked difference between the two clay gouges. As shown in Figure 6, reactivation tended to occur when the average pore pressure exceeded the yield strength of kaolinite, whereas in Ball Clay reactivation occurred at a stress below the initial starting shear strength. This results in two different reactivation envelopes as shown in Figure 6c. This clearly shows that mica/illite and/or quartz reduces the stress at which a fault will reactivate. However, considering data in the effective mean stress versus differential stress space (Q-P) results in a well constrained single reactivation envelope, as seen in Figure 6d. Effective mean stress (P) is defined simply as the mean stress minus the effect of pore pressure, i.e. $P = ((\sigma_1 + \sigma_2 + \sigma_3)/3) - P_f$. The differential stress (Q) is simply defined as the difference between the maximum and minimum principal stresses, i.e. $Q = \sigma_1 - \sigma_3$. This suggests that in Q-P, mineralogy plays no role in determining reactivation. This envelope suggests that reactivation will occur when differential stress is 2.5 times the effective mean stress:

418
$$Q = 2.5P$$
 [3]

This relationship can be used to determine the pore pressure likely to cause fault reactivation along existing features. Therefore the likelihood of fault reactivation is dependent on pressure within the storage reservoir, the magnitude of which will depend on the quantity of fluid injected and the flow properties of the reservoir.

A marked difference was noted for fault reactivation when gas was injected into the clay 423 gouge. In general, it can be stated that fault reactivation was not possible when gas was 424 injected. As shown in Figure 8a, modelled pore pressure distribution in the clay gouge 425 assuming radial flow would result in a pore pressure of approximately 300 kPa at the 426 monitoring pore pressure filter location on the fault surface given the experimental boundary 427 conditions. However, Figure 8b shows typical data recorded during gas and water injection 428 experiments (tests reported in Cuss et al., 2014^a), showing that pore pressure within the 429 gouge was significantly less than 300 kPa. For the case of gas injection the pore pressure 430 observed in the gouge was effectively atmospheric, indicating no elevation of pore pressure 431 as a result of gas injection. All tests were typical of this response. In order to understand gas 432 and water flow in clay gouge a number of observations can be drawn upon. In Cuss et al., 433 (2011) it was reported that less than 50 % of a fracture surface was hydraulically conductive 434

435 in Opalinus Clay, as identified from the injection of fluorescein. In Sathar et al. (2012) it was reported that localised streams of bubbles were seen following gas breakthrough in injection 436 experiments. These observations led to the development of the Fracture Visualisation Rig 437 438 (see Wiseall et al., 2015). Using a 50 mm thick 110 mm diameter quartz fused glass window, water and gas injection into clay gouge can be observed. As shown in Figure 8c, the injection 439 of gas into a kaolinite gouge results in the formation of a number of dilatant gas pathways, 440 441 until a pathway reaches the outside of the apparatus and facilitates breakthrough, resulting in the elastic closure of the dilatant pathways. This helps to explain the low pore pressure within 442 443 the gouge, with no pathway intercepting the pore pressure observation ports. As reported in Cuss *et al.* (2012^a; 2014^a), clay rich materials are able to sustain very high pressure gradients 444 445 when gas is injected. Even when gas is flowing, the elevated gas pressure is not transmitted 446 to the bulk pore fluid. Therefore this is not a phenomena restricted to the geometry of the 447 current experimental apparatus, the clay gouge selected, or saturation of the gouge.

Figure 9 shows the conceptual model to explain the differences seen between water and gas 448 449 injection. During water injection, radial flow is observed resulting in a pore pressure 450 distribution within the clay gouge. The force exerted perpendicular to the fault can be equated as the average pore pressure within the gouge. This means that an elevated pressure sufficient 451 to overcome cohesion within the gouge is possible, resulting in slip. In the case of gas 452 injection, localised dilatant gas pathways are formed. This compresses the clay walls either 453 side of the pathway, but results in only a localised perturbation of the clay. Although large 454 gas pressures may be present within the dilatant features, the average pore pressure within the 455 gouge is much less than for corresponding pressures of water injection. Figure 8d suggests 456 that a maximum of 15 % of the gouge would be made of dilatant gas pathways, meaning that 457 458 the force exerted perpendicular to the fault would be much less than for water injection; a multiplier of injection pressure of 0.35 for water and 0.14 for gas. The flow properties of 459

kaolinite and Ball Clay are such that it is much easier for a dilatant pathway to form and
propagate to a condition of breakthrough, than it is to result in an average force sufficient to
overcome the vertical stress and cohesion of the gouge, which would result in slip.

One anomalous observation was the single gas injection experiment that resulted in fault 463 reactivation (test ASR BigCCS 23Kg). This occurred at a gas pressure of 4.71 MPa, which 464 is less than the absolute water pressure (average of 6 MPa) seen to cause reactivation during 465 hydraulic testing. As discussed above, pore pressure is not well transmitted from the gas 466 phase to the water-saturated clay, as seen by low pore pressure within the gouge. Therefore, 467 the upward force acting on the surfaces of the fault would be highly localised. Each test was 468 469 conducted as identical as practicable, using the same mixture of clay, setting up procedures, quantity of gas, and gas injection rate. As seen in Figure 2a and Table 2, the mechanical part 470 of the experiment gave near identical results for test ASR_BigCCS_23Kg as 471 472 ASR_BigCCS_26Kg, the latter of which did not reactivate. However, Figure 5 clearly shows a reactivation event at a time that does not correspond with initial gas entry, with a small 473 474 reduction in shear stress and change in vertical displacement. This shear movement resulted in an increased gas flow into the gouge. Repeating the experiment (test ASR_BigCCS_26Kg) 475 and conducting a further experiment at lower vertical stress (test ASR_BigCCS_27Kg) 476 showed no evidence of reactivation. Close examination of the test data for test 477 ASR BigCCS 23Kg has not identified anything different between this and the non-478 reactivating gas injection tests and the reason for slip remains undetermined. 479

Gas transport properties showed no sensitivity to vertical stress, with a constant gas entry and maximum gas pressure. Part one of the current study, as defined in the introduction and reported in Cuss *et al.* (2016), examined the hydraulic flow properties of kaolinite gouge as a function of vertical stress. This data showed a clear reduction in hydraulic transmissivity of kaolinite gouge, reducing from 4.3 to 1.5×10^{-14} m² s⁻¹ between a vertical stress of 0.8 and 10

MPa. Such a reduction would be expected for gas flow. As described in Cuss et al. (2015), 485 repeat testing in the current apparatus resulted in a repeatable gas entry pressure, but once gas 486 flow was initiated, little repeatability in flow properties was observed. This was attributed to 487 488 differences in the number and distribution of pathways, as shown during fracture visualisation tests (Wiseall et al., 2015; Figure 8c). The pressure at which gas pathways form 489 is reproducible as dictated by the strength of the gouge. Once formation begins, the number 490 491 of pathways arbitrarily alters and therefore transport properties also vary. It would be expected that as the gouge is compressed to a greater degree by increased vertical stress that 492 493 gas entry would increase. However, the nano-metre scale of clay minerals means that the entry pressure is not altered. This might change at greater vertical stresses or if gouge was not 494 able to be squeezed out from between the thrust blocks. Cuss et al. (2015) report the variation 495 496 in flow properties for fractures of varying orientation to the shear direction under constant vertical stress. Experiments conducted at 0, 15, 30 and 45° degrees to the shear orientation at 497 constant vertical stress can be viewed as variations in normal stress to a single fracture. As 498 499 with the current study, little variation in gas entry pressure was observed.

The primary aim of this study was to test experimentally the controls on fault reactivation and the safe operational pressure limits of CCS. It is common to apply Mohr-Coulomb concepts to estimate fault reactivation potential and therefore the current study is presented in Mohr space in Figure 10, . with the frictional sliding envelope determined from the coefficient of friction shown in Figure 4b. The fault angle represents the slip-plane with respect to the direction of shear. For the current experimental set-up the 2-D Mohr circle has been used, with the size of the Mohr circle bound by the vertical stress and the horizontal stress.

507 Some tests resulted in fault reactivation at a pressure very close to that predicted by the Mohr 508 approach (e.g. Figure 10a,b). Contrary, tests shown in Figure 10c,d show that reactivation 509 occurred at a stress far below the pressure predicted from the frictional sliding envelope.

510 These tests show a stress state that should be stable. Figure 10e shows an example of a test where reactivation occurred at a pore pressure greater than predicted. Generally these results 511 are mixed. Some tests are successfully predicted, some under-estimated and some over-512 513 estimated. An under-estimate of pore-pressure variation is acceptable, where an over-estimate 514 means that faults that are predicted to be stable would in fact slip. Figure 10f shows the results for the single gas test that resulted in reactivation. As seen, the Mohr approach shows 515 516 that reactivation should have occurred at this gas pressure and that the approach would appear valid. However, Figure 10g,h show that at least three tests, with possibly a fourth, were at a 517 518 stress condition where reactivation should have been observed. Therefore the localised nature of gas pathway formation is not fully accounted for in the approach. Given the mixed results, 519 caution needs to be used when using the Mohr approach to determining fault reactivation 520 521 potential. Should a maximum pore pressure be restricted to 0.5 - 0.75 of the pore pressure predicted by the Mohr approach then this approach may be satisfactory. 522

The Mohr-Coulomb approach to predicting fault reactivation is used by many studies 523 524 reported, e.g. Cappa & Rutqvist, 2011, 2012; Rinaldi & Rutqvist, 2013; Rinaldi et al., 2015. 525 The current study suggests that as a first approximation the approach is valid, although the complete prediction of the pore-pressure is more complex. This may be due to artefacts of the 526 527 experimental set-up or be associated with complex coupling that occurs as a result of the hydro-mechanical properties of the clay gouge that are not fully described by the simplified 528 approach presented here. It is clear that this is an area that requires further research in order to 529 fully appreciate the physics driving fault reactivation. The observations of the current study 530 also suggest that free-gas will not result in fault reactivation. However, it should be 531 acknowledged that the experimental geometry meant that gas was able to drain from the fault 532 gouge and that in nature sufficient quantities of gas may become present within faults to 533 initiate reactivation. 534

535 One limitation of the current study was not being able to inject super-critical CO₂. Therefore the emphasis of the study was on changes in pore-water pressure as a result of CO₂ injection 536 and should free-gas be present in the reservoir, the consequence of elevated gas pressure on 537 538 existing faults. The influence of super-critical CO₂ directly in contact with faults was not investigated, nor was the influence of CO₂ should a gaseous phase form. The study was 539 conducted at low pressures compared with in situ stress states and further investigation is 540 needed to determine whether similar findings would be found at representative reservoir 541 542 pressures.

543 **5** Conclusions

This paper presents results from an experimental study of 28 shear tests on a simulated fault angled 30° to the shear direction with a fault gouge of kaolinite or Ball Clay. The main conclusions of the study were:

Mechanical data showed good repeatability, with Ball Clay having less frictional strength, but becomes stiffer than kaolinite at vertical stresses greater than 5 MPa. Good linear relationships were seen for starting, yield and peak shear stress; the latter corresponding to the coefficient of friction for the gouge material, with achieved results correspond with Byerlee's law.

The addition of mica/illite and/or quartz reduces the cohesive strength of the gouge. As
 Crawford *et al.* (2008) showed that quartz content increases the frictional properties it is
 likely that mica/illite is responsible for the reduction in cohesion.

• Fault reactivation occurred at pressure related to the yield strength in kaolinite and at a pressure less than the starting shear stress in Ball Clay. This shows that Ball Clay has a much lower frictional strength than kaolinite. A single envelope was achieved for fault reactivation potential when data were viewed in the differential (*Q*) versus effective

mean stress (*P*) space; stating reactivation will occur when Q = 2.5 P. This suggests that the *Q*-*P* representation is irrespective of mineralogy, at least for the range of conditions tested in the current work.

• During gas injection, only one test showed reactivation and this occurred at a pressure predicted by the Mohr approach. However, 3 further tests predicted to slip showed no evidence of movement.

Gas entry and maximum gas pressure showed no pressure sensitivity to vertical stress.
 The gas entry pressure is dictated by the frictional properties of the clay gouge, which do
 not significantly alter over the range of vertical stresses investigated. The maximum
 pressure achieved is also related to the frictional properties and therefore also showed
 little to no sensitivity to vertical stress over the limited stresses investigated.

• Gas injection results in localised discrete pathways, with pressure elevated in approximately 15 % of the fault area. This means that the average pressure exerted normally to the fault is not sufficient to induce slip. During hydraulic injection the pore pressure distribution is more evenly dispersed and results in a greater normal force that is sufficient to initiate slip. No difference is seen in the mechanical data, demonstrating that the lack of reactivation is only due to the localisation of gas flow.

• The frictional properties of the fault gouge dictate that it is more likely to become 577 conductive to gas than to reactivate.

The Mohr approach of assessing fault reactivity had mixed results, but is generally viewed as a valid approach. Some tests had good predictions of pore pressure at reactivation, whilst most where either under or over-estimated. An over-estimate of pore pressure adds a safety margin to predictions and is acceptable. However, an under-estimate in gas pressure means that faults predicted to be stable may in reality reactivate.
Given the mixed results, caution needs to be used when using the Mohr approach to

determining fault reactivation potential. A safety margin can be used to ensure that favourably oriented faults do not reactivate. In the simple form presented, the Mohr-Coulomb approach did not capture the full complexity observed. This is likely a result of flow localisation resulting in complex pore-pressure distributions or due to hydromechanical coupling, which is complex in clays.

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750 Figure 1 Schematic of the Angled Shear Rig (ASR).



Figure 2 Mechanical strength data for shear tests conducted on (a) kaolinite and (b) Ball
Clay gouge materials. From these data it is possible to identify starting shear stress, yield
shear stress, peak shear stress, and shear modulus (c).



Figure 3 Strength parameters for shear tests conducted on (a) kaolinite and (b) Ball Clay
gouge materials. Clear linear trends are seen for the starting shear stress, the yield shear

stress, and the peak shear stress. Comparison can be made between kaolinite and Ball Clay

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766 gouges (c).
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Figure 4 Shear properties for tests conducted on kaolinite and Ball Clay gouge. A) Shear modulus data. At stresses below 5 MPa it can be seen that kaolinite is a more stiff material, whereas Ball Clay becomes stiffer above these stress levels. B) Calculation of coefficient of internal friction, showing that the current data correspond to Byerlee's law (Byerlee, 1978).



Figure 5 Example results from fault reactivation tests using water (a-c) and gas (d-f) as injection fluid. A) The injection of water creates a pore pressure increase. Fault reactivation is identified by a reduction in shear stress (b) and dilation on the fault plane (c). A total of 24 slip events were identified until the fault could no longer hold pore pressure. d) The injection of gas creates a pore pressure increase. Fault reactivation is identified by a reduction in shear stress (e) and dilation on the fault plane (f). As shown, only one slip event was identified. Gas flow is seen to increase following slip, as seen by a reduction in gas gradient (d).



Figure 6 Results from the fault reactivation study using water as an injection fluid. A) Reactivation pressure for kaolinite can be seen to approximate the yield shear stress. B) In Ball Clay the reactivation stress approximates the starting shear stress. C) Plotting reactivation stress against vertical stress gives two relationships, whereas plotting data in the effective mean stress versus differential stress (Q-P) space gives a unified envelope for predicting fault reactivation (d).



Figure 7 Results for fault reactivation using gas. A) Gas entry pressure and maximum gas
pressure show no sensitivity to vertical stress loading. B) Comparing the shear stress at gas
entry with the level seen at reactivation for water experiments shows no difference between
the injection fluids.



Figure 8 Observations of pore pressure within the fault gouge. A) Modelled result for pore
pressure distribution assuming radial flow, indicating that pore pressure at the monitoring

- 802 ports should be approximately 300 kPa. B) Observed pore pressure at the monitoring filter
- 803 location shown in (a) during testing shows pore pressure is greatly below that modelled, with
- a very low pressure seen during gas injection (Cuss *et al.*, 2014^a). C) Processed photograph
- 805 from a Fracture Visualisation test showing a 60×60 mm square area with dilatant gas
- 806 pathways. D) Location of pathways predicting < 15 % coverage.



Figure 9 Model for fault reactivation. A) Water injection: The elevated water pressure results in a pore pressure profile as shown. The average pore pressure acting vertically is sufficient to cause fault reactivation. B) Gas injection: Pore pressure within the gouge is only locally increased by gas injection. The gouge compresses to accommodate dilatant pathways, as opposed to classical two-phase flow, resulting in a low average pore pressure acting vertically that isn't sufficient to cause reactivation.



Figure 10 Representation of the test data in Mohr space. (a-b) Examples of where the Mohr approach gives good approximation for fault reactivation; (c-d) examples where reactivation occurred at pressures lower than the Mohr approach would predict; (e) example where reactivation didn't occur until a magnitude greater than predicted; (f) gas pressure sufficient to result in reactivation; (g-h) demonstration that four tests during gas injection would have been predicted to reactivate.

Gouge	Supplier	Geological information	Location	Composition
Kaolinite	Imerys	well-ordered form, coarse hexagonal platelets ¹	St Austell, UK	100 % kaolinite
Ball Clay		A1 seam; Tertiary, Poole Formation, Oakdale Clay Member)	Arne Clay Pit, Wareham, UK	37% kaolinite, 35% mica/illite and 26% quartz, together with some feldspar ²

Table 1 – Description of the clay gouge materials used during the current study. ¹ Highley, (1984): ² Donohew *et al.* (2000).

Comula Tura			-			Vertical stress (MPa)				Shear stress (MPa)								
	Experiment	Material	of test	orientation	pore press (MPa)	Average	Start	Yield	Peak	Reactivatio	Start	Shear	Yield	Peak	Reactivatio			
4		Ka aliaita			(ini 0) 2 17	2.67	2 1 1	2.50	2 66	" 2 70	0.75	216	1 27	1 5 8	1 51			
1	ASR_BIGCCS_07K	Kaolinite			2.17	2.07	2.44	2.50	2.00	2.70	0.73	318	1.37	1.58	1.51			
2	ASR_BIGCCS_08K	Kaolinite			1.05	2.53	2.44	2.50	2.07	2.09	0.72	303	1.35	1.71	1.55			
3	ASR_BIGCCS_09K	Kaolinite	ater		2.24	5 12	2.43	2.51	5 1/	5 16	1.21	302	2.44	2.02	2.07			
4	ASR_BIGCCS_TUK	Kaolinite	N L		2.34	3.88	4.77	4.04	3 90	3.10	0.98	396	2.44	2 / 1	2 30			
5	ASR_BIGCCS_TIK	Kaolinite	witl		2.43	6 35	5.97	5.07	6.38	6./1	1 53	/31	2.00	2.41 / 12	2.55			
6	ASR_BIGCCS_12K	Kaolinite	ion	200	2.51	3.86	3.52	3.33	2.90	2 80	1.55	431	1.05	4.12	2.91			
/	ASR_BIGCCS_13K	Kaolinite	ivat	30	2.30	2.65	2.02	2.70	2.67	2.65	0.98	236	1.97	1.52	1.50			
8	ASR_BIGCCS_14BC	Ball Clay	t reacti		1.27	2.05	2.40	2.47	2.07	2.00	1 13	230	2.06	2.28	2.26			
10	ASR_BIGCCS_15BC	Ball Clay		lt re		1.17	5.05	1.82	1.88	5.07	5.07	1.15	403	2.00	2.20	2.20		
10	ASR_BIGCCS_TOBC	Ball Clay	Fau		2.74	6.27	6.00	6.08	6.30	6.30	2.01	405	2.55	3.60	2.50			
11	ASR_BIGCCS_17BC	Ball Clay					1 10	5.04	1 93	1.88	5.08	5.08	1 71	401	2.55	2 80	2.27	
12	ASR_BIGCUS_18BC	Ball Clay			2 75	6.20	6.04	4.00	6.27	6.25	2.71	1/0	2.30	2.09	2.00			
13	ASR_BIGCCS_19BC	Ball Clay			2.75	0.20 E 24	0.04 E 02	0.08	6.26	0.25	2.30	149	2.02	2.90	2.95			
14	ASR_BigCCS_20K	Kaolinite	#1	30°	/	5.34	5.92	6.01	0.20	/	1.53	453	3.19	3.94	/			
15	ASR_BigCCS_21K	Kaolinite	tion 2#	tion 2#	#2	#2		/	6.17	5.93	6.04	6.31	/	1.51	489	3.46	3.96	/
16	ASR_BigCCS_22Kg	Kaolinite						/	4.99	4.72	4.84	5.10	/	1.33	399	2.36	3.07	/
17	ASR_BigCCS_23Kg	Kaolinite				1.65	2.57	2.41	2.45	2.61	2.60	0.65	318	1.33	1.58	1.56		
18	ASR_BigCCS_24Kg	Kaolinite	tiva gas		/	3.76	3.60	3.67	3.78	/	0.96	386	1.93	2.36	/			
19	ASR_BigCCS_25Kg	Kaolinite	reac /ith	30°	/	6.21	5.92	6.05	6.29	/	1.04	905	3.26	3.98	/			
20	ASR_BigCCS_26Kg	Kaolinite	ult i v		/	2.58	2.38	2.46	2.62	/	0.64	316	1.28	1.58	/			
21	ASR_BigCCS_27Kg	Kaolinite	Fa		/	1.13	1.00	1.07	1.15	/	0.44	149	0.59	0.68	/			
22	ASR_BigCCS_28Kg	Kaolinite			/	3.82	3.57	3.66	3.89	/	1.32	283	1.77	2.22	/			
23	ASR_BigCCS_29Ksh	Kaolinite			/	6.16	5.96	6.08	6.27	/	1.61	333	3.30	3.89	/			
24	ASR_BigCCS_30Ksh	Kaolinite	ess history tests		/	6.19	5.96	6.06	6.31	/	1.53	445	3.32	3.88	/			
25	ASR_BigCCS_31Ksh	Kaolinite		30°	/	6.17	5.95	6.07	6.29	/	1.58	431	3.21	3.87	/			
26	ASR_BigCCS_32Ksh	Kaolinite		50	/	6.19	5.95	6.07	6.27	/	1.56	428	3.21	3.93	/			
27	ASR_BigCCS_33Ksh	Kaolinite	Str		/	3.55	3.55	3.55	3.54	/	0.78	436	0.78	0.78	/			
28	ASR_BigCCS_34Ksh	Kaolinite			/	6.21	5.93	6.06	6.30	/	1.57	445	3.14	3.91	/			

- **Table 2** List of all experiments undertaken as part of the current study. #1 = stress history test, mechanical data only reported here; #2 = flow
- test, only mechanical test reported here.

Deletienskin	Starting shear stress			Yield shear stress			Peak shear stress			Reactivation pressure		
Relationship	Slope	Intercept	R ²	Slope	Intercept	R ²	Slope	Intercept	R ²	Slope	Intercept	R ²
Kaolinite ¹	0.30	/	0.84	0.61	/	0.99	0.72	/	0.99	0.63	/	0.39
Kaolinite ²	0.25	0.24	0.88	0.61	0.00	0.99	0.74	-0.11	0.99	0.37	1.14	0.91
Ball Clay ¹	0.41	/	0.93	0.55	/	0.90	0.63	/	0.88	0.36	/	0.56
Ball Clay ²	0.44	-0.13	0.93	0.46	0.45	0.93	0.56	0.38	0.90	0.38	-0.09	0.56

Table 3 – Relationship between vertical and shear stress for kaolinite and Ball Clay gouge. Note condition (1) has the intercept set as 0, whereas

829 condition (2) does not.

Parameter		Kaolinite ¹	Kaolinite ²	Ball Clay ¹	Ball Clay ²	Average ¹	Average ²
Coefficient of friction	μ	0.717	0.738	0.634	0.561	0.697	0.706
Cohesion (MPa)	С	/	(-0.09)	/	0.33	/	(-0.4)
\mathbf{R}^2		0.99	0.99	0.88	0.90	0.96	0.96
Angle of internal friction	ϕ	35.6	36.4	32.4	29.2	34.9	35.2
Fault angle	θ	27.2	26.8	28.8	30.4	27.6	27.4

Table 4 – Shear properties of the test gouge. Note condition (1) has the intercept set as 0,

831 whereas condition (2) does not. Linear regression has resulted in two tests showing a negative

cohesion, these are shown in parenthesis as cohesion should not be less than zero for these

833 experiments.

Test	Gas entry pressure (MPa)	Maximum gas pressure (MPa)	Reactivation pressure (MPa)
ASR_BigCCS_22Kg	/	5.35	
ASR_BigCCS_23Kg	2.40	5.58	4.71
ASR_BigCCS_24Kg	2.26	5.58	
ASR_BigCCS_25Kg	2.27	5.66	
ASR_BigCCS_26Kg	2.29	5.57	
ASR_BigCCS_27Kg	2.19	5.80	
ASR_BigCCS_28Kg	2.39	5.54	
Average	2.30	5.58	4.71

Table 5 – Gas testing properties.