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1 **Influence of water on deformation and failure of gypsum rock**

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9 **Abstract**

10 While water is known to significantly reduce the strength of rocks, there remains a paucity of data on
11 water-weakening of gypsum. Here, we quantify water-weakening in a natural gypsum facies from
12 Monferrato (Italy) by performing experiments on nominally dry, oil-saturated, and water-saturated
13 samples. Uniaxial and conventional triaxial experiments revealed significant water-weakening in
14 Monferrato gypsum as well as a strong strain-rate dependence of uniaxial compressive strength.
15 Moreover, uniaxial creep tests showed significant time-dependent deformation in samples saturated
16 with sulphate over-saturated water, but not in dry and oil-saturated samples. The creep
17 phenomenology is similar to that observed in other rock types and is consistent with stress-corrosion
18 microcracking, which is supported by our microstructural observations. However, we systematically
19 recorded more inelastic strain in samples deformed at low strain-rates suggesting that additional
20 mechanisms were also active. Comparing our new data on short-term strength with published results
21 for other rock types, we conclude that, when saturated with water in equilibrium with the rock,
22 weakening in gypsum is not notably higher than in other rocks and is partially due to a reduction of
23 fracture toughness in the presence of water.

24

25 **Keywords**

26 Branching selenite gypsum, mechanical strength, creep, microcracking, kinking, dissolution

27

28 **1. Introduction**

29 Gypsum is an evaporitic mineral that plays an important role in several areas of structural geology
30 and civil engineering. It is involved in orogenic tectonics, influences basin dynamics, and is
31 associated with many economic activities including oil exploration, mining, and waste repositories
32 (e.g. Heard & Ruby 1986; de Meer and Spiers, 1999; Cristallini & Ramos 2000; Zucali et al., 2010,

33 Liang et al., 2012). In particular, underground mining excavations in gypsum have often resulted in
34 roof collapses, pillar failures, water intrusions, and generation of surface subsidence, especially after
35 unexpected water circulation and in abandoned or old underground sites (e.g. Bonetto et al. 2008;
36 Wang et al., 2008; Sadeghiamirshahidi and Vitton 2019).

37 From a crystallographic point of view, gypsum is a layered mineral, with pairs of adjacent sheets of
38 Ca^{2+} and $(\text{SO}_4)^{2-}$ tetrahedra separated by double-sheets of water molecules (Figure 1a), resulting in a
39 pervasive cleavage in gypsum crystals (Figure 1b).

40 **Figure 1**

41 This anisotropy at the crystal scale controls some of the principal physical features of gypsum,
42 including the mineral rheology, as first suggested by Craker and Schiller (1962). In that study, the
43 authors experimentally tested the deformation of a single gypsum crystal with a three-point loading
44 system, showing that, when the applied stress is perpendicular to the mineral cleavage (010 plane)
45 the crystal bends and significant plastic deformation may be observed even at relatively fast strain
46 rates. When, on the other hand, the stress is applied parallel to the cleavage, the crystal fractures
47 before any detectable bending occurs.

48 At the rock scale, the variability of gypsum facies all over the world (with differences in grain size,
49 porosity, gypsum content and rock structure) results in a broad range of values of mechanical strength,
50 both under uniaxial and triaxial loading conditions (e.g. Papadopoulos et al., 1994; Yilmaz, 2007;
51 Caselle et al., 2019a-b). Under triaxial loading conditions, gypsum may exhibit micro-plasticity
52 resulting from grain kinking (Brantut et al. 2011, Caselle et al., 2020a-b).

53 The solubility of gypsum in water is 0.015 mol/kg H_2O , which is significantly higher than for several
54 other minerals. As such, the mechanical properties of gypsum are very sensitive to the presence of
55 water, which may cause important weathering and weakening effects. Auvray et al. (2004) observed
56 that the external portions of gypsum pillars in an abandoned underground mine, being more exposed
57 to the humid atmosphere of the drifts, showed evidence of dissolution and corrosion using scanning
58 electron microscopy in secondary electron acquisition mode (SEM-SE). In agreement with these
59 observations, they measured significant changes in material properties (e.g. increase in porosity and
60 decreases in seismic wave velocity and mechanical strength) from the core to the external surface of
61 the pillars. In terms of uniaxial compressive strength, Yilmaz (2010) measured a weakening of about
62 50% in water-saturated gypsum samples compared to dry samples. The decrease of mechanical
63 properties (strength, elastic moduli) was also observed by Castellanza et al. (2008) and Castellanza
64 et al. (2010), who reported on the stability assessment of an abandoned underground gypsum quarry,

65 proposing an evaluation of pillar stability based on the deteriorating effect of water. The experimental
66 investigation by Liang et al. (2012) introduced an additional element, considering the effect of
67 temperature and NaCl in the soaking brine. Their results suggest a positive correlation between
68 weakening and NaCl concentration and temperature during saturation. Zhu et al. (2019)'s multiscale
69 investigation of the phenomenon suggests that the reduction of mechanical strength results from the
70 hydrolysing and weakening of crystal bonds at microcrack tips and is exacerbated by the continuous
71 increase of immersion time.

72 While there is clear evidence of water-weakening in gypsum, the processes that result in this strength
73 reduction remain unclear. Previous studies on water-weakening in different rock types have
74 concluded that various physical mechanisms could contribute to water-weakening in rocks (Baud et
75 al., 2000, Nicolas et al., 2016, Noel et al., 2021, Geremia et al., 2021): 1) A reduction of the surface
76 energy and of the fracture toughness in the presence of water, sometimes called the Rhebinder effect
77 (see for example Røyne et al., 2011); 2) Stress corrosion effects observed in most rock types (Brantut
78 et al., 2014a-b); 3) intergranular pressure solution (Croizé et al., 2013); 4) Capillary effects due to the
79 presence of water, even in a nominally dry rock (Delage et al., 1996; Risnes et al., 2005); and 5)
80 Dissolution at the grain surfaces (Ciantia et al., 2015). Additionally, the mechanical effect of water
81 could result in complex effective pressure behavior, as observed for example in clayey sandstone and
82 dual porosity carbonates (Meng et al., 2020).

83 The microstructures produced in gypsum during its dissolution in water have been investigated using
84 SEM (e.g. Yu et al., 2016) and X-ray computed microtomography (CT, e.g. Meng et al., 2018). Yu
85 et al. (2016), in particular, described a process of crystal splitting along cleavage during dissolution,
86 in accordance with experimental evidence that dissolution on (010) crystallographic faces occurs at a
87 higher rate than in other crystallographic directions (Fan and Teng, 2007).

88 Water has also been observed to influence the creep behaviour of gypsum. De Meer and Spiers (1995,
89 1997, 1999) measured a clear difference in creep behaviour of gypsum powder samples: dry and oil
90 saturated samples exhibited little to no creep, while under water saturated conditions, samples
91 exhibited significant creep. Based on their experimental results, the authors proposed a creep model
92 driven by pressure solution but also suggested that precipitation of gypsum on the pore walls acts as
93 a rate-limiting mechanism. More recently, Hoxha et al. (2005, 2006) measured a clear dependence of
94 creep strain rate of gypsum rock samples on relative humidity. Their experimental results
95 demonstrated strong time-dependent and humidity-dependent dilatancy, that the authors considered
96 as an indication of a damage-like mechanism. Following the authors' interpretation, this mechanism
97 would be unrelated to the growth of new cracks and would consist in the creation of a water layer

98 along the crystal contacts that would ease the sliding between one crystal and another. According to
99 the authors, these water layers would be created by water molecules from both the humid atmosphere
100 and the crystalline structure of gypsum that, when the material is under stress, migrate from their sites
101 to the pores of the rock. However, this complex mechanism involves gypsum dehydration that is
102 difficult to obtain under the experimental conditions investigated by Hoxha et al. (2005; 2006). A
103 clear explanation of the micro-mechanical mechanisms involved in creep in gypsum rock is therefore
104 still needed.

105 The impact of water on the mechanical strength and physical properties of gypsum rock is particularly
106 relevant to underground excavation (Ramon et al., 2021). Gypsum quarries are often located below
107 the static level of the groundwater table and thus require continuous water pumping to permit
108 excavation of drifts. The end of quarry activity, coinciding with the interruption of de-watering
109 operations and the re-establishment of the original water level, results in the re-saturation of the
110 gypsum body. Under these conditions, the water fills the connected porosity of the rock, influencing
111 both short-term and long-term stability of the underground quarries. For these reasons, the current
112 study aims to analyse the micromechanical mechanisms that control the changes in mechanical
113 response of gypsum rock in the presence of water. We use a natural gypsum rock facies (i.e. branching
114 selenite) that is exploited in underground environments in several areas of the Mediterranean basin.
115 Our experimental investigation also aims to quantify the specific effect of water-gypsum chemical
116 interactions, comparing the results obtained by saturating the material with water and with a non-
117 reactive oil. Hence, mechanical tests were performed under dry, oil-saturated, and water-saturated
118 conditions. Our investigation includes uniaxial compression tests, uniaxial creep tests, and
119 conventional triaxial experiments. Microstructural analysis of deformed samples was performed to
120 describe the mechanisms involved in gypsum deformation and failure.

121 **2. Tested material and sample preparation**

122 All our tests were performed on a Miocene microcrystalline gypsum in branching selenite facies
123 (sensu Lugli et al., 2010). Samples were cored in the Monferrato domain of the Tertiary Piedmont
124 Basin (TPB), a complex sedimentary basin located on the inner side of the SW Alps arc that occupied
125 large areas of Piedmont (NW Italy) from the Upper Eocene to the end of the Miocene (Clari et al.,
126 1995; Piana and Polino, 1995; Dela Pierre et al., 2011). The sediments of the TPB stratigraphically
127 overlie a complex tectonic wedge of Alpine, Ligurian and Adria basement units juxtaposed in
128 response to the collision between the European and Adria plates (e.g. Rossi et al 2009). The Cenozoic
129 sediments are presently exposed in the southern (Langhe, Alto Monferrato and Borbera Grue
130 domains) and the northern (Torino Hill–Monferrato arc) sectors of the TPB. The relationships

131 between the two outcropping belts are masked by the Pliocene to Holocene deposits of the Savigliano
132 and Alessandria basins, but are well imaged by seismic profiles (Bertotti et al 2009). Thick gypsum
133 bodies from the Messinian Salinity Crisis (late Miocene) are observed and described in the
134 Monferrato and Langhe domains (Figure 2a).

135 The stratigraphic succession of the test site comprises deep-sea marls overlaid by a geological unit
136 that includes a thick evaporitic succession that counts 13 gypsum – marl cycles (Dela Pierre et al.,
137 2016). The material tested for the present study was sampled from the fourth cycle (the 6th from the
138 onset of the Messinian Salinity Crisis), that corresponds to the appearance of the so-called “branching
139 selenite facies” (Lugli et al. 2010). This layer, that has a thickness of about 10 m, is recognized in the
140 geological literature as the “Sturani Key Bed” (SKB) (Dela Pierre et al., 2011).

141 At the sample scale, branching selenite facies are organized in nodular aggregates consisting of mm-
142 to cm-sized gypsum crystals (Figure 2d). In the aggregates, the gypsum crystals are closely
143 interlocked, creating a dense structure; their habit is usually prismatic and several of them show
144 twinning. Most of the crystals show the presence of both fluid and solid inclusions (Natalicchio et al.
145 2021, Cipriani et al. 2021). These nodules are immersed in a fine matrix organized in laminae
146 composed of gypsum, calcite and terrigenous minerals (mainly clays, quartz and feldspars). A single
147 dominant orientation of the elongated gypsum crystals in nodules is apparent (Caselle et al. 2020c –
148 Figure 2c). The gypsum content of this rock generally ranges between 85 and 95 wt% (Caselle et al.
149 2019a).

150 **Figure 2**

151 *2.1 Sampling and porosity determination*

152 Mechanical tests were performed on 77 cylindrical samples (height: 40 mm; diameter: 20 mm –
153 Figure 2b), re-sampled from two bigger 80 mm in diameter pieces of borehole core. All samples were
154 cored in the vertical (i.e. borehole-parallel) direction from both core pieces. Both core pieces are from
155 the same axial borehole, drilled as part of a survey campaign included in the exploitation plane of an
156 underground quarry. The vertical borehole is oriented perpendicular to the sub-horizontal
157 stratification and main sedimentary discontinuities; the borehole crosses the branching selenite layer
158 between 77 m to 87 m of depth. The two core pieces used in this study were drilled between 82.4 to
159 82.9 m (Core 1) and 78.0 to 78.7 m (Core 2) depth.

160 The porosity of all 40mm by 20mm samples was measured using a helium pycnometer, following
161 EN ISO 17892-3:2016 recommendations. Measured values of porosity range between 0.034 and
162 0.098, solid density ranges between 2.33 and 2.43 g/cm³ and bulk density is between 2.15 and 2.27

163 g/cm³. The values of porosity and bulk density are shown in Figure 3. Density decreases linearly with
164 the porosity suggesting that the solid density was about the same in the studied cores, and therefore
165 that the mineralogical composition of all our samples was comparable. In detail, a comparison of
166 these measurements with the theoretical curve of pure gypsum (solid density $\rho_s = 2.4 \text{ g/cm}^3$)
167 confirmed the presence of other minerals beside gypsum in the studied rock, as well as the lower
168 density of Core 1 with respect to Core 2. This is supported by the X-ray powder diffraction (XRPD)
169 analysis of non-gypsum portions of Core 1 and Core 2. Since the XRPD analyses were specifically
170 performed to characterize non-gypsum mineralogical content, the material was ground using a mortar
171 and pestle and dissolved in water, ensuring that the solid/water ratio reached a concentration of
172 calcium sulfate lower than gypsum solubility (2 g/l at 20°C). After 24 hours, the solution was filtered
173 and the residual solid portion was smeared on glass slides. This sample preparation method allowed
174 XRPD characterization of the insoluble minerals without the interference of the gypsum diffraction
175 pattern. Analyses were performed using a Miniflex 600 diffractometer, with lambda value of 1.54
176 angstrom and 2-theta ranging from 0 to 50 (complete procedure and data may be found in Caselle et
177 al, 2022). Results (Figure 3b) show that only calcite is present in Core 2 (blue line), while Core 1 (red
178 line) also contains clay minerals, calcite and minor quartz and feldspars, consistent with the higher
179 density of Core 2 with respect to Core 1 (complete procedure and data may be found in Caselle et al,
180 2022).

181 Gas permeability was measured on three samples, selected on the basis of the connected porosity
182 (maximum, minimum and mean values in the investigated dataset), using the pulse decay method
183 detailed by Heap et al. (2017). Results show that permeability ranged between $2 \cdot 10^{-17} \text{ m}^2$ and less
184 than 10^{-19} m^2 .

185 **Figure 3**

186 *2.2 Water saturation*

187 Due to the high solubility of gypsum, it was important to ensure that our water-saturated samples
188 were in equilibrium with the saturating fluid over long periods of time, in particular during the
189 mechanical tests performed at low strain rates and under creep conditions. To do this, the saturation
190 process was performed with a gypsum-water solution in chemical equilibrium with the material. To
191 create this gypsum-saturated water solution, we placed gypsum powder and rock offcuts in distilled
192 water. Dissolution was monitored by measuring the electrical conductivity of the solution over time;
193 after 10 days, a stable value of 1.8 mS/cm was obtained. This solution, *a priori* at equilibrium, was
194 then used to saturate the samples. Samples were vacuum-saturated and then left in the solution for a
195 period of at least two weeks. The conductivity, monitored throughout the saturation period, remained
196 stable at 1.8 mS/cm.

197 *2.3 Oil saturation*

198 Our experimental program also included compression of oil-saturated samples, aiming to examine
199 the inert reaction between gypsum and an apolar fluid. Despite the absence of a dissolution process,
200 the saturation with dearomatized oil produced an intense red discolouration on the surface of the
201 samples (Figure 4a). This red discolouration was particularly concentrated along the fine-grained
202 layers of the branching selenite structure and persisted even after the samples were allowed to
203 desaturate under ambient laboratory conditions (i.e. dry out), as shown in Figure 4b (after a period of
204 about one month). In order to identify the source of the red discolouration, one of these oil-saturated
205 samples was analysed using SEM energy dispersive X-ray spectroscopy (EDS). The resulting
206 compositional maps (obtained on one of the circular faces of the sample) show the classical features
207 of branching selenite facies, with 1 to 2 mm sulphate crystals (in Figure 4c, gypsum in green and
208 celestine in orange), carbonate minerals of about 0.1 mm (in blue) and a finer, siliceous matrix (in
209 red on the map in Figure 4c). The areas of red discolouration in the macroscopic samples correspond
210 to the siliceous matrix. Point EDS analyses showed the presence of Si, Al, Mg, Na and minor K and
211 Fe (Figure 4d) that indicate the presence of multi-layered clay minerals (mainly illite and smectite).
212 This suggests that the red discolouration results from the absorption of dearomatized oil in the
213 swelling layers of the clay minerals. To confirm if the dearomatized oil was absorbed by the clay
214 minerals, an oil-saturated sample was heated to 350°C (i.e. the decomposition temperature of
215 smectite). Following heat treatment, the red portions of the sample turned to black, confirming an
216 expulsion of the red/dearomatized oil due to the collapse of the clay structure (Figure 4e).

217 **Figure 4**

218 **3. Experimental procedures**

219 *3.1 Uniaxial compression*

220 23 uniaxial compression tests were performed at the Institut Terre et Environnement de Strasbourg
221 (ITES; the Strasbourg Institute for the Earth and the Environment), using a servo-controlled uniaxial
222 press. Axial displacement was measured throughout the test using an LVDT with an accuracy of
223 $\pm 0.15 \mu\text{m}$, while the axial force was measured with an accuracy of $\pm 9 \text{ N}$ (corresponding to an accuracy
224 of the applied stress of $\pm 0.03 \text{ MPa}$). Details about this set-up can be found in Heap et al. (2014). All
225 the tests were performed up to axial strains of about 2%. This limit was chosen because we observed
226 that our samples had reached stable post-peak conditions at these strains. Of the 23 total tests we
227 carried out, 5 tests dedicated to microstructural analyses were stopped just after peak stress, at an
228 axial strain of 0.8%. Constant strain rates between 10^{-4} and 10^{-8} s^{-1} were used in order to investigate
229 the strain-rate dependence of the material response (see Table 1 for details on the applied strain rates).

230 The Young's modulus for all the tests was calculated as the slope of the elastic portion of the stress-
231 strain curve (i.e. the tangent modulus).

232 In addition to the uniaxial compressive strength tests described above, 14 uniaxial creep tests were
233 performed using the same servo-controlled apparatus. In these experiments, samples were first loaded
234 at a constant strain rate of $10^{-5}/s$. After stopping the test for a few seconds, the experiment was
235 continued by controlling for load and, and thus, a constant level of stress (creep conditions) was
236 imposed on the sample (see Heap et al., 2009a-b for details). Because of the variability between
237 samples, the creep stress level was chosen based on the shape of the stress-strain curve beyond the
238 elastic regime. As illustrated in Figure 5, we performed both conventional creep tests and step creep
239 tests, in which the stress level was increased during the experiments by small steps of 1 MPa until
240 sample failure. Specifically, 9 conventional creep and 5 step creep tests were performed. The error of
241 the measured strain rates for both conventional creep and step tests was estimated to be less than 10%.

242

Figure 5

243 As previously mentioned, all the tests were performed either under nominally dry or water/oil
244 saturated conditions. For dry conditions, the samples were dried under vacuum at a temperature of
245 $40^{\circ}C$ for a minimum of 48h before the tests. Saturated tests (both oil-saturated and water-saturated)
246 were performed after a saturation period of at least two weeks. Tests were performed on samples
247 immersed in the saturating fluid to ensure that samples did not dry out over the course of the
248 experiments (see Heap et al., 2018b for details).

249 In the discussion, we supplement the above-described laboratory program with some additional tests
250 that have been performed at the purpose to evaluate volumetric evolution during tests.

251 *3.2 Triaxial compression*

252 9 conventional triaxial tests were conducted at room temperature in the servo-controlled triaxial
253 apparatus at ITES, which can reach a maximum confining pressure of 200 MPa. Confining pressure
254 was regulated by a computer-controlled servo motor connected to a pressure transducer with an
255 accuracy of 0.05 MPa. Axial load was applied by a piston and regulated by a second computer-
256 controlled servo motor. Axial displacement was measured with an accuracy of $0.2 \mu m$ outside the
257 pressure vessel, with a capacitive transducer mounted on the moving piston, which was servo-
258 controlled to advance at a fixed displacement rate (corresponding to a nominal strain rate of $10^{-5}/s$).
259 Details about the set-up and experimental procedure can be found in Baud et al. (2015). Volumetric
260 strain was recorded by monitoring the piston displacement of the confining pressure generator with

261 an angular encoder. This methodology was previously used for porous limestone by Baud et al.
262 (2009).

263 The gypsum over-saturated water solution was unsuitable for use in the pore pressure circuit of the
264 triaxial press due to the risk of gypsum precipitation in the pipes. Therefore, to study the influence of
265 water on gypsum under triaxial conditions, we deformed partially saturated samples under undrained
266 conditions, following a procedure recently used by Pijenburg et al. (2019) on sandstone. Samples
267 were initially saturated with the gypsum-water solution. Then, before the tests, the to-be-tested
268 sample was removed from the solution and allowed to partially de-saturate. The weight of the sample
269 was constantly monitored until 25% of the pore fluid had evaporated. At that moment (with a water
270 saturation of 75%), the sample was mounted into the triaxial press. A specific set of uniaxial tests
271 confirmed the general correspondence of the mechanical data for 100% and 75% water saturation.
272 This is consistent with the study of Schmitt et al. (1994) who did not observe significant changes in
273 the Uniaxial Compression Strength (UCS) of sandstone for water saturation between 100% and 20%.
274 Since our experiments were performed to low levels of volumetric strain (typically <1%), no
275 significant over-pressure could develop in the undrained samples.

276 *3.3 Microstructural analyses*

277 Among the previously described mechanical tests, 12 dedicated tests (5 under uniaxial compression,
278 4 under creep conditions and 3 under triaxial compression) were performed for microstructural
279 analysis. For these, the experiments were stopped before the complete failure of the sample, slightly
280 after the peak stress. Stress-induced damage in the samples was then analysed on longitudinal thin
281 sections, prepared after saturation with epoxy. Carbon-coated thin sections were investigated with a
282 Cambridge S-360 scanning electron microscope at the University of Turin. Backscattered electron
283 images (BSE) were obtained at an accelerating voltage of 15 kV. Standard petrographic images were
284 also acquired with an Olympus BX4 reflected and transmitted polarized light optical microscope with
285 JENOPTIK ProgResC5 digital colour camera.

286

287 **4. Results**

288 The experimental dataset consists of 46 mechanical tests (including the 12 dedicated to
289 microstructural analysis): 23 uniaxial loading tests, 14 uniaxial creep tests (9 conventional creep and
290 5 step creep tests), and 9 conventional triaxial tests. The test conditions and results are reported in the
291 supplementary material and summarised in Table 1, Table 2, and Table 3 respectively.

292

293

Table 1

294

295 *4.1 Uniaxial loading mechanical tests*

296 Figure 6a shows some representative stress-strain curves for dry, water-saturated, and oil-saturated
297 samples deformed uniaxially at a strain rate of 10^{-5} s^{-1} . Some variability is typically observed in the
298 uniaxial compressive strength (UCS) of branching selenite gypsum, but water-weakening is clear in
299 Figure 6a. We observed an average reduction of the UCS of about 50%, from 25 MPa under dry
300 conditions to 12 MPa under water-saturated conditions. The Young's modulus was also significantly
301 smaller in the presence of water, with an average reduction of 50%, from 10 GPa for dry samples to
302 5 GPa for wet samples. The strength of the oil-saturated samples was intermediate between these end-
303 members, with mean UCSs of 20 MPa and Young's moduli of 8.5 GPa. In all tests, the peak stress
304 occurred approximately at 0.4% axial strain.

305 We also noted two important differences in the post-peak part of the stress-strain curves between dry
306 and water-saturated samples. Under dry conditions, strain-softening was significantly greater than for
307 water-saturated samples, and was also marked by frequent stress drops with amplitudes ranging from
308 1 to 5 MPa. For water-saturated samples, few to no stress drops were observed and the post peak
309 behaviour was more continuous, with limited softening. The mechanical data for oil-saturated
310 samples were similar to the dry data, showing significant strain-softening characterised by the
311 presence of stress-drops between 1 and 2.5 MPa in amplitude.

312 All UCS values of dry, oil-saturated, and water-saturated tests are summarised in Figure 6b, as a
313 function of porosity. Water saturated samples show a good inverse linear relationship between UCS
314 and porosity, while dry and oil-saturated samples are more scattered. Despite the scatter in the dry
315 data, we observe a clear strength difference between dry and water-saturated samples, with water-
316 saturated samples being weaker. For a given value of porosity, oil-saturated values are lower than dry
317 but higher than water-saturated values.

318

Figure 6

319 The effect of strain rate for oil-saturated and water-saturated uniaxial tests is shown in Figure 7 for
320 strain rates between 10^{-4} and 10^{-8} s^{-1} . For oil-saturated samples (Figure 7a), the change in strain rate
321 did not result in significant variations in deformation and failure behaviour: UCS range, softening
322 behaviour and stress-drops in the post peak remained mostly unchanged in all the curves. We assume
323 that similar results could be obtained under dry conditions; we have, however, been unable to verify
324 this due to the difficulty of maintaining stable dry conditions over the experimental timescale (i.e. we
325 cannot control for sample humidity under ambient laboratory conditions).

326 On the other hand, decreasing strain rate had a significant influence on the results for water-saturated
327 tests (Figure 7b). In particular, the UCS decreased with decreasing strain rate and the post peak
328 behavior changed from strain softening (typical of brittle behavior) to more ductile behavior. A
329 systematic decrease of Young's modulus with decreasing strain rate is also highlighted in Figure 7b.

330 **Figure 7**

331 *4.2 Uniaxial creep tests*

332 Table 2 summarises the results of our uniaxial creep tests (conventional and step tests). The strain-time
333 curves of the conventional creep tests, all performed on water-saturated samples, are shown in Figure
334 8a. They have the typical features of brittle creep behaviour, as observed in most rock types (see
335 Brantut et al., 2013 for a review). They show an initial phase of decreasing strain rate followed by an
336 inverse trend where the strain rate progressively increases towards failure. There is therefore a
337 minimum strain rate around which the creep strain rate remains quasi-constant for a significant
338 amount of time. We observed significant variability in the recorded minimum strain rates for the same
339 value of applied stress. Figure 8b, showing the values of measured strain rate against initial porosity,
340 suggests the existence of a direct relationship between these parameters.

341 **Figure 8**

342 **Table 2**

343
344 Because of the variability between samples, we studied the influence of the saturating fluid on time-
345 dependent deformation with step tests. Results, showed in Figure 9 and summarised in Table 2,
346 highlight the clear difference in the strain rate for dry and oil-saturated samples, compared to water-
347 saturated samples. The dry and oil-saturated samples show basically no creep up to relatively high
348 stresses. Close to the short-term strength, a negligible amount of creep was observed before failure.
349 Most of measured strain occurred in the first few seconds of each step (i.e. immediately after the
350 application of the stress). This absence of creep was still observed in the last step before the failure
351 of the sample (i.e. with an applied stress less than 1 MPa lower than the material strength). Water-
352 saturated samples, on the other hand, showed significant creep in all cases with strain rates larger or
353 equal to 5×10^{-9} /s in the first steps. As expected, the strain-rate quickly reached a constant value for
354 the following steps and increased significantly with the applied stress. After a certain amount of strain,
355 the strain rate did not stabilize but accelerated exponentially before the failure of the sample. The
356 maximum strain rate recorded before this last stage of the step tests was 5×10^{-7} /s.

357 **Figure 9**

358 Figure 10 shows the evolution of the strain rate with the applied stress for all our step tests. We found
359 very similar trends (with some offset due to sample variability) for the three water-saturated samples,
360 in agreement with what has been observed in similar tests performed on sandstone (Brantut et al.,
361 2013). Taken together, these data show that the strain rate evolution is the same for all samples,
362 despite the material variability. In all cases, an increase of 5 MPa of the applied stress resulted in an
363 increase of the creep strain of two orders of magnitude. As explained above, no significant creep was
364 observed in oil-saturated and dry samples. For a large interval of stresses, creep occurred at strain
365 rates below the resolution of our system ($\approx 10^{-10}/s$). Then, the maximum strain rate recorded before
366 failure of a dry sample was about 1/4 of the minimum strain rate observed in water-saturated samples.

367 **Figure 10**

368 *4.3 Triaxial experiments*

369 Figure 11 shows the mechanical data from the triaxial experiments performed on dry and partially
370 saturated samples. The results are also summarised in Table 3.

371 **Table 3**

372 Experiments on dry samples were performed at confining pressures ranging from 10 to 100 MPa
373 (Figures 11a – black curves). At a confining pressure $P_c=10$ MPa, the behaviour was typical of brittle
374 deformation with a peak stress followed by strain softening. At all tested confining pressures above
375 10 MPa, significant strain hardening was observed (Figure 11a – black curves). In addition, the stress
376 strain curves were punctured in most cases by small stress-drops, similar to the ones observed in
377 uniaxial compression. Our volumetric strain data revealed dilatancy at all tested pressures up to 100
378 MPa.

379 In presence of water (Figure 11a – blue curves), significant weakening is clear at all tested pressures.
380 We noted the absence of softening even at low confining pressures. Between 10 and 80 MPa, we also
381 observed dilatancy (Figure 11b – blue curves). Consistent with our uniaxial data, the presence of
382 water also causes the stress drops to disappear under triaxial conditions.

383 Figure 11c summarises the results of the triaxial tests in the stress space. As suggested by Brantut et
384 al. (2011), we quantified the yield point using the values of two critical stresses (i.e. the onset
385 dilatancy and the first stress-drop in the dry data). These points are both easy to identify and clear
386 indicators of inelastic behaviour. The values, also reported in Table 3, were found to be similar, except
387 at the highest tested pressure of 100 MPa. The failure envelopes in dry and wet conditions were

388 mostly parallel, indicating that water-weakening was of the same magnitude in the tested pressure
389 range.

390 **Figure 11**

391

392 **5. Microstructural observations**

393 *5.1 Uniaxial compression*

394 Figure 12 summarises our microstructural observations on samples deformed in uniaxial compression
395 under dry conditions. The interruption of the test just after the peak (with an axial strain of 0.8%)
396 preserved a pattern of coalescing cracks. The main cracks show an aperture of a few tens of μm and
397 sharp borders (Figure 12a-c), occasionally cut through the gypsum grains and are often rimmed by
398 fine-grained material (Figure 12c). Some of these cracks are aligned along a plane with an angle of
399 30° with respect to the applied stress (Figure 12b), while others follow the sub-horizontal layering of
400 the branching selenite (Figure 12d). Since at 0.8% of axial strain the process of failure coalescence is
401 not completed, the cracks show interruptions that correspond to changes in the textural features of the
402 rock (e.g. presence of a layer of finer material – Figure 12a-c). These discontinuities in the failure
403 plane suggest a coalescence “by steps”, consistent with the stress-drops in the mechanical data.

404

Figure 12

405 In contrast to dry samples, the microstructure of samples deformed under water-saturated conditions
406 (Figure 13) showed the presence of intra-crystalline microcracks, that usually consist of straight
407 parallel fractures that follow the mineral cleavage (Figure 13a-b).

408 Despite this general feature, common to microstructures of all wet samples, some differences may be
409 observed between samples deformed “quickly” (i.e. strain rate = $10^{-5}/\text{s}$) and “slowly” (i.e. strain rate
410 = $10^{-7}/\text{s}$ or lower or creep conditions). In the former, the majority of cracks are concentrated along a
411 diagonal band of deformation, suggesting a process of coalescence towards a coherent failure plane.
412 In the latter, microcracks are more uniformly distributed in the sample without showing any area of
413 preferential concentration, despite the accumulation of significant amounts of strain (about 2% both
414 in the low-strain rate sample shown in Figures 13b and in the creep sample shown in Figure 13c).
415 This can be more easily understood in Figure 14, which shows the SEM images of the entire thin
416 sections of samples deformed “quickly” (Figures 14a and c) and “slowly” (Figures 14b, d, and e).
417 Figures 14a and c (sample deformed “quickly”) show the coalescence of microcracks into the
418 beginnings of a failure surface, while Figures 14b-d-e (sample deformed “slowly”) show that the
419 main cracks are oriented subvertically. In addition, slow strain rate and creep samples show the

420 presence of particular intra-crystalline structures that consist of a series of narrow cracks along the
421 mineral cleavage that, being oriented sub-parallel to the axial applied stress, accommodate the strain
422 by the folding of each separated slice and by the creation of short perpendicular cracks (Figure 13c).
423 The bending of the crystals in creep samples, especially in crystals oriented perpendicular to the
424 applied load, is also evident under the optical microscope. As shown in the Figure 15a-c, some of the
425 larger gypsum crystals in samples tested under creep conditions contain “bands” with a different
426 interference colour than the surrounding portions of the mineral, suggesting a change in the
427 crystallographic orientation. These structures are absent in the initial material (Figure 15d).

428 All samples deformed under water saturated conditions showed evidence of dissolution. For example,
429 Figure 13d shows an intergranular crack with a 10 to 20 μm aperture and rounded edges that we posit
430 was created by the initial effect of mechanical loading, but enlarged by the dissolution of water.

431 **Figure 13**

432 **Figure 14**

433 **Figure 15**

434 *5.2 Triaxial tests*

435 Consistent with our mechanical data, we only observed a clear, coalesced, macroscopic failure plane
436 in the dry sample deformed at a confining pressure of 10 MPa. As in the uniaxially loaded samples,
437 this failure plane was oriented 30° with respect to the direction of the applied stress (axial). In all
438 other experiments, the final samples did not macroscopically show any failure plane (Figure 16).

439 **Figure 16**

440 Despite this, optical and electron microscope observations showed the pervasive presence of brittle
441 deformation features (i.e. microcracks) as well as evident intra-crystalline plastic structures that
442 deform most of the main crystals in the samples. These structures, shown in Figure 17, consist of
443 bands between a few μm to a few tens of μm thick, oriented perpendicular to the elongation of the
444 crystals (i.e. to the mineral cleavage). In optical microscopy, these bands are highlighted by a change
445 in the birefringence colour (Figure 17a) and in BSE-SEM (Figure 17b) and in SE-SEM (Figure 17c)
446 their boundaries are clearly delineated. Based on these observations, these structures were classified
447 as kink bands (i.e. bands created by two parallel folds with straight limbs and pointed hinges) formed
448 by the folding of the (010) mineral cleavage.

449 At the tips of the kink bands, intracrystalline cracks that accommodate the plastic deformation created
450 by kinking are commonly observed within the same crystal or in neighbouring ones (Figure 17d).

451 With the increase of confining pressure, these kink structures appear to be organized in bands (Figure
452 18).

453 We observed that also the microstructure of samples deformed under wet conditions was dominated
454 by the kinking of the grains.

455 **Figure 17**

456 **Figure 18**

457 **6. Discussion**

458 *6.1 Water weakening in gypsum*

459 The experimental results show the important influence of water on the mechanical response of
460 gypsum rock over a wide range of conditions. Under uniaxial compression, water-weakening resulted
461 in a decrease in peak strength and Young's modulus of 50% (Figure 6). Similar water-weakening has
462 been reported previously in different gypsum facies (e.g. Yilmaz, 2010). As an illustration, Figure
463 19a compiles the results of dry and water-saturated uniaxial compression tests on gypsum samples
464 from three different facies: branching selenite gypsum (this study); a massive gypsum facies from the
465 Hafik formation in Sivas basin, Turkey (Yilmaz, 2010); and Volterra gypsum, a very-pure alabastrine
466 gypsum facies with very low porosity and very fine grain-size. To permit direct comparison, we
467 performed a series of uniaxial tests on Volterra gypsum, presented in Annex 5 (supplementary
468 material). Despite the differences in strength (likely related to the differences in microstructural
469 attributes such as texture, porosity, grain size, composition, etc.), an average water-weakening of 52%
470 was obtained in these gypsums and has no clear relationship with porosity.

471 We note that water-weakening is more significant in tests performed in an open circuit with a
472 continuous flow of fresh water, as observed by Castellanza et al., (2008) These authors reported an
473 exponential weakening of up to 75% over 1 week.

474 **Figure 19**

475 Water-weakening is observed in most rock types, including sandstone (Bell, 1978; 1995; Baud et al.,
476 2000; Duda and Renner, 2013; Tang, 2018; Heap et al., 2019), siltstone (Erguler and Ulusay, 2009;
477 Li et al., 2019), mudstone (Erguler and Ulusay, 2009), granite (Zhuang et al., 2020), basalt (Zhu et
478 al., 2016), andesite (Hashiba et al., 2019), limestone (Ciantia et al., 2015; Baud et al., 2016; Nicolas
479 et al., 2016), and tuff (Erguler and Ulusay 2009; Zhu et al., 2011; Heap et al., 2018a). In Figure 19b,
480 we compare water-weakening in gypsum with existing published data on these rock types. We note
481 that in most cases the authors confirm that uniaxial compression tests were performed on saturated
482 samples after some chemical equilibrium was reached, as in this study. Figure 19b shows that water

483 weakening in gypsum is not significantly larger than what has been previously observed in other
484 rocks, including sandstones, limestones, tuffs, andesites and basalts.

485 In sandstone, Baud et al. (2000) used micromechanics to conclude that water-weakening can be
486 explained by a reduction in fracture surface energy and, consequently, of the fracture toughness in
487 the presence of water. In the brittle regime, micromechanical models, such as the pore-emanated
488 crack model (Sammis and Ashby, 1986), predict that the ratio UCS^{wet}/UCS^{dry} is equal to the ratio
489 $K_{IC}^{wet}/K_{IC}^{dry}$, if K_{IC}^{dry} and K_{IC}^{wet} are the fracture toughness in nominally dry and water-saturated
490 samples, respectively. It should be noted that this effect would cause a strength reduction in the
491 presence of water even at high strain rates and also for incomplete saturation, as observed in this
492 study. At ambient temperature, Meng et al. (2015) measured a reduction of K_{IC} in a water-saturated
493 gypsum (a marine facies with gypsum content between 60% and 85%) of about 21%. This suggests
494 that water-weakening observed in our gypsum samples can only be partially explained by the
495 reduction of the fracture surface energy.

496 Other causes commonly suggested to explain water-weakening also involve the dissolution of specific
497 elements of the rock, such as the cement (e.g. depositional bonds among the grains of a calcarenite,
498 Ciantia et al., 2015) or increased solubility of some grains in the rock (e.g. zeolites in volcanic tuffs,
499 Heap et al., 2018a). Our new data suggest a change of failure mechanism in the presence of water,
500 consistent with such processes. Under dry conditions, failure occurs as an unstable stepping
501 mechanism of crack coalescence, as suggested by the episodic stress-drops in the mechanical data
502 (Figure 11a) and by microstructural observations (Figure 12). Under water-saturated conditions,
503 water dissolves material along the grain boundaries and along the surfaces of the mineral cleavage.
504 The observation that cracks propagate along the mineral cleavage (Figure 13) is consistent with the
505 mechanical weakness created by the oriented crystallographic structure of gypsum, that, in the
506 presence of water, is further enhanced by the faster dissolution rate on (010) crystallographic faces
507 (Yu et al., 2016, Fan and Teng, 2007), resulting in a weakening of the grains (see the dense intra-
508 crystalline cracking shown in Figure 13). These processes reduce the overall strength of the rock.
509 This scenario is supported by the disappearance of stress drops in the post-peak stress-strain curves
510 for water-saturated samples (Figure 6a). Indeed, the weakening of grains and intra-grains connections
511 changes the unstable step-wise crack coalescence into a more gradual failure process.

512 *6.2 Time-dependent behaviour*

513 While no significant time-dependent behaviour was observed in samples deformed under dry
514 conditions or saturated with an apolar fluid (i.e. oil), our new data reveal a clear strain rate dependence
515 of strength and significant creep in all samples saturated with gypsum-saturated water. The creep

516 behaviour of branching selenite gypsum shows the following features: All our creep experiments
517 ended by brittle failure of the samples, meaning that we observed brittle creep in all cases. The brittle
518 creep phenomenology in gypsum was similar to that observed in most other rock types (Brantut et
519 al., 2013). Significantly more strain was recorded before failure in samples deformed at low strain
520 rates and samples with higher porosity (Figure 8 and Table 2).

521 To delve deeper into the mechanism(s) leading to this time dependent behaviour, we performed
522 additional, targeted experiments on sample of Monferrato gypsum aimed at quantifying porosity
523 change during creep. Porosity was measured before and after creep experiments that were stopped at
524 the onset of the acceleration towards failure (Figure 20a). Because of the significant impact of
525 porosity on the mechanical behaviour of gypsum, and also of sample availability, we focused on end-
526 members: we deformed two samples with high porosity and one with low porosity. The data show
527 that in the three cases significant dilatancy occurred during creep (the difference in porosity was
528 between +0.4% and +1.3%). This is consistent with the previous results of Hoxha et al. (2006), who
529 also reported dilatancy in triaxial creep experiments on samples of a highly pure orogenic gypsum
530 facies from Jura (France), under conditions of high relative humidity. Comparing these results with
531 similar porosity measurements performed on samples deformed at constant strain rates (Figure 20b),
532 we found an overall similar trend in creep and conventional compression tests. Considering the small
533 differences that may be attributed to the initial porosity, higher amounts of accumulated strain
534 correspond to more dilatancy, with the exception of the sample loaded at a strain rate of 10^{-5} s^{-1} , which
535 has significantly higher volumetric increase. Both this dilatancy and the failure mode of the samples
536 suggest that stress-corrosion cracking was the main micromechanism of time-dependent deformation
537 in the branching selenite gypsum.

538

Figure 20

539 As discussed by Brantut et al. (2014a-b), an important feature of brittle creep driven by stress
540 corrosion is the fact that the typical increase in strain rate towards failure begins when the inelastic
541 strain for a given creep stress reaches the same value as that of a (“fast”) constant strain rate test, as
542 shown for Darley Dale sandstone in Figure 21a. Brantut et al (2014b), however, described a somehow
543 different situation in Purbeck limestone in which they observed significantly more inelastic strain
544 under creep conditions, in particular with low strain rates, than in faster constant strain rate tests
545 (Figure 21b). This was interpreted by the authors to be due to additional mechanisms potentially
546 acting on the top of the dominant action of stress corrosion: plastic flow at microcrack tips (that results
547 in less efficient crack propagation and interaction, producing higher overall strain at failure, since
548 more cracks can be accommodated before coalescence) and pressure solution within the fine-grained

549 matrix (considered as the main mechanism responsible for the compaction observed in the mechanical
550 tests in that study). As shown by Figure 21c, our new data on gypsum suggest a comparable scenario.
551 Indeed, we systematically observe significantly more inelastic strain in all creep tests and tests
552 performed at slow constant strain rates. Figure 21c shows that the difference could in some cases be
553 as high as a factor of 3, suggesting that mechanisms other than stress corrosion must have been active
554 in the material.

555

Figure 21

556 As illustrated in Figure 13, the microstructure of the samples that experienced large amounts of
557 inelastic strain (up to 2%) is dominated by microcracking, with a prevalence of intracrystalline cracks
558 along the mineral cleavage (Figure 13). We also observe evidence of plastic structures (Figure 13c
559 and Figure 15).

565 Overall, our microstructural observations, in agreement with the increase of volume identified by the
566 sample porosities reported in Figure 20, do not support pressure solution as a major mechanism of
567 time dependent deformation in our gypsum. Further, the layered crystallographic structure of gypsum
568 allows for the creation of plastic structures (i.e. Figure 13c and Figure 15) with relative ease. Under
569 creep conditions, the lower crack growth rate may result in a more efficient activation of plastic
570 mechanisms and, therefore, predominantly plastic flow. Consequently, shorter cracks and fewer crack
571 interactions are expected resulting in higher overall strain at failure (Brantut et al., 2014b).

572 Brittle creep has been reported in most rock types, under different stress, temperature and
573 environmental conditions - e.g. in sandstone (Baud and Meredith, 1997; Heap et al., 2009a, 2009b;
574 Ngwenya et al., 2001; Tsai et al., 2008), clay (Gasc-Barbier et al., 2004), tuff (Martin et al., 1997),
575 limestone (Brantut et al., 2013), basalt (Heap et al., 2011), and granite (Fujii et al., 1999). Figure 22
576 shows a comparison of our new data on branching selenite gypsum with uniaxial creep data on a
577 saccharoide gypsum (Moiriat et al., 2006), Darley sandstone (Chen et al., 2018), and Purbeck
578 limestone (unpublished data from Brantut et al., 2014b). It should be noted that both datasets on
579 Darley Dale sandstone and Purbeck limestone were obtained in Strasbourg using the same
580 experimental set-up and procedure (see Section 2). For gypsum, the evolution of strain rate with
581 applied stress is well-described by an exponential or power law as observed in other rock types, again
582 supporting stress-corrosion cracking as the main creep micromechanism. We note that the evolution
583 in our data is consistent with Moirat et al. (2006). These authors studied creep in a saccharoide
584 gypsum with textural and mechanical features similar to the facies investigated in this work (i.e. grain
585 size between 0.1 and 1 mm and wet uniaxial strength of 13.7 MPa). Their creep tests were performed,

586 as in our study, under saturated conditions with gypsum-saturated water, but at significantly lower
587 stresses (not higher than 50% of the UCS). Their creep strain rates were consistently less than 2×10^{-9}
588 $^9/s$ measured over up to 1 year and, as in our study, their associated microstructural observations
589 revealed microcracks, cracking and fragmentation of grains, which is in overall agreement with our
590 observations. As shown in Figure 22, despite the difference in applied stress and duration of the
591 measurements, the trend suggested by our data is compatible with the order of magnitude of strain
592 rate obtained by Moirat et al., (2016), which was consistently less than $2 \times 10^{-9}/s$ measured over up
593 to 1 year.

594 The comparison between gypsum and other rocks in Figure 22 shows that the creep strain rate in
595 gypsum was significantly less sensitive to the applied stress than in Darley Dale sandstone and
596 Purbeck limestone, as confirmed by the exponent's constant (about 14 in gypsum and 30 in the other
597 rocks). This can be interpreted either as a lower stress corrosion index for gypsum or due to the
598 complex influence of several mechanisms, though the latter is unlikely based on our observations.
599 More systematic creep experiments under confinement would be needed to provide definitive
600 conclusions and this should be a target for future studies. From a geotechnical point of view, the data
601 presented in Figure 22 suggest that the long-term evolution of gypsum's mechanical behaviour could
602 be easier to predict than in other rock types. This is because, according to data in Figure 10 and in
603 Figure 22, the strain rates observed in gypsum are less sensitive to the applied stress.

604 **Figure 22**

605 *6.3 Impact of effective pressure*

606 Our triaxial data on branching selenite gypsum show a classical increase of the strength with
607 confining pressure under both dry and wet conditions without any major macroscopic change in
608 failure mechanism between confining pressures of 20 and 100 MPa (Figure 16). The water weakening
609 under triaxial conditions was consistent with our uniaxial data, analysed in section 6.1. Focusing on
610 the dry data, we note the very strong similarities between our mechanical data and those from the
611 earlier study of Brantut et al. (2011) on Volterra gypsum. For both rocks, clear strain softening was
612 only observed up to a confinement of 10 MPa and associated with a major shear band. At higher
613 pressures, deformation was localized along multiple shear bands (see Figure 18) and small stress
614 drops of comparable amplitudes were observed. In addition, a similarity of microstructures was
615 observed between our material and Brantut et al. (2011)'s: in both studies, deformation appears to be
616 driven by intracrystalline plastic mechanisms that result in kink bands. As suggested by Brantut et al.
617 (2011), these structures are consistent with the hardening in the mechanical data because of the finite
618 strain that can be accommodated by a single grain. Once the maximum folding was reached, the

619 energy required to start to kink a new grain was lower than the energy needed to bring the grain to
620 failure.

621 In contrast to Brantut et al. (2011)'s study, we could measure the volumetric strain during our
622 experiments on branching selenite gypsum. Dilatancy was observed even at the highest tested
623 pressures, which suggests that mostly dilatant structures developed in our samples. This is however
624 consistent with the kink deformation observed in microstructures. Indeed, as shown in Figure 17d,
625 the creation of kink bands is often associated with the opening of intracrystalline cracks at the tip of
626 the bands. Since this is similar to the observed post-deformation microstructures in Volterra gypsum
627 (Brantut et al., 2011), we can speculate that this was also the case in that rock. This macroscopic
628 volume increase does not however rule out the possibility that local compaction occurred in some
629 parts of our samples, particularly at high effective pressures, as it has recently been shown in
630 carbonates using X-ray computed tomography and digital volume correlation by Baud et al. (2021).

631 Data on triaxially deformed gypsum are scarce and it was therefore interesting to compare our new
632 results with existing data on Volterra gypsum. However, due to the strong impact of sample size on
633 strength (Paterson and Wong, 2005, Bozorgzadeh et al, 2017, Gao et al., 2018), we were unable to
634 perform this comparison using the results of Brantut et al. (2011), despite the similarities with our
635 data. We instead present in Figure 23 a comparison of the dry failure envelopes of branching selenite
636 gypsum (from this study) and of Volterra gypsum based on the study of Olgaard et al. (1995), who
637 used a comparable sample size. Two important observations can be made. First, Volterra gypsum is
638 significantly stronger than branching selenite gypsum, in agreement with our uniaxial data (see
639 supplementary material S1). This large difference is most certainly due to a combination of factors:
640 the very low porosity and smaller grain size of Volterra (about 1 mm in branching selenite and 100
641 μm in Volterra gypsum) and the presence of secondary minerals such as clay in branching selenite
642 gypsum (compared to 99% gypsum in Volterra). Secondly, the shape of the failure envelope of
643 branching selenite gypsum suggests that beyond an effective pressure of 150 MPa, the behavior may
644 switch to a cap-like envelope associated with shear-enhanced compaction, as observed in other tight
645 rocks (e.g. Solnhofen limestone Baud et al., 2000 or basalts Zhu et al., 2016). To our knowledge, this
646 switch in gypsum behaviour has not been observed to date. Further tests, under high confining
647 pressures, are required to confirm this hypothesis.

648 **Figure 23**

649 *6.4 Implications for quarry exploitation*

650 As confirmed by Figure 19a, a water weakening of 50% in strength is observed in most gypsum
651 facies, despite differences in dry strength.

652 This may have impact on several societally relevant fields, the most obvious being the reduction of
653 geological risks underground. There are many examples of subsurface rock bodies in which water
654 circulates. The impact of water on the mechanical performance of the rock is therefore relevant,
655 necessitating dedicated stability assessments of long-term underground excavations (e.g. civil and
656 mining tunnels, caverns, etc.). The decrease of gypsum strength, both under uniaxial and triaxial
657 loading conditions, and the enhancement of creep deformation in water described in this paper may
658 significantly affect the general stability of the voids.

659 The water-related weakening of gypsum, particularly if associated with underground tunnels, may
660 also enhance sinkhole and surface subsidence risk scenarios.

661 In recent years, the behaviour of gypsum and other salt minerals has raised the interest of the scientific
662 community due to the possibility of using salt caverns for energy storage including Underground Gas
663 Storage (UGS) and Compressed Air Energy Storage (CAES). Salt caverns have also been identified
664 as key elements for the bulk storage of hydrogen, nowadays considered as the best decarbonization
665 option for long-term seasonal energy storage. However, these ‘new’ applications are not yet cost
666 competitive, in part because of the gaps in knowledge about the mechanical response of evaporite
667 rocks.

668 In this context, the outcomes of this study provide new fundamental elements that may inform our
669 understanding of the feasibility of energy storage strategies.

670 **7. Conclusions**

671 In this work, we study the impact of water on the strength and rheology of gypsum rock. Our main
672 results may be summarised as follows:

- 673 1. Under uniaxial compression, the strength of water-saturated branching selenite gypsum is
674 about half of its value under nominally dry conditions. Moreover, water also induces certain
675 changes in the mechanical behaviour (e.g. disappearance of stress drops, limited strain
676 softening). Our microstructural observations suggest that water weakening is due to the
677 combined effect of the reduction of the fracture surface energy and dissolution along the
678 surfaces of the mineral cleavage, weakening the crystals’ strength.
- 679 2. Under uniaxial compression, significant time dependent behaviour is observed in the presence
680 of water but not in dry or oil-saturated samples. The UCS of gypsum decreases with
681 decreasing strain rate. Moreover, brittle creep is observed at stresses beyond the elastic

682 regime. Creep in gypsum is strongly influenced by sample variability but our data show that
683 the initial porosity of the material has a major impact on creep strain rates. The main attributes
684 of brittle creep in gypsum are broadly similar to those of other rock types. Dilatancy and
685 stress-induced microcracking point to stress corrosion as the main mechanism of time
686 dependent deformation in gypsum. At low strain rates however, more strain is observed
687 suggesting the action of other mechanisms.

688 3. Deformation of gypsum under triaxial compression is driven by kinking of the gypsum grains
689 and is dilatant up to high confining pressures (i.e. 100 MPa). The water-weakening quantified
690 at high pressures is similar to our observations under uniaxial compression. Comparison of
691 our new data with published results on Volterra gypsum highlights the inverse relationship
692 between porosity and triaxial yield point in gypsum. Our triaxial data suggest that shear-
693 enhanced compaction, which was not observed at the conditions of our experiments, could
694 possibly develop in branching selenite gypsum at confining pressures beyond 150 MPa.

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701

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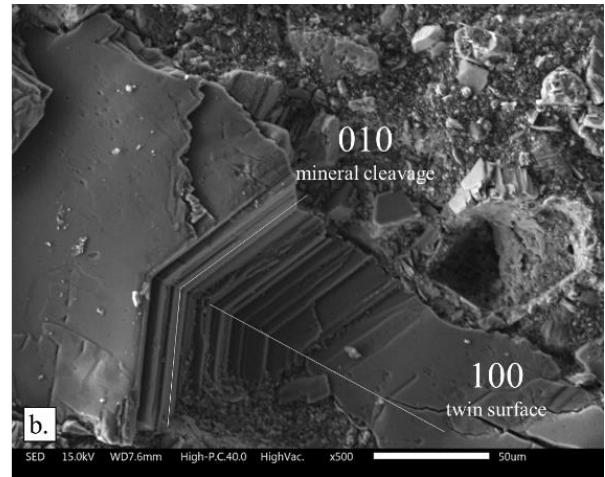
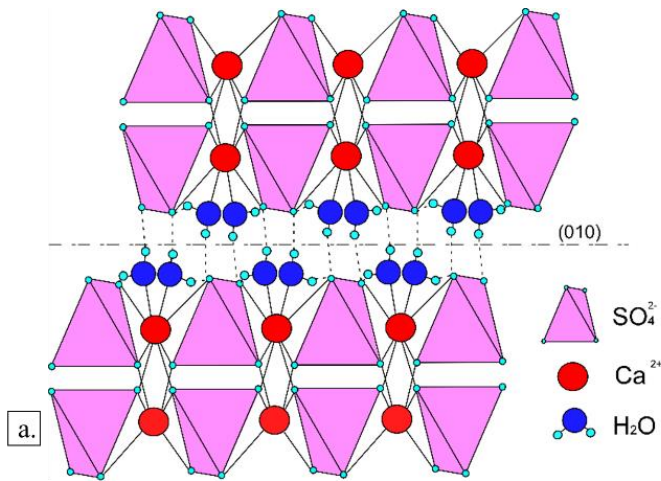
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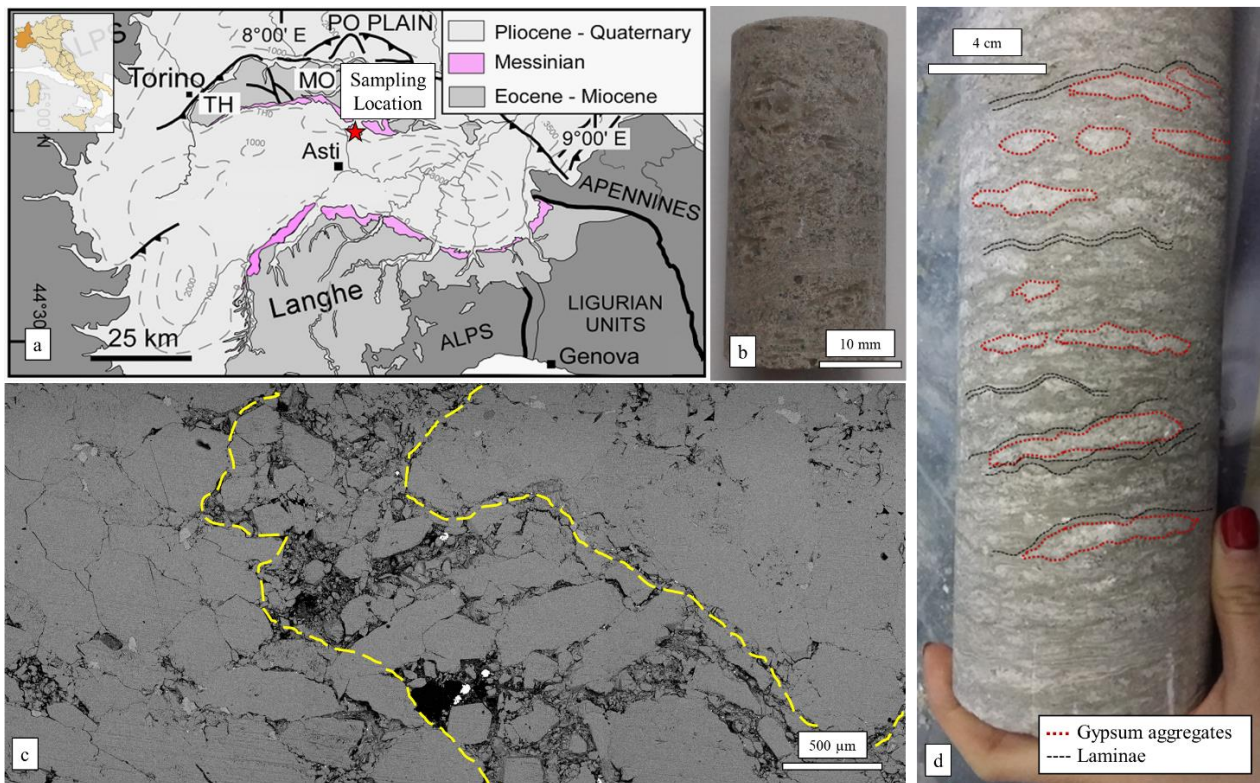
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969 deformation of gypsum aggregates experimentally deformed in torsion to high strains:
970 Quantitative microstructural and texture analysis from optical and diffraction data. *Geol.*
971 *Soc. Lond. Spec. Publ.* 332, 79-98. doi:10.1144/SP332.6. <https://doi.org/10.1144/SP332.6>
- 972



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975 Figure 1. (a) Crystallographic structure of gypsum. (b) Selenite crystal, with the perfect mineral
976 cleavage on the (010) crystallographic direction and the contact twin on the (100) crystallographic
977 plane.

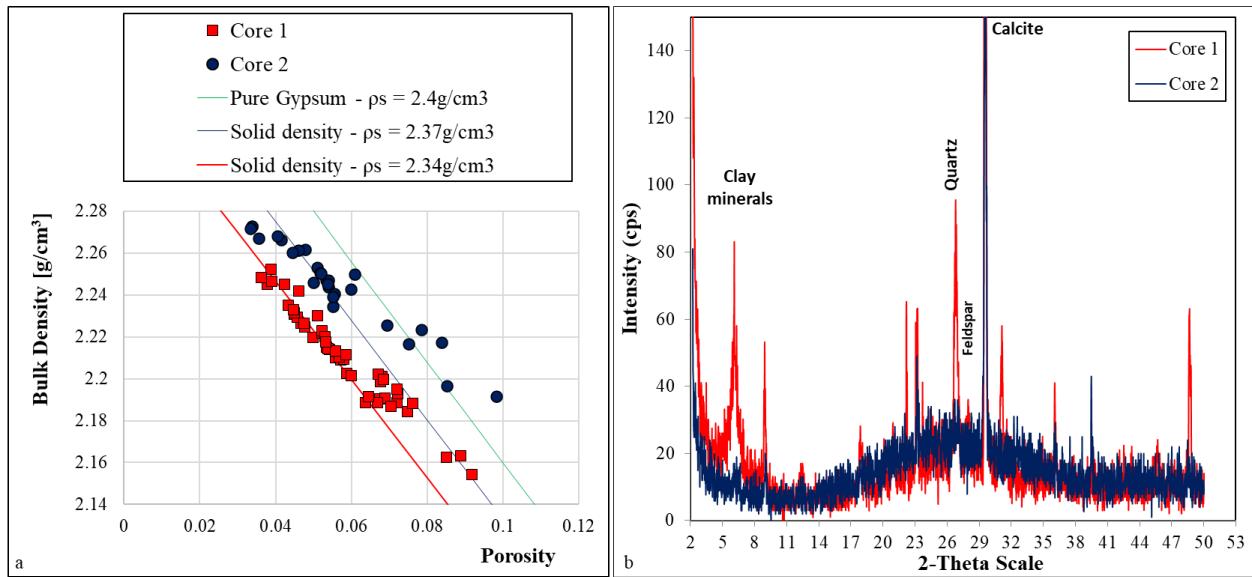
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980 Figure 2. (a) Map of the test area, showing the areal distribution of Messinian gypsum deposits in
 981 Piedmont and the geographical position of the sampling site (red star); TH = Torino Hill, MO =
 982 Monferrato. (b) 40mm by 20mm sample of branching selenite gypsum, before deformation. (c) BSE-
 983 SEM microstructure of the branching selenite gypsum, showing the main features of this facies: the
 984 yellow dashed lines show the boundary between the nodules of gypsum crystals (on the right and on
 985 the left) and the finer material (in the middle). (d) Initial borehole core, before resampling. Examples
 986 of gypsum aggregates and laminae are highlighted using red and black dotted lines respectively.

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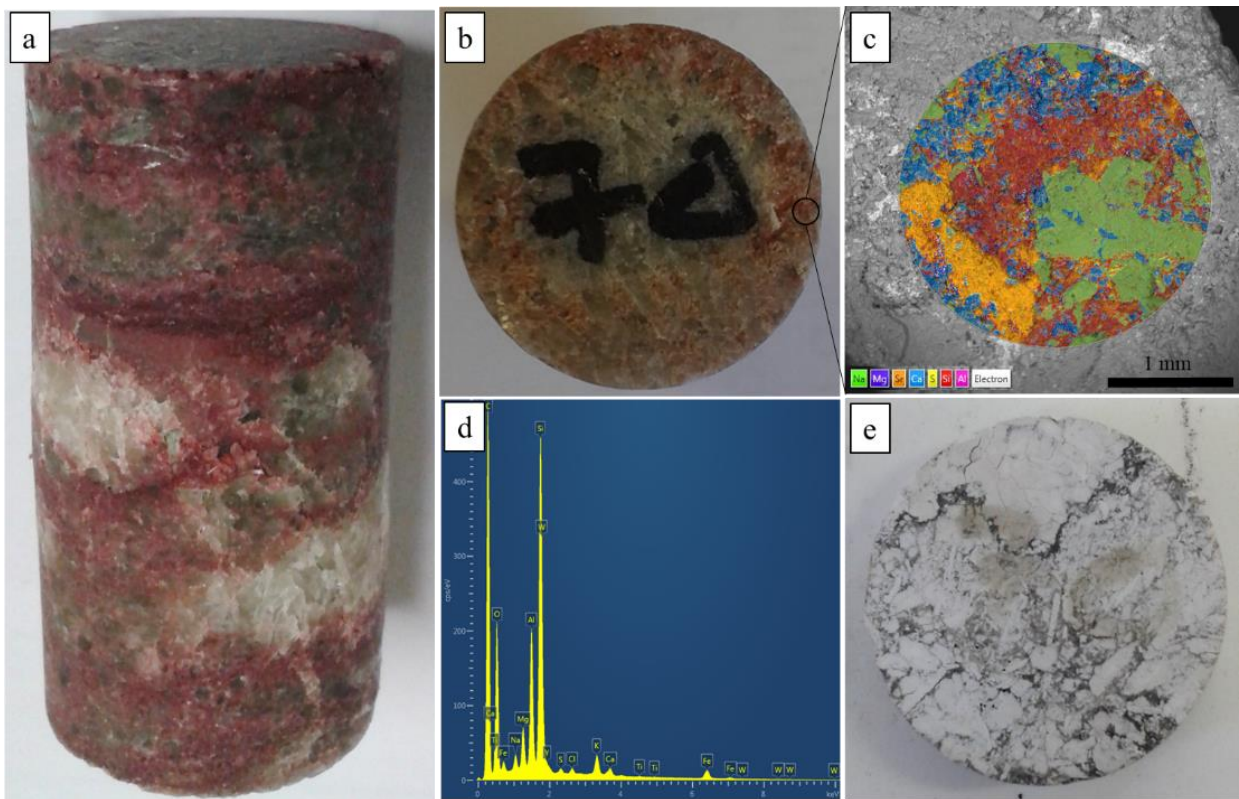


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990 Figure 3. (a) Measurements of porosity against bulk density in the 77 samples, divided on the basis
 991 of the starting core (Core 1 – 82.4-82.9 m of depth; Core 2 – 78.0-78.7 m). The low dispersion of the
 992 data suggests compositional homogeneity of samples. The lines represent the theoretical value of
 993 solid density for pure gypsum (2.4 g/cm³) and the estimated values of solid density for Core 1 and
 994 Core 2 respectively. (b) XRPD analyses of the not-gypsum portion of material from Core 1 (red line)
 995 and Core 2 (blue line), showing respectively the abundant presence of clay minerals, in addition to
 996 quartz feldspars and calcite, in Core 1 and the prevalence of calcite in Core 2 (Caselle et al. 2022)

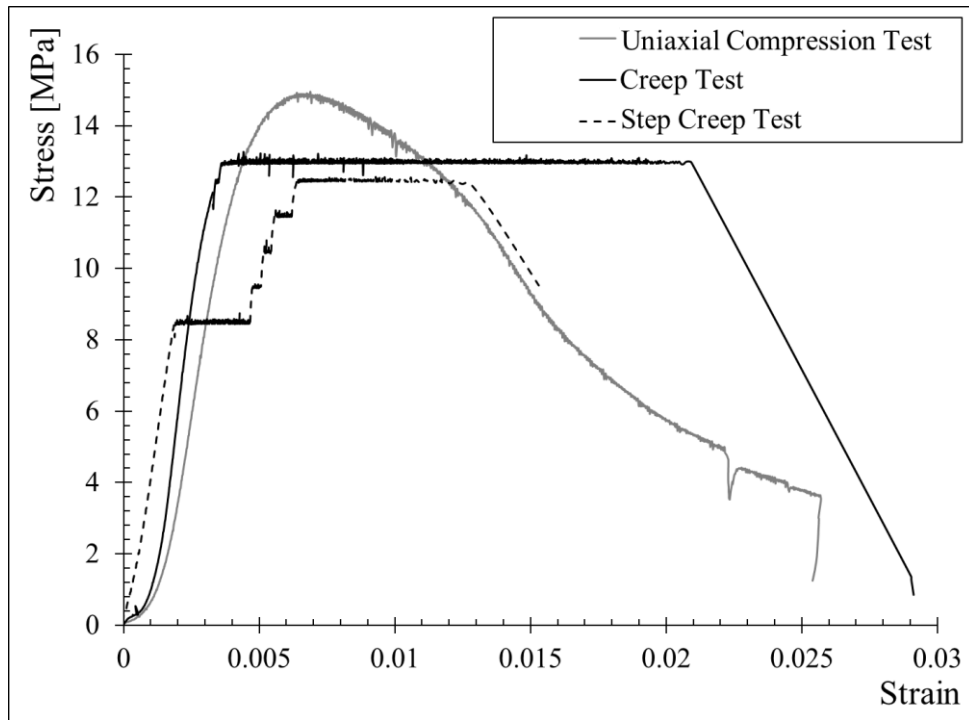
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1000 Figure 4. (a) Red discolouration of an oil-saturated sample immediately after a compression test. (b)
 1001 Red colouration of oil-saturated sample after one month (c) SEM-EDS compositional map. The green
 1002 portions correspond to gypsum (Ca + S), the orange portions to celestine (Sr + S), blue crystals are
 1003 carbonate minerals (mainly Ca), while red portions are the thin-grained matrix of the rock
 1004 (corresponding to main Si and Al, with minor S, Sr and Ca). (d) Punctual SEM-EDS analysis of red
 1005 portions. (e) Oil-saturated gypsum sample in Figure 4b after heating at temperature of 350°C: gypsum
 1006 crystals are turned to anhydrite and red portions are turned to black after the expulsion of
 1007 dearomatized oil.

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1010 Figure 5. Comparison of typical data for uniaxial compression, conventional uniaxial creep and step
 1011 creep tests.

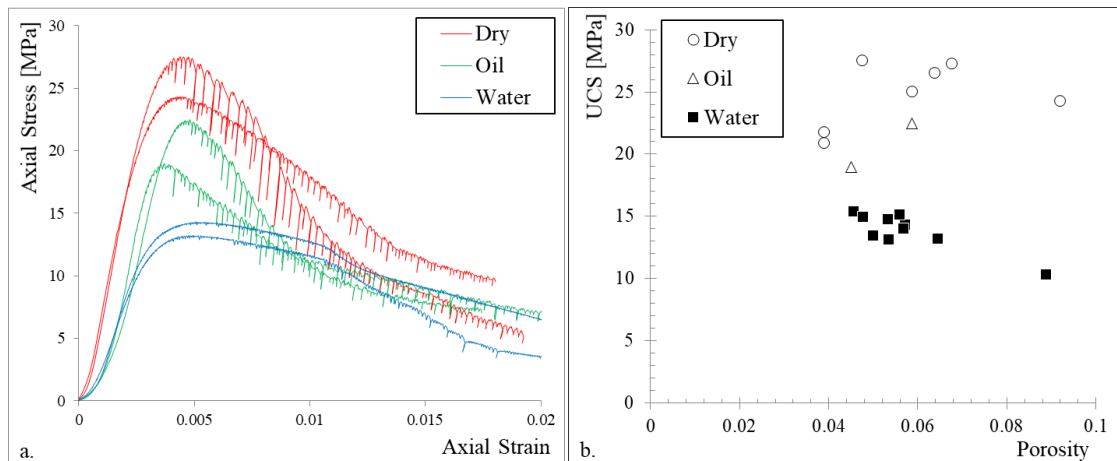
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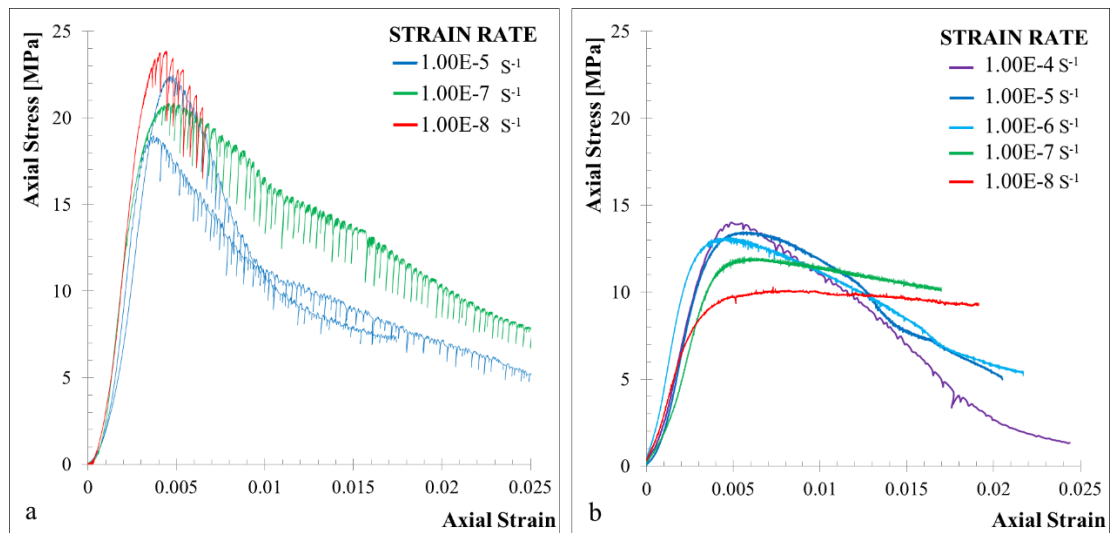
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1018 Figure 6. (a) Stress-strain curves of UCS tests on gypsum for dry (red lines), oil-saturated (green
 1019 lines) and water-saturated (blue lines) samples. (b) UCS as a function of porosity for dry (open
 1020 circles), oil-saturated (open triangles) and water-saturated (black squares) conditions.

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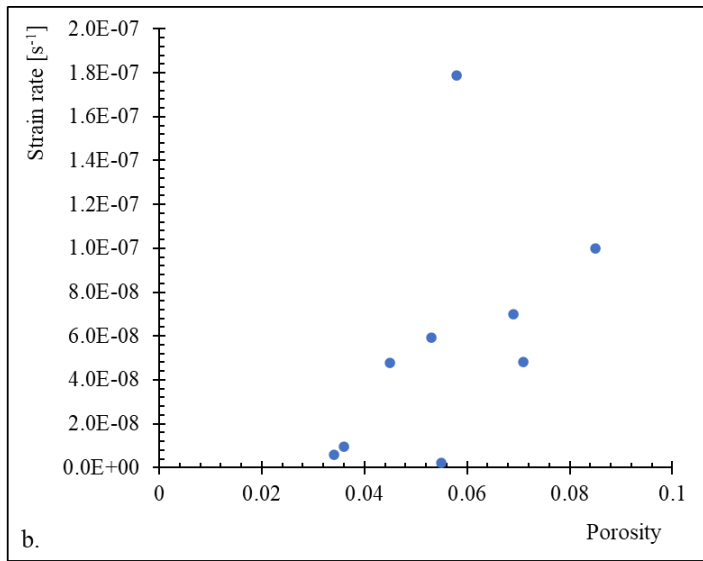
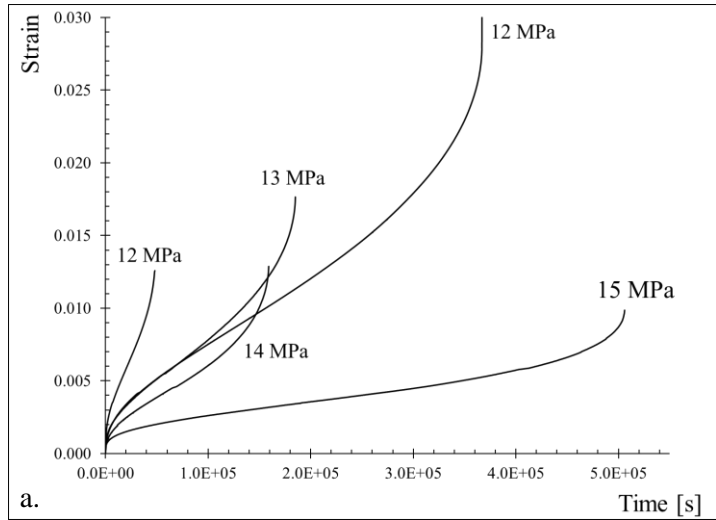
1023 Figure 7. Oil-saturated samples deformed in uniaxial compression with strain rate of $10^{-5}/\text{s}$ (blue
 1024 lines), $10^{-7}/\text{s}$ (green line) and $10^{-8}/\text{s}$ (red line). (b) Water saturated samples deformed in uniaxial
 1025 compression with strain rate of $10^{-4}/\text{s}$ (purple line), $10^{-5}/\text{s}$ (blue line), $10^{-6}/\text{s}$ (light blue line), 10^{-7}
 1026 (green line) and $10^{-8}/\text{s}$ (red line).

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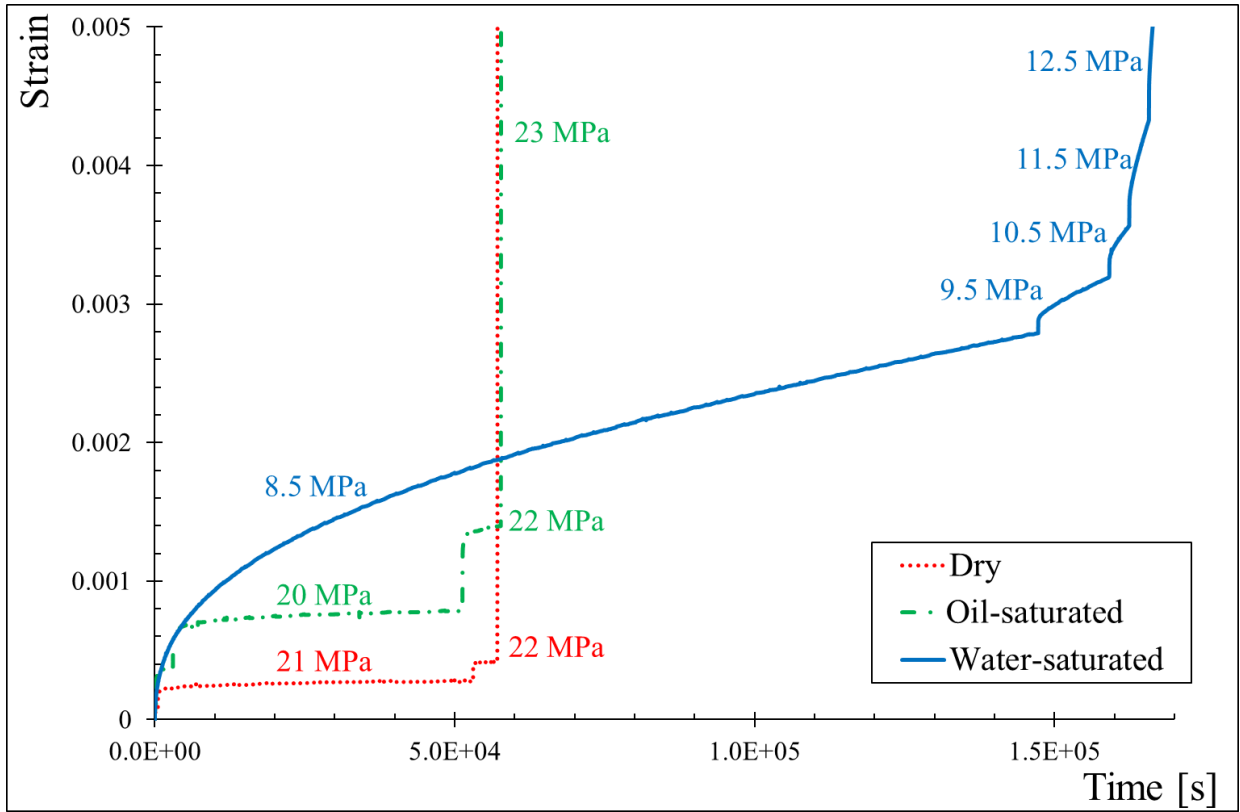
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1033 Figure 8. a. Representative strain-time curves obtained from creep tests on water-saturated samples.
 1034 The applied stress is indicated next to each curve b. Values of strain rate measured in creep tests on
 1035 water saturated samples against their initial porosity.



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1037 Figure 9. Results of step tests on dry (red line), oil-saturated (green line) and water-saturated (blue
 1038 line) samples. The stresses applied at each step are reported next to the curves.

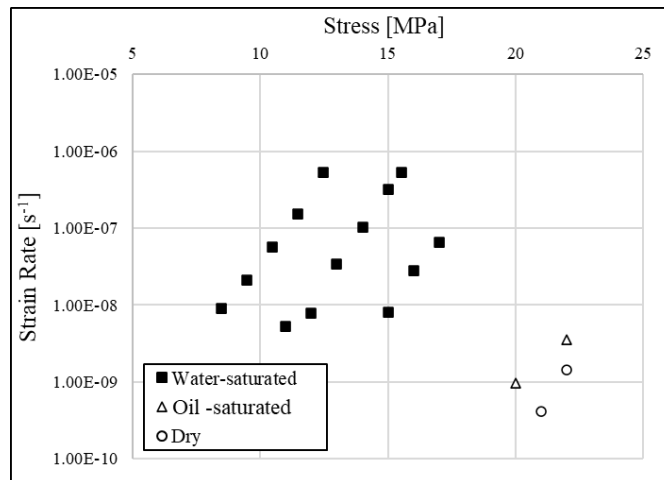
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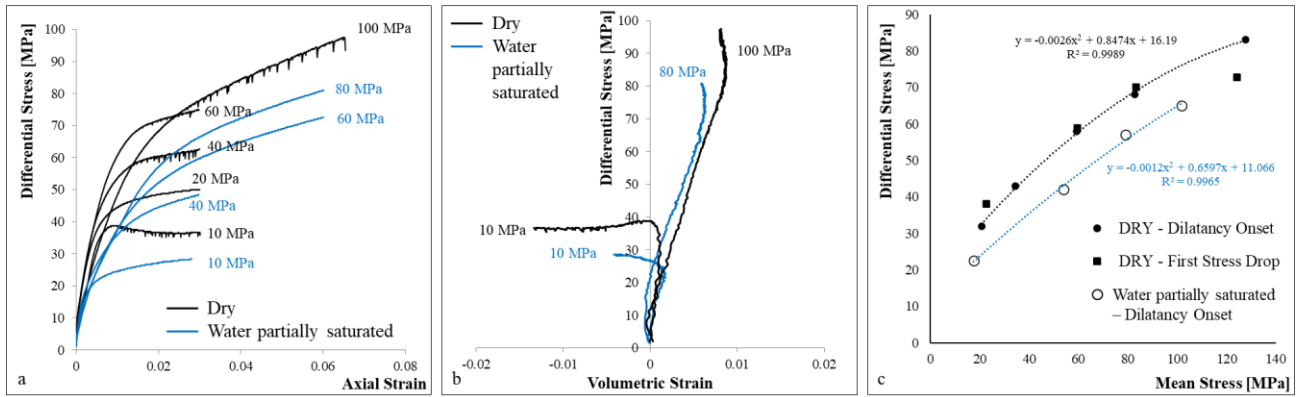
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1045 Figure 10. Creep strain-rate as a function of the applied stress for step tests performed on water-
 1046 saturated (black squares), oil-saturated (open triangles) and dry (open circles) samples.



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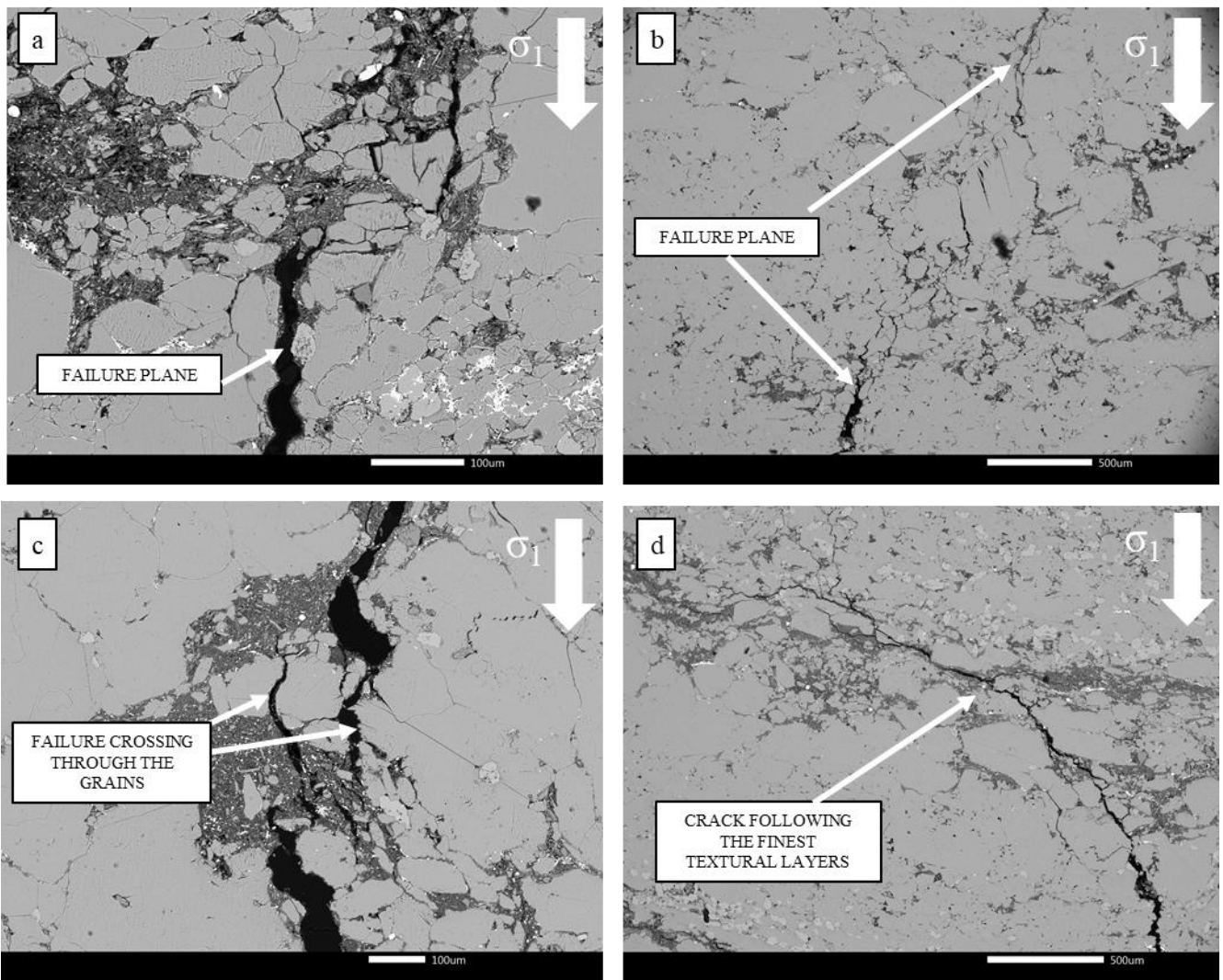
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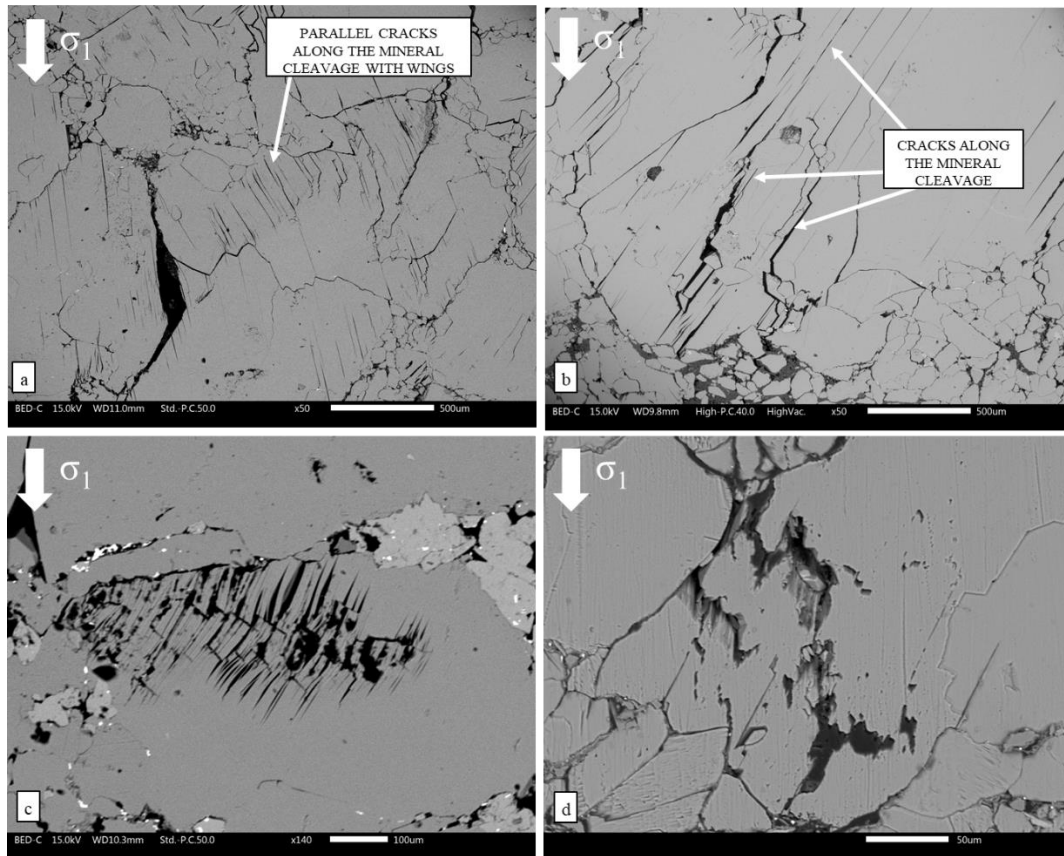
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Figure 11. Triaxial data for branching selenite gypsum. (a) Differential stress as a function of axial strain for dry (black) and partially saturated (blue) samples. (b) Differential stress as a function of volumetric strain for dry (black) and partially saturated (blue) samples deformed under the highest and the lowest confining pressure (c) Critical stresses: onset of dilatancy (circles) and first stress-drop in dry conditions (squares). The data are presented in the stress space (differential stress-mean stress) for dry (closed symbols) and wet (open symbols) samples. Failure envelopes are reported in black for dry and in blue for wet samples.



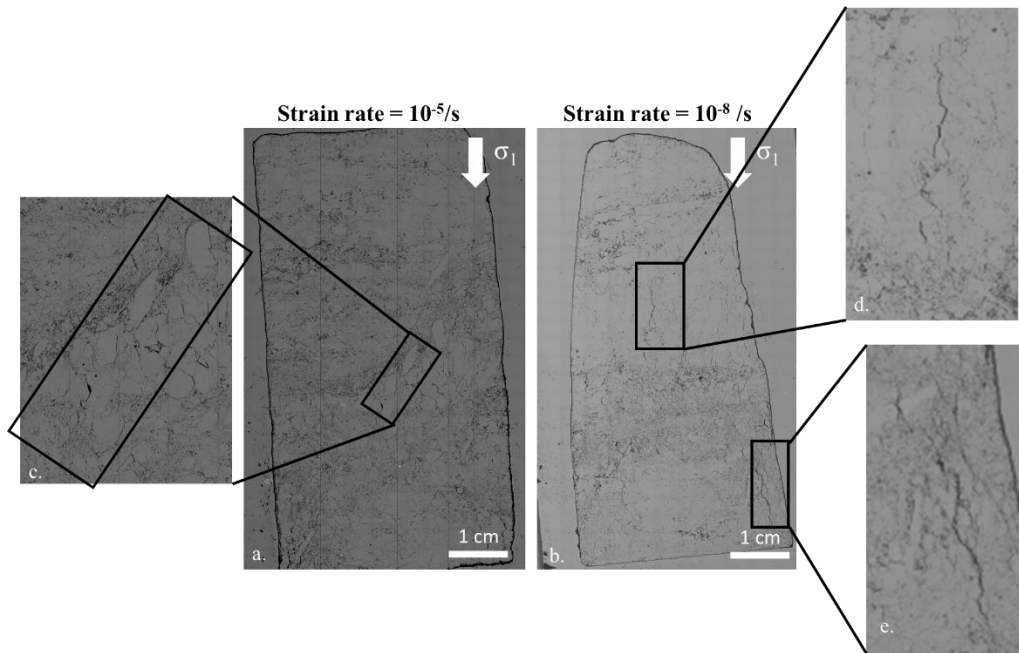
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1058 Figure 12 BSE-SEM micrographs of sample deformed uniaxially under dry conditions (sample 34).
 1059 (a) Failure plane interrupted by changes in microtexture (b) Two crack surfaces, highlighted in the
 1060 upper right and lower left of the image, that are aligned along a plane but are not continuous in the
 1061 central portion of the image. (c) Cracks that are interrupted by finer material and cut through gypsum
 1062 grains (d) Crack following the sub-horizontal layering of the branching selenite structure.



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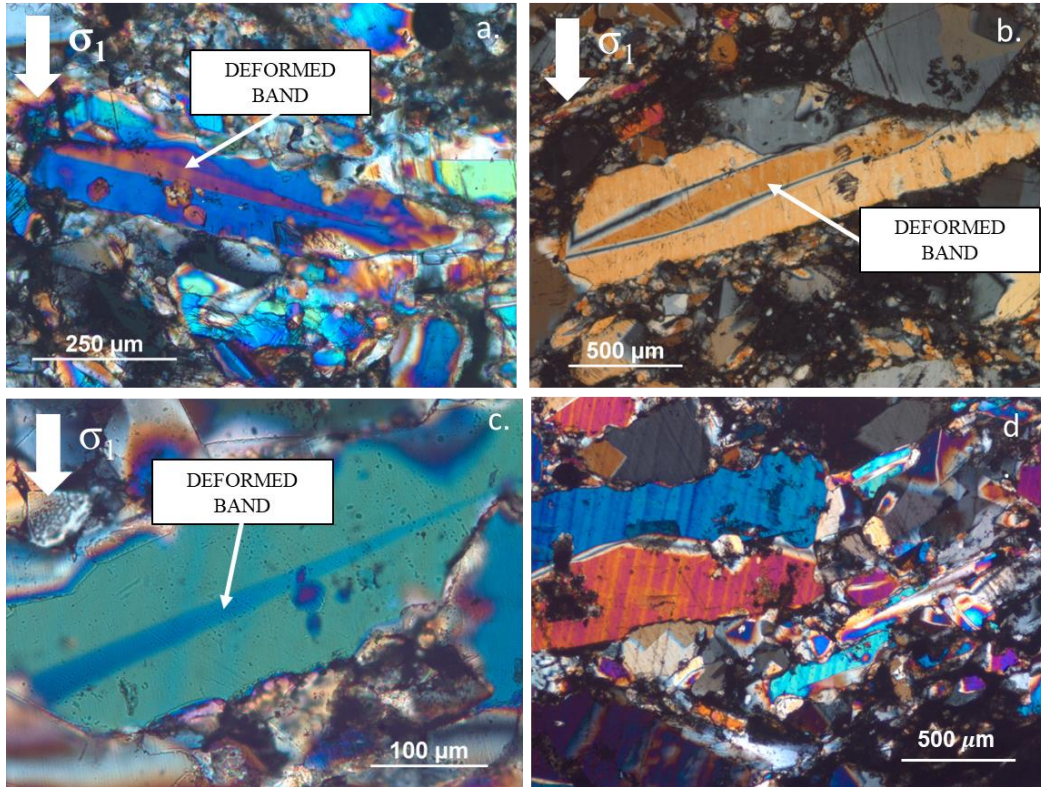
1064 Figure 13 BSE-SEM microstructures of samples deformed uniaxially under wet conditions. Figures
 1065 show samples 13, 8 and 11, deformed at a standard strain rate of 10⁻⁵/s (a and d), slow strain rate 10⁻⁷/
 1066 7/s (b) and under creep conditions (c), respectively. (a) Gypsum crystal with straight parallel
 1067 microcracks with wings consistent with the axial direction of the applied stress. (b) Microcracks
 1068 opening along the gypsum cleavage (c) Intra-crystalline deformation structure, common in slow strain
 1069 rate and creep samples, that consists of a series of narrow cracks along the mineral cleavage that,
 1070 being oriented sub-parallel to the axial applied stress, accommodate the strain by the folding of each
 1071 separated slice and by the creation of short cracks perpendicular to the major principal stress σ_1 (d)
 1072 Detail of a crack with rounded edges and aperture of 10 to 20 μm that was interpreted as the combined
 1073 effect of mechanical loading and dissolution.



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1075 Figure 14 BSE-SEM images of entire thin sections of water-saturated samples deformed uniaxially
 1076 under strain rates of $10^{-5}/s$ (a) and $10^{-8}/s$ (b) (samples 13 and 8, respectively). Magnified images in c.,
 1077 d. and e. highlight the mean orientation of main cracks: with an angle of 30° with respect to the axial
 1078 load in sample 13 (Figure c) and subvertical in sample 8 (Figures d and e).

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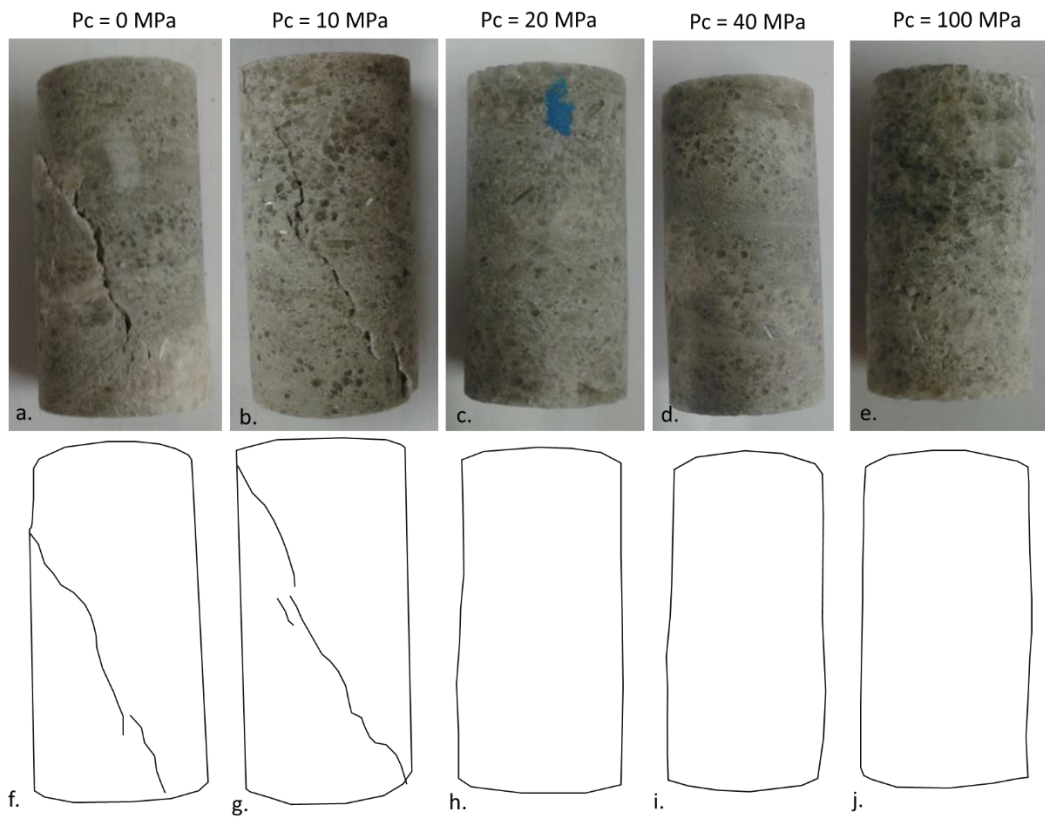


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1081 Figure 15 a, b, c. Crossed polar optical microscope images of sample 11 (water saturated, step creep
 1082 test) showing the presence of deformation bands in the bigger crystals oriented sub-perpendicular to
 1083 the applied load. d. Example of gypsum crystal in the undeformed material.

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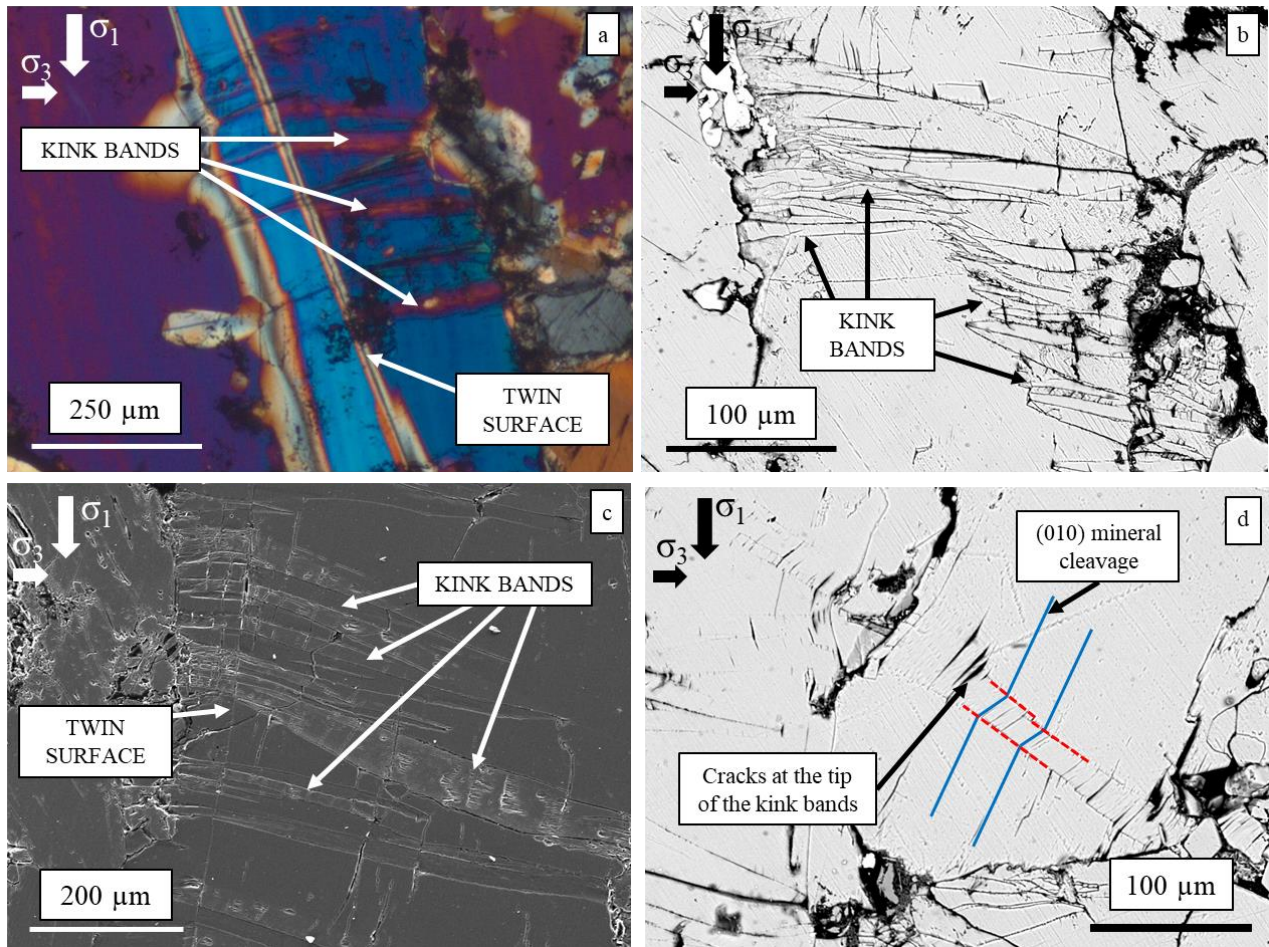
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 1087 Figure 16 Post-deformation photos of samples deformed under dry conditions (a to e) and outlines of
 1088 the external shape of the samples and of the main macroscopically visible fractures (f to j).

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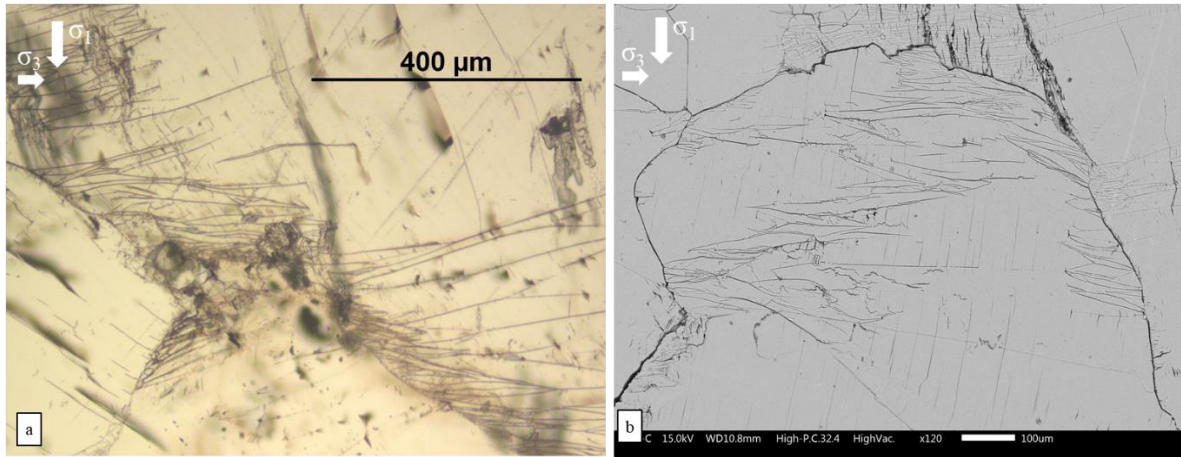
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1092 Figure 17 Kinked grains in the microstructures of samples deformed triaxially (samples 28 and 29).
 1093 (a) Cross-polar optical microscope image of a sample deformed dry with confining pressure of 20
 1094 MPa. The twinned gypsum crystal in the image (light blue) is deformed by a series of subhorizontal
 1095 kink bands: the change of orientation of the crystal leads to in a change in optical properties, resulting
 1096 in a change of birefringence colour from light blue to purple. (b) SEM-BSE image of a crystal
 1097 showing intense kink deformation in a sample deformed dry under a confining pressure of 20 MPa.
 1098 The boundaries of kink bands are clearly marked. (c) SEM-BSE image showing a twinned crystal
 1099 with subhorizontal kink bands. The sample was deformed wet under a confining pressure of 40 MPa.
 1100 (d) Detail of a kinked grain. The red dashed lines define the boundaries of the kink band; blue lines
 1101 are parallel to the mineral cleavage, showing how it is deflected by the kinking deformation. The
 1102 image, acquired with SEM-BSE, refers to a sample deformed dry under a confining pressure of 20
 1103 MPa with stress directions parallel to the black arrows.

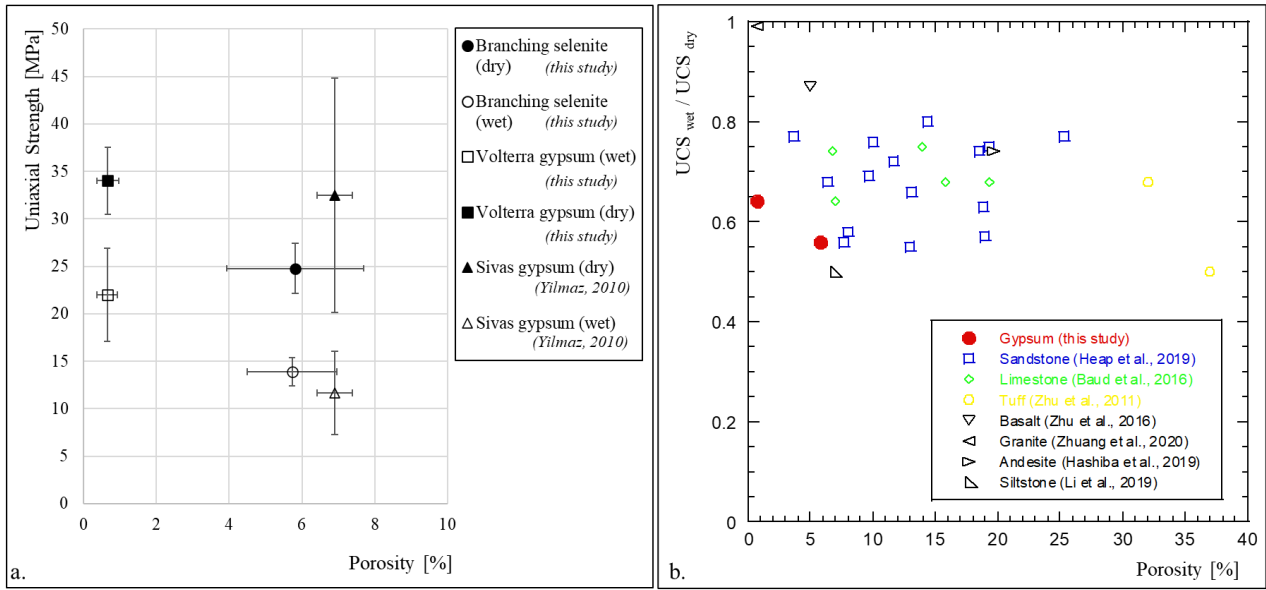
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1106 Figure 18 Images of shear bands created by the alignment of kink structures in a sample deformed at
1107 a confining pressure of 40 MPa (sample 28). Images were acquired at optical microscope (a) and BSE
1108 electron microscope (b).

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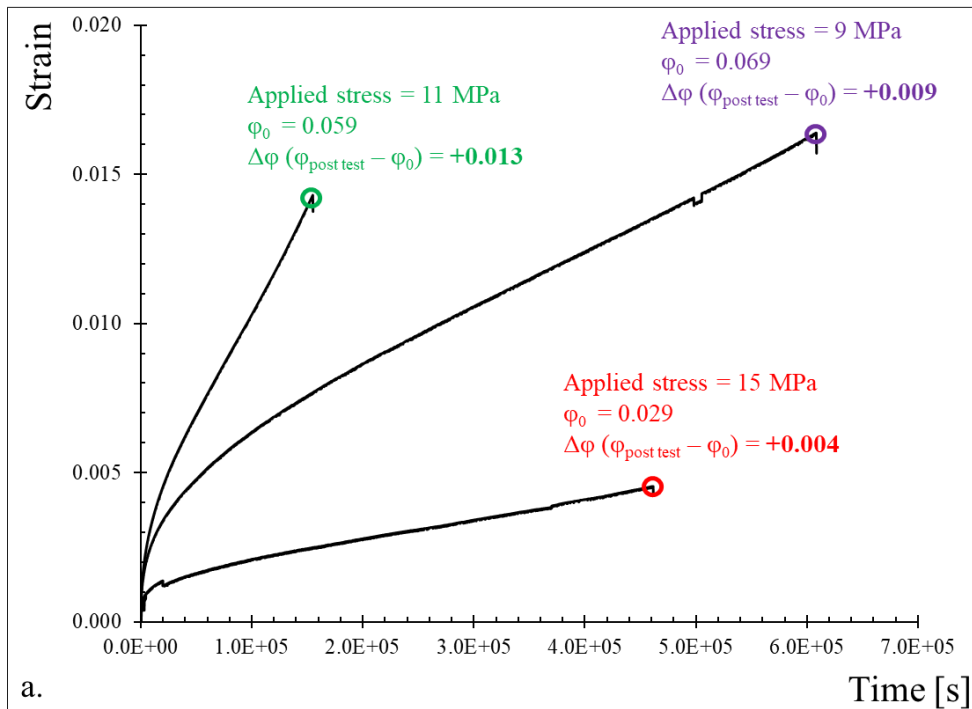


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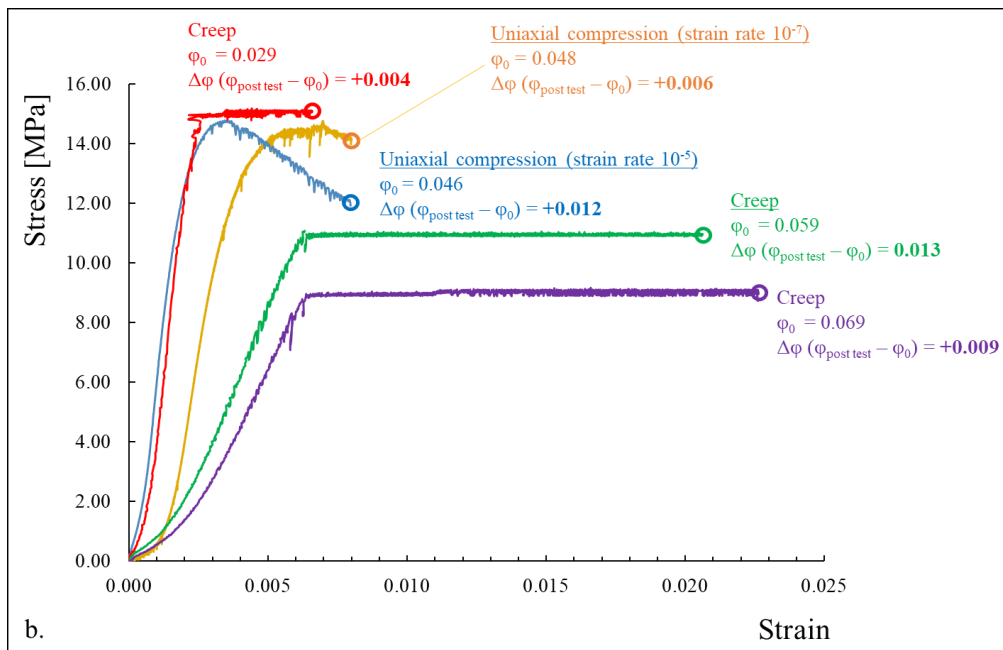
1111 Figure 19. (a) UCS as a function of porosity for branching selenite (circles), Volterra gypsum
 1112 (squares) and gypsum from the Sivas basin studied by Yilmaz (2010) (triangles). Closed and open
 1113 symbols are for dry and wet samples, respectively. (b) Ratio of wet UCS to dry UCS as a function of
 1114 porosity for gypsum (red circles), sandstone (blue squares), tuff (yellow circles), basalt, granite,
 1115 andesite, and siltstone (black symbols).

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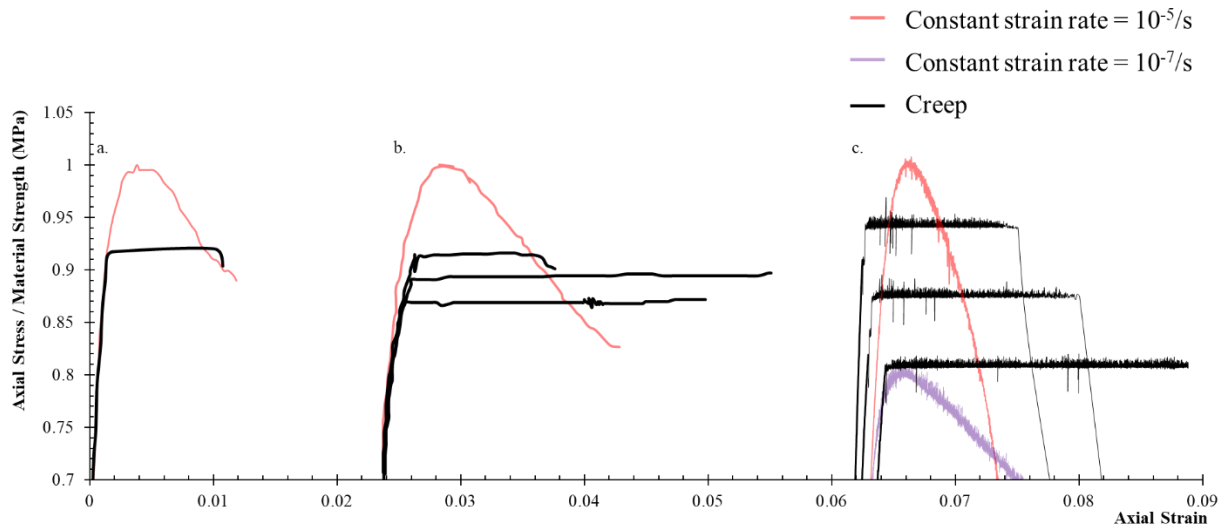
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1123 Figure 20. (a) Creep data (strain as a function of time) for targeted experiments on wet gypsum

1124 samples stopped before failure. The initial porosity and the total porosity change are indicated next

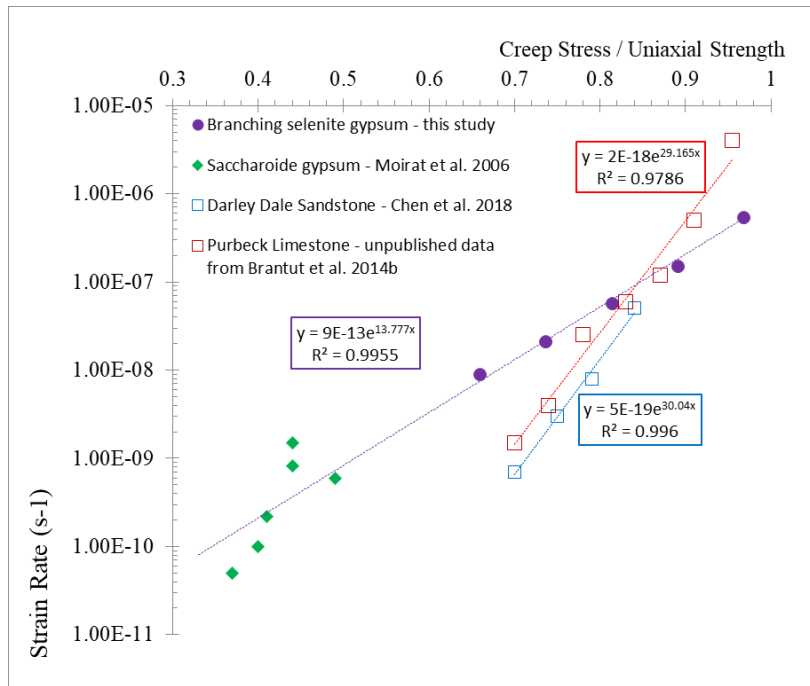
1125 to the curves. (b) Comparison between constant strain rate and creep data. Stress is presented as a

1126 function of strain. ϕ_0 represents the initial porosity of the samples while $\Delta\phi$ is the difference between
1127 the final porosity ($\phi_{\text{post test}}$) and the initial porosity ϕ_0 .



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1129 Figure 21 Differential stress as a function of axial strain for a constant strain rate (red) and a creep
1130 test (black) on Darley Dale sandstone (Brantut et al., 2014a). (b) Differential stress as a function of
1131 axial strain for a constant strain rate (red) and a creep test (black) on Purbeck limestone (Brantut et
1132 al., 2014b). (c) Comparison between constant strain rate uniaxial data (red and violet) and creep data
1133 on branching selenite gypsum (black). Significantly more strain accumulated during the creep
1134 experiments in Purbeck limestone and branching selenite gypsum.

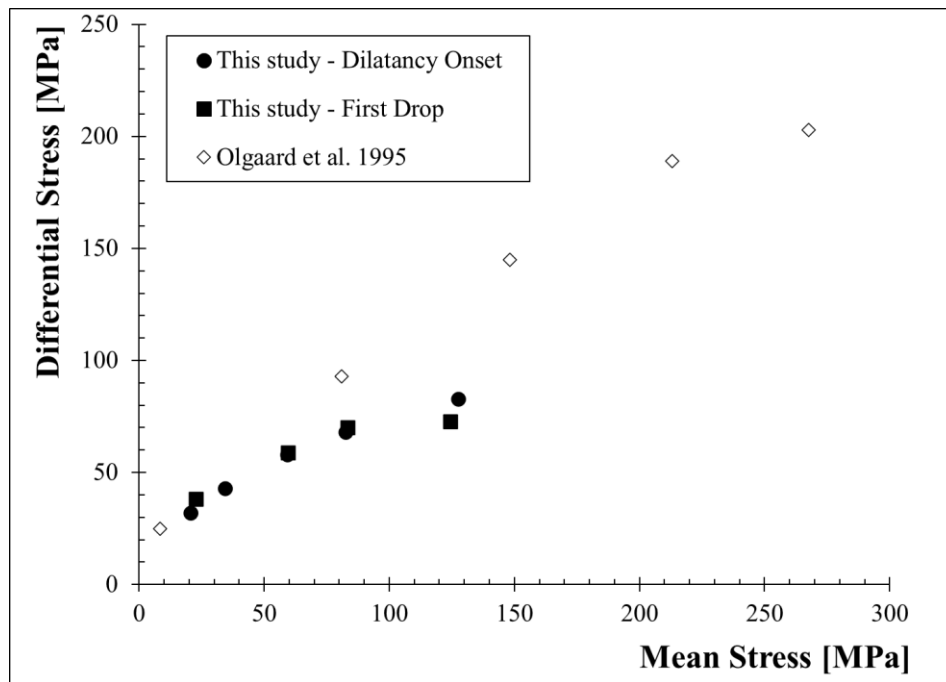
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1137 Figure 22 Creep strain rate as a function of the normalized stress for uniaxial creep experiments
 1138 performed on branching selenite gypsum (violet circles), saccharoide gypsum (green diamonds,
 1139 Moirat et al., 2006), Darley dale sandstone (blue squares, Chen et al., 2018) and Purbeck limestone
 1140 (red squares, unpublished data by Brantut et al., 2014b). Exponential fits of the data are also shown.

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1143 Figure 23 Comparison of the yield envelopes of branching selenite gypsum (black symbols) and of
 1144 Volterra gypsum (open diamonds) from Olgaard et al. (1995).

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1163 Table 1 Test conditions and results of uniaxial compression experiments

Strain Rate s ⁻¹	Number of tests	Saturation conditions	Porosity	UCS (range of values) MPa
10 ⁻⁵	7	Dry	0.039 – 0.092	20.92 - 27.52
	2	Oil	0.045 – 0.059	18.97 - 22.48
	7	Water	0.046 – 0.089	10.34 - 15.40
10 ⁻⁴	1	Water	0.057	14.00
10 ⁻⁶	2	Water	0.054 – 0.059	13.15 - 15.15
10 ⁻⁷	2	Water	0.054	12.04 - 14.76
	1	Oil	0.061	20.86
10 ⁻⁸	1	Water	0.060	10.31
	1	Oil	0.060	23.86

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1168 Table 2. Summary of conventional and step creep tests performed in this study.

	Saturation	Porosity	Stress MPa	Strain Rate s ⁻¹
Creep tests	Water	0.058	12	2E-07
	Water	0.071	12	5E-08
	Water	0.045	14	5E-08
	Water	0.053	13	6E-08
	Water	0.036	15	1E-08
	Water	0.055	8.9	2E-09
	Water	0.034	15	6E-09
	Water	0.069	11	7E-08
	Water	0.085	9.5	1E-07
Step creep tests	Water	0.038	11; 12; 13; 14; 15; 15.5	5E-9; 8E-9; 3E-8; 1E-7; 3E-7; 5E-7
	Water	0.067	8.5; 9.5; 10.5; 11.5; 12.5	9E-9; 2E-8; 6E-8; 2E-7; 5E-7
	Water	0.051	15; 16; 17	8E-9; 3E-8; 6.5E-8
	Oil	0.067	20; 22	1E-9; 4E-9
	Dry	0.042	21; 22	4E-10; 1E-9

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1173 Table 3. Summary of the mechanical data for triaxial experiments for dry and partially saturated
1174 samples of branching selenite gypsum.

Confining Pressure [MPa]	Onset Dilatancy		First Stress Drop	
	Mean Stress [MPa]	Differential Stress [MPa]	Mean Stress [MPa]	Differential Stress [MPa]

	10.00	20.67	32.00	22.70	38.10
	20.00	34.33	43.00		
Dry	40.00	59.33	58.00	59.63	58.90
	60.00	82.67	68.00	83.36	70.09
	100.00	127.67	83.00	124.27	72.80
	10.00	17.50	22.50		
Wet	40.00	54.00	42.00		
	60.00	79.00	57.00		
	80.00	101.67	65.00		

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