

1 **Dynamic in situ three-dimensional imaging and digital volume correlation**
2 **analysis quantify strain localization and fracture coalescence in sandstone**

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20
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22 Fontainebleau sandstone, digital volume correlation

23
24 **Abstract**

25 Advances in triaxial compression deformation apparatus design, dynamic X-ray
26 microtomography imaging, data analysis techniques, and digital volume correlation analysis
27 provide unparalleled access to the in situ four-dimensional distribution of developing strain
28 within rocks. To demonstrate the power of these new techniques and acquire detailed information
29 about the micromechanics of damage evolution, deformation and failure of porous rocks, we
30 deformed three centimeter-scale cylindrical specimens of low porosity Fontainebleau sandstone
31 in an X-ray transparent triaxial compression apparatus, and repeatedly recorded three-

32 dimensional tomograms of the specimens as the differential stress was increased until
33 macroscopic failure occurred. Experiments were performed at room temperature with a confining
34 pressure in the range 10-20 MPa. Distinct gray-scale subsets, indicative of density, enabled
35 segmentation of the three-dimensional tomograms into intact rock matrix, pore space, and
36 fractures. Digital volume correlation analysis of pairs of tomograms provided time series of
37 three-dimensional incremental strain tensor fields throughout the experiments. After the yield
38 stress was reached, the samples deformed first by dilatant opening and propagation of
39 microfractures, and then by shear sliding via grain rotation and strain localization along faults.
40 For two samples, damage and dilatancy occurred by grain boundary opening and then a sudden
41 collapse of the granular rock framework at failure. For the third sample, a fault nucleated near the
42 yield point and propagated in the sample through the development of transgranular
43 microfractures. The results confirm findings of previous experimental studies on the same rock
44 and provide new detailed quantifications of: 1) the proportion of shear versus dilatant strain in the
45 sample; 2) the amount of dilatancy due to microfracture opening versus pore opening when a
46 fault develops; 3) the role of grain boundaries and pore walls in pinning microfracture
47 propagation and slowing down the rate of damage accumulation as failure is approached. Our
48 study demonstrates how the combination of high resolution in-situ dynamic X-ray
49 microtomography imaging and digital volume image correlation analysis can be used to provide
50 additional information to unravel brittle failure processes in rocks under stress conditions relevant
51 to the upper crust.

52

53 **1. Introduction**

54 Acquiring detailed observations about the processes that control the propagation and
55 coalescence of microfractures that lead to system-size failure is critical for robust understanding
56 of borehole and tunnel stability, the geometry of fractures and faults in crustal reservoirs, and
57 earthquake physics. Microfractures may coalesce and lead to macroscopic failure (Scholz, 1968;
58 Wawersik and Fairhurst, 1970; Mogi, 1971; Peng and Johnson 1972; Tapponnier and Brace,
59 1976; Lockner et al., 1991; Dresen and Guéguen, 2004; Paterson and Wong, 2005) and evolving
60 microfractures within fault damage zones alter the stress field surrounding faults (Otsuki and
61 Dilov, 2005; Faulkner et al. 2006). Fracture networks may influence fluid flow near major faults,
62 which can decrease the effective stress on the fault plane and lower the shear stress required to

63 trigger an earthquake (Miller et al., 2004). In addition, growing microfracture networks that
64 evolve as rocks approach macroscopic failure may change the mechanical properties of the rock
65 (Heap and Faulkner, 2008), upon which accurate seismic imaging of fault damage zones depends.

66 Many laboratory studies have relied on acoustic emission monitoring to provide insights
67 into deformation preceding failure. This technique was the first method used to probe inside
68 rocks during in situ deformation, and it has very successfully provided information about failure
69 modes and the approximate spatial and temporal distribution of damage events in crystalline
70 rocks and in porous sedimentary rocks (e.g. Scholz 1968; Lockner et al., 1991, 1992; Cox and
71 Meredith, 1993; Wu et al., 2000; Stanchits et al., 2006; Fortin et al., 2009; Ghaffari et al., 2014).
72 A limitation of using acoustic emission recording is that it only captures events that emit acoustic
73 waves with frequencies and intensities that can be detected in the presence of background noise.
74 Furthermore, the velocities, attenuation, scattering and diffraction of acoustic waves depend on
75 spatially varying heterogeneities that evolve with the developing fracture networks.

76 Consequently, the error in the acoustic source location is on the order of several millimeters
77 (Stanchits et al., 2006), which is typically larger than the grain size of the rock. Additional
78 information about the acoustic source size and orientation can be obtained from moment tensor
79 analysis (Kwiatek et al., 2013). This analysis requires high quality data, a large number of well-
80 calibrated and high dynamic range acoustic sensors, and knowledge of the elastodynamic tensor
81 Green's function. Like other mechanical properties, the Green's function changes during
82 fracturing, and can be accurately determined for heterogeneous materials only if its local
83 variations can be measured. Consequently, accurate information about the fracture sizes, shapes
84 and orientations can be obtained from acoustic emission experiments only when high quality and
85 low noise data are available and heavy data processing is performed (e.g. Kwiatek et al., 2013).

86 In situ three-dimensional dynamic X-ray microtomography imaging combined with
87 digital volume correlation (DVC) analysis is complementary to acoustic emissions tomography
88 because it provides detailed information about the evolution of the microscopic and macroscopic
89 strain fields at micrometer-scale spatial resolution and precision. Data may include aseismic
90 deformation that cannot be measured by acoustic emissions. Here, we describe this recent
91 experimental technique, discuss its potential limits and show how it can be applied to study the
92 initiation of faulting in rocks. We focus on the application of this technique to an experimental
93 investigation of the microscopic deformation of three specimens of Fontainebleau sandstone

94 under triaxial compression. Using in situ three-dimensional X-ray tomography, digital volume
95 correlation analysis, and scaling statistics, we quantified the evolution of: 1) porosity, 2)
96 nucleation, growth and coalescence of microfractures and, 3) interactions between local
97 microscopic dilation, contraction and shear strain that leads to system-size shear failure with
98 increasing stress. Segmentation of the three-dimensional tomograms into rock matrix, pore space,
99 and fractures provided four-dimensional spatiotemporal information about the evolving pore
100 space and fracture network. The results provided insights into the deformation mechanisms at the
101 grain scale, and on how small-scale strain concentrations evolved preceding macroscopic failure,
102 confirming the results of previous studies of sandstones (Handin et al., 1963; Menéndez et al.,
103 1996; Wu et al., 2000; El Bied et al., 2002; Schubnel et al. 2007; Nasserli et al. 2014; Goodfellow
104 et al., 2015). In addition, digital volume correlation analysis enabled calculation of the three-
105 dimensional incremental strain fields between successive tomograms at a strain resolution one
106 order of magnitude higher than the imaging resolution. Information about the magnitude and
107 distribution of local volumetric and shear strains preceding failure was used to determine the
108 probability density distribution of the incremental strain magnitudes. Combining analysis of
109 segmented three-dimensional tomograms with DVC analysis improved our understanding of
110 deformation mechanisms preceding macroscopic failure by enabling quantification of the
111 evolving four-dimensional pore network and strain field. Post-failure scanning electron
112 microscopy (SEM) provided supplementary information concerning deformation and failure
113 mechanisms.

114 In the present study, we illustrate the potential of this emergent experimental technique
115 that can provide data with unprecedented spatial resolution, that is complementary to other
116 experimental techniques such as acoustic emissions analysis, and that can contribute to a better
117 understanding of deformation processes in rocks. Because this technique enables imaging of both
118 aseismic and seismic deformation and damage in the sample with unprecedented spatial
119 resolution, questions that existing experimental techniques can hardly address may be answered.
120 These include: What is the proportion and spatial distribution of shear relative to volumetric
121 strain events inside the rock prior to failure? What is the proportion of microfracture opening
122 relative to pore opening during dilation? How does the rate of aseismic and seismic damage
123 accumulation evolve as failure is approached?

124

125 **2. Background**

126 **2.1 Failure of intact rocks**

127 In rock deformation experiments, the evolution of a specimen is usually characterized
128 while either a constant strain rate or increasing stress is imposed at the boundaries. In true triaxial
129 compression tests, all three principle global strains or stresses, $\sigma_1 > \sigma_2 > \sigma_3$, are controlled. In the
130 experiments reported here, $\sigma_1 > \sigma_2 = \sigma_3 = P_c$, where P_c , is the confining pressure, and the
131 maximum principle stress, σ_1 , was increased from an initial value of $\sigma_1 = P_c$ at constant P_c , until
132 the sample failed. The sign convention that compressive stress is positive and compressive strain
133 (shortening or a decrease in volume) is positive, which is most commonly used in rock physics, is
134 adopted in this article.

135 Previous experiments indicate that, for confining pressures on the order of 10 MPa, tensile
136 microcracks nucleate and then undergo dilation, propagation and coalescence as the differential
137 stress increases, and thereby promote macroscopic failure (e.g., Peng and Johnson, 1972;
138 Tapponnier and Brace, 1976; Paterson and Wong, 2005). Early experiments suggested that
139 dilating microfractures produce macroscopic dilation of rock samples preceding failure (Brace et
140 al., 1966), and that a critical density of microfractures develops preceding macroscopic shear
141 failure (Scholz, 1968; Lockner et al., 1991). The spatial distribution of acoustic emissions
142 indicates that microfractures initially nucleate and grow at apparently random locations in
143 Westerly granite (Lockner et al., 1991). Reches and Lockner (1994) proposed that as a granite
144 approaches failure, microfractures form elongated arrays inclined at an angle of about 30°
145 relative to the direction of the maximum principle stress until they coalesce into a macroscopic
146 fault. Using high resolution two-dimensional image correlation analysis, Tal et al. (2016)
147 observed that both local compaction and local dilation occurs before failure in a Carrara marble.
148 Experiments on crystalline rocks such as granite, experiments on other brittle materials with pre-
149 existing fractures (e.g. PMMA), and numerical models suggest that macroscopic dilation occurs
150 through the development of microfractures that are dominated by tensile wing-cracks (Horii and
151 Nemat Nasser, 1986; Asbhy and Sammis, 1990; Kemeny and Cook, 1991). Laboratory
152 experiments on analog rock material that contain frictional flaws suggest that pre-existing
153 microfractures coalesce through the propagation and linkage of such tensile wing-cracks and
154 secondary shear fractures (e.g., Wong et al., 2001; Dresen and Guéguen, 2004). Experiments
155 have also documented how the geometries of pre-existing fractures control the coalescence

156 pattern of the resulting wing-cracks and shear fractures (e.g., Wong & Einstein, 2009). Discrete
157 element method models (Hazzard et al., 2000) and analytical and numerical damage models
158 (Asbhy and Sammis, 1990; Lyakhovsky et al., 1997; Girard et al., 2010) have produced
159 macroscopic fracture propagation via tensile crack development and long range elastic
160 interactions.

161 Analysis of time-lapse three-dimensional X-ray tomograms acquired during deformation
162 of a quartz monzonite rock specimen indicate that the total volume of microfractures, the rate of
163 damage accumulation, and the size of the largest microfracture all increase as power laws and
164 diverge with increasing differential stress as failure is approached (Renard et al. 2018). This
165 behavior suggests that fracture growth within low porosity crystalline rocks such as monzonite
166 evolves as a critical phenomenon in which an acceleration of damage accumulation precedes
167 system-size failure, confirming models developed in statistical physics (Dahmen et al., 2009;
168 Girard et al., 2010). In these experiments, most of the fractures formed within grains (i.e., by
169 transgranular fracturing), likely because the rock had a low initial porosity (<1%).

170

171 **2.2. Micromechanical models of sandstone deformation**

172 Several experimental and analytical studies have characterized the micromechanisms of
173 deformation in sandstones. With increasing effective stresses, the deformation of sandstones
174 transitions from brittle faulting to cataclastic processes (i.e. grain comminution) (Handin et al.,
175 1963; Wong et al., 1997). Depending on initial rock porosity, in the brittle regime dilation or
176 shear-enhanced compaction may initiate near a yield point, defined as the point at which a
177 significant deviation from linear elastic behavior occurs (point C' defined in Brace et al., 1966).
178 At higher effective confining pressures (between ~100 and 200 MPa), failure occurs through
179 grain crushing and pore collapse facilitated by microscopic fractures (Wong et al., 1997). At high
180 temperature (900 °C), the failure envelope may transition from a dilatant Mohr-Coulomb
181 relationship to a failure envelope with an elliptical shape as the confining stress increases (e.g.,
182 Kanaya and Hirth, 2018).

183 For sandstones with porosities above ~13%, deformation may be dominated by three
184 different mechanisms: shear localization, compaction localization or cataclastic deformation.
185 Compaction may dominate strain localization, depending on the loading path (e. g. Fortin et al.,
186 2009). Localized compaction bands may be formed in sandstones under high confining pressure

187 conditions (Fortin et al., 2009). For example, at an effective confining pressure of 10 MPa, strain
188 softening and brittle failure occurred in Bleurswiler sandstone specimens with porosities of
189 23.5% to 25.3% as the differential stress increased (Baud et al., 2015). At larger effective
190 confining pressures (30-50 MPa), shear-enhanced compaction and shear bands oriented at $\sim 30^\circ$ to
191 the maximum principle stress promoted macroscopic failure, and at even higher effective
192 confining pressures (70-90 MPa) compaction bands developed (Baud et al., 2015). At the grain
193 scale, fractures within grains and along grain contacts, as well as the collapse of high porosity
194 volumes produced the deformation bands.

195 Menéndez et al. (1996) conducted a series of experiments on Berea sandstone with 21%
196 porosity at a constant pore pressure of 10 MPa and confining pressures of 20, 50 and 260 MPa,
197 and characterized post-failure damage in thin sections prepared after failure. At low confining
198 stresses (< 50 MPa), the breakage of grain contacts produced the majority of the damage, and few
199 microfractures propagated through grains. As failure approached, pore collapse and transgranular
200 fractures provided a greater contribution to the overall damage. Tensile fracture along grain
201 contacts dominated the fracture of quartz grains, and shear localization occurred through the
202 coalescence of groups of microfractures. The few observed transgranular fractures led to the
203 conclusion that the development of tensile wing cracks did not play a significant role during the
204 initiation of failure (Menéndez et al., 1996).

205 During the deformation of a Gosford sandstone with grain size in the range 0.1-1 mm and
206 13% porosity, an acceleration of acoustic emissions as failure was approached was measured, as
207 well as a power-law distribution of acoustic emissions sizes (Cox and Meredith, 1993). A
208 relationship between these acoustic emissions parameters and crack dimensions was developed to
209 reconstruct the strain-stress curve and the weakening of the rock as failure was approached.

210 Zhang et al. (1990) developed a model for the micromechanics of grain crushing under
211 hydrostatic conditions based on the Hertzian contact concept. This model describes the failure of
212 porous rocks using the maximum tangential tensile stress at the edge of the contact area between
213 spherical grains, and linear elastic fracture mechanics assuming a high density of microscopic
214 crack-like flaws with a characteristic length at the grain surfaces (Johnson, 1982). This model
215 successfully described the failure envelopes of several sandstones (Baud et al., 2000).

216

217 **2.3. Deformation of Fontainebleau sandstone**

218 The relatively homogeneous mineralogy and microstructure of Fontainebleau sandstone at
219 scales greater than the grain size makes it an ideal target for deformation experiments. These
220 properties also make this sandstone an ideal candidate for three-dimensional X-ray
221 microtomography imaging studies, and so it was one of the first rocks imaged using this
222 technique (e.g., [Auzerais et al., 1996](#); [Coker et al., 1996](#)). Separation of the rock matrix from the
223 pore structure and fracture network by segmentation enabled the first quantitative analysis of the
224 topological and geometrical properties of the pore structure of a sandstone ([Lindquist et al.,](#)
225 [2000](#)), and led to important insights into the distribution of multiphase fluids in the pore space,
226 and modelling of flow and permeability ([Auzerais et al., 1996](#)). These analyses initiated the new
227 field of digital rock physics (e. g., [Andrä et al., 2013](#) and references therein).

228 Previous studies have analyzed how elastic properties and damage of this rock evolve
229 during deformation ([El-Bied et al., 2002](#); [Schubnel et al., 2007](#); [Nasseri et al., 2014](#); [Ghaffari et](#)
230 [al., 2014](#); [Goodfellow et al., 2015](#)). During triaxial deformation of a 14% porosity Fontainebleau
231 sandstone, [Schubnel et al. \(2007\)](#) recorded series of acoustic emission events preceding
232 macroscopic failure by attaching sensors directly to the rock sample. These recorded precursory
233 events reflect the acoustic energy released by nucleating and propagating microfractures. The
234 acoustic emissions highlighted distinct failure stages: 1) clustered acoustic emissions and strain
235 localization along an incipient fault plane, 2) lack of acoustic emissions within an aseismic
236 nucleation zone, 3) unstable rupture propagation within the previously aseismic, and perhaps
237 locked, zone, and 4) triggering of multiple sets of aftershocks, with the second set of aftershock
238 emitted from the rupture plane as the pore pressure rapidly dropped ([Schubnel et al., 2007](#)). The
239 increasing number of acoustic emissions prior to failure followed an inverse Omori law with a
240 power law time dependence with an exponent close to 1 ([Schubnel et al., 2007](#)). In another series
241 of experiments using a true triaxial stress apparatus with independent control of the three normal
242 stress magnitudes, the failure of a 4.52% porosity Fontainebleau sandstone included: 1) initial
243 compaction of the rock via crack closure, 2) macroscopic dilation of the sample due to
244 microfractures that opened perpendicular to the minimum stress direction, and 3) accumulation of
245 microfractures until macroscopic failure occurred ([Goodfellow et al., 2015](#)).

246 In the present study, we used X-ray microtomography and three-dimensional DVC
247 analysis to obtain quantitative information about the evolving microfracture networks and pore
248 structure throughout a series of triaxial compression experiments on three air-saturated

249 Fontainebleau sandstone cores. The X-ray attenuation contrast between air and quartz allowed the
250 evolving pore volumes and open microfractures to be distinguished from the rock matrix, thus
251 enabling investigation of deformation via image segmentation ([Videos S1 and S2](#)). Three-
252 dimensional digital volume correlation analysis of tomograms can identify diffuse deformation
253 zones, as well as fractures that do not dilate sufficiently to locally decrease the X-ray attenuation
254 ([Videos S3, S4 S5](#)). The main goal of this work is to analyze evolving porosity, and transgranular
255 and intergranular fractures using segmentation of dynamic X-ray tomograms and digital volume
256 correlation. The results provide new experimental observations of the micromechanisms of
257 deformation and strain localization in sandstone. The results confirm those obtained in previous
258 studies on Fontainebleau sandstone and other porous sandstones ([Handin et al., 1963](#); [Menéndez](#)
259 [et al., 1996](#); [Wu et al., 2000](#); [El Bied et al., 2002](#); [Schubnel et al. 2007](#); [Nasseri et al. 2014](#);
260 [Goodfellow et al., 2015](#)) and new information was also obtained.

261

262 **3. Methods and Material**

263 **3.1. X-ray transparent triaxial deformation apparatus**

264 The samples were deformed in the X-ray transparent HADES triaxial apparatus ([Renard](#)
265 [et al., 2016, 2017](#)), installed on the X-ray microtomography beamline ID19 at the European
266 Synchrotron Radiation Facility. This apparatus enables time-lapse imaging of the sample during
267 compressive deformation. With the full white beam of the beamline with X-ray energies up to
268 200 keV, the average energy of X-rays that cross the sample is close to 120 keV after the
269 attenuation of X-rays by the wall of the triaxial rig. Acquisition of a three-dimensional data set of
270 two-dimensional radiographs required about 1.5 minutes. The experiments were performed at
271 room temperature (24°C) on dry cylindrical specimens of 10 mm in length and 5 mm in diameter,
272 corresponding to ~50 grains in the axial direction and ~20 grains in the radial direction. The
273 voxel size was 6.5 μm. The table in [Figure 1](#) describes the imposed loading conditions used in
274 these experiments.

275 The specimens were installed in the rig between two stainless steel pistons. The lower
276 piston was immobile and the axial load was imposed on the specimen by displacement of the
277 upper piston. The interfaces between the rock sample and the pistons were not lubricated. Two
278 independent pumps controlled the axial load and confining pressure. In most laboratory
279 experiments on the deformation and failure of rocks, a constant strain rate is imposed as principal

280 stresses and other quantities of interest, such as acoustic emissions, are measured. This approach
281 cannot be used with X-ray tomography because the resulting tomogram would capture average
282 density contrast data as the specimens deformed over the data acquisition time interval, or 1.5
283 minutes in these experiments. Instead, in order to capture snapshots of deformation, the stress is
284 increased in small steps and X-ray attenuation data is acquired while the strain or stress is held
285 constant. In principle, the structure of the specimen might change during the acquisition time
286 under constant loading conditions if processes such as creep or subcritical fracture propagation
287 occur. However, in these experiments, we did not find evidence for significant changes in
288 structure during data acquisition, such as blurring of the distinct edges between void space and
289 quartz in the tomograms. For other materials or under different conditions, the mechanical
290 behavior may be different, and unacceptable image blurring could occur.

291 Three experiments were conducted by increasing the differential stress (the difference
292 between the axial stress, σ_1 , and the confining pressure) in steps of 2 or 5 MPa far from failure,
293 and steps of 0.5 or 1 MPa close to failure. A jacket made of Viton® fluoropolymer elastomer
294 encased each rock sample, and silicone oil applied the confining pressure to this jacket. In
295 experiment F1, instability of the confining pressure pump produced undulations in the volumetric
296 strain curve (Figure 1). In this experiment, the confining pressure was held at 20 MPa until the
297 axial stress reached 199 MPa, which closely approached the maximum permissible axial stress of
298 the HADES rig (200 MPa). Under these loading conditions, the sandstone core had not
299 macroscopically failed, and so the differential stress was then increased by reducing the confining
300 pressure in steps of 1 MPa from 20 MPa to 14 MPa, at which failure occurred. In this experiment,
301 failure occurred at a differential stress between 185 MPa and 186 MPa. Experiments F2 and F3
302 were conducted with a constant confining pressure of 10 MPa, and the samples failed before the
303 axial loading reached the limit of the HADES rig.

304 After each differential stress increase, three-dimensional X-ray micro-tomography
305 imaging of the specimen was performed. After failure, both the axial stress and confining
306 pressure was decreased, allowing voids to relax and open, and a final imaging step was
307 performed. Each X-ray imaging acquisition required 1.5 minutes and each stress increase
308 between scans required 1 minute.

309 Between 49 and 184 data sets, each consisting of 1600 radiographs, were acquired during
310 each deformation experiment, with a total of 288 for the three experiments. From the two-

311 dimensional radiographs, three-dimensional volumes of the specimens (1600x1600x1600 voxels)
312 were reconstructed in 16-bit grayscale using a phase contrast retrieval algorithm (Mirone et al.,
313 2014). During the reconstruction, filters were applied to remove ring artefacts and other spurious
314 patterns, and to correct for the fluctuation of the X-ray source intensity. The grayscale value of
315 each voxel in the three-dimensional volume is proportional to the X-ray attenuation coefficient,
316 itself proportional to local density. Low gray levels (dark shade of gray) correspond to low
317 attenuating materials (i.e., air), and high gray levels (light shades of gray) correspond to highly
318 attenuating materials (i.e., quartz), and intermediate gray level correspond to voxels that contain
319 both air and solid (i.e. voxels that are intersected by void boundaries).

320 In experiments with strain rate loading conditions, the differential stress typically reaches
321 a maximum and then decreases if the confining pressure is not too large. Under stress loading
322 conditions, similar to those used in our experiments, macroscopic failure typically occurs within
323 one stress increment step in brittle rocks. Failure is associated with a macroscopic stress drop. In
324 our experiments, macroscopic failure pulverized the sandstones, producing sand-like material, as
325 well as discrete core-spanning fractures within one stress increment step, and so strain softening
326 following macroscopic failure could not be investigated. The stress-strain relationship and the
327 micromechanical processes that occur prior to failure do not strongly depend on whether the
328 imposed loading are strains or stresses on the time scales of our experiments. Furthermore, our
329 stress loading conditions provided a rich data set of coalescing fractures prior to macroscopic
330 failure.

331

332 **3.2 Macroscopic stress and strain**

333 The three-dimensional tomograms were used to calculate the axial strain, radial strain and
334 volumetric strain curves prior to failure (Figures 1, S1). The macroscopic axial strain was
335 calculated from the distance between the two pistons visible in the tomograms at two locations in
336 an axial plane perpendicular to the piston faces. The average value of these two distances was
337 taken as the height of the sample. The macroscopic radial strain (inset, Figure S1) was
338 determined by measuring the lengths of two mutually perpendicular transects within horizontal
339 cross sections that intersected the vertical axis at heights of 1/3, 1/2, and 2/3 of the sample height,
340 providing a total of six measurements of the sample diameter, from which the mean value of
341 radial strain was calculated (Figure S1b). The volumetric strain was calculated from the average

342 height and average radius of the sample during deformation, assuming a cylindrical shape (Figure
343 1).

344 Because of the friction between the ends of the sandstone specimens, we expected that the
345 sandstone specimens would have a slight barrel-like shape under imposed differential stresses,
346 and this was observed (Figure 4a). So, the volumetric dilatational strain will be somewhat smaller
347 than the volumetric strain calculated with our method that uses core diameters at least 3 mm from
348 the piston-sandstone interfaces. The resolution (and the error) of this macroscopic strain
349 measurement was $10^{-4} l_0$ for the axial strain and $\sim 3 \times 10^{-4} r_0$ for the axial and radial strains, where
350 l_0 and r_0 are the initial length and radius. Consequently, the resolution for the volumetric strain
351 was $\sim 5 \times 10^{-4} V_0$, where V_0 is the initial volume. The resolution was higher for the axial strain
352 because there was a higher X-ray attenuation contrast between the pistons and the sample, which
353 controlled the axial strain measurement, than between the jacket and the sample, which controlled
354 the radial strain measurement.

355

356 3.3 Segmentation procedure and microscopy imaging

357 To determine the pore and fracture sizes and shapes from the tomograms (Figure 2b), the
358 following procedure was applied using the software AvizoFire®: 1) denoising of the three-
359 dimensional volumes with a non-local mean filter (Buades et al., 2005); 2) application of a mask
360 to remove the jacket, pistons, and confining oil around the rock sample; 3) application of a non-
361 local median filter to sharpen the boundaries between intact rock material and voids (pore space
362 or fractures); and 4) thresholding the data to partition the voxels occupied primarily by air from
363 those occupied primarily by quartz (Figure 2a).

364 Representative histograms from a three-dimensional data set (experiment F3) show how
365 thresholding can differentiate between voids and the rock matrix, which is composed primarily of
366 quartz (Figure 2). The gray scale values of the tomograms correspond to the X-ray attenuation
367 produced by materials, which is lower for air than for quartz grains. Both the original and filtered
368 histograms show two peaks corresponding to the voids (low X-ray attenuation) and the quartz
369 grains (high X-ray attenuation). The local minimum in the histogram of the filtered data was used
370 to separate the voids from the grains (red markers in Figure 2). We also tested segmentation of
371 the data using the mid-point between the two peaks and the results were similar to those using the
372 minima. We conclude that at least two criteria could be used to select a threshold gray value to

373 separate voids and grains. Here, we chose the minimum value between the two peaks of the
374 histogram to segment the data.

375 Filtering the tomograms reduced the noise and enhanced the contrast between pores and
376 grains, but it produced only small differences in the histogram and minimum. Thresholding with
377 the histogram minimum enabled extraction of pores and major microfractures from the three-
378 dimensional volumes. For the complete series of tomograms acquired during each experiment,
379 the same threshold value was used to segment the data. However, this thresholding technique did
380 not unambiguously capture micro-cracks with apertures less than or approximately equal to the
381 imaging resolution, although they could be identified by inspection of the tomograms by eye.

382 Some parts of the specimens were recovered in their jackets at the end of experiments F1
383 and F2 after unmounting them from the HADES rig. For experiment F2, almost the entire
384 specimen could be recovered, but specimen F3 was mostly reduced to quartz particles. The
385 specimens from experiments F1 and F2 were impregnated with liquid epoxy resin and then cut
386 along the axial plane after the liquid epoxy had formed a cross-linked polymeric solid. The
387 exposed surface was polished, coated with 10 nanometers gold, and imaged using a Hitachi
388 SU5000 scanning electron microscope at the University of Oslo with a voltage of 15 kV.

389

390 **3.4 Digital volume correlation analysis**

391 Three-dimensional digital volume correlation (DVC) analysis was initially developed for
392 applications in mechanics and engineering (e.g. Bay et al., 1999 and references therein). DVC
393 analysis finds the translations and rotations that best map sub-volumes within a three-dimensional
394 data set onto sub-volumes in another three-dimensional data set by identifying similar patterns
395 within those sub-volumes. This technique has been used to study the development of shear bands
396 in soils (Viggiani et al., 2004), and the compaction of a Rothbach sandstone specimen with a
397 strain resolution of 10^{-3} (Louis et al., 2007). We used the open source DVC analysis software
398 TomoWarp2 for our DVC analyses (Hall et al., 2010; Tudisco et al., 2015, 2017).

399 DVC analysis is based on finding the displacement field that maximizes the correlation
400 between voxel intensity subsets (sub-volumes) within pairs of sequential tomograms.
401 Interpolation methods enable sub-voxel scale displacement resolution to be obtained. However, if
402 there is little variation in X-ray attenuation coefficients within the sub-volumes, DVC analysis
403 may lead to unreliable incremental displacement fields. For example, there is little or no variation

404 in the X-ray attenuation coefficients within individual quartz grains, except perhaps lower density
 405 fluid inclusions. Consequently, the parameters of DVC analyses must be tuned to capture
 406 displacement fields that include sub-volumes that include several grains or grain boundaries, or
 407 other contrasts in the X-ray attenuation coefficient fields.

408 By identifying similar patterns across successive volumes, digital volume correlation
 409 produces three-dimensional displacement fields from which the six independent components of
 410 the second rank three-dimensional strain tensor may be calculated. These incremental strain fields
 411 reveal strain localization that occurred within the time interval between the acquisitions of the
 412 pair of microtomograms. The cumulative strain from the onset of loading could also be
 413 determined. Following the approach of [McBeck et al. \(2018\)](#), we calculated three-dimensional
 414 incremental displacement fields between pairs of three-dimensional tomograms corresponding to
 415 approximately constant increments in the macroscopic axial strain, ε_{zz}^M , throughout each
 416 experiment. We calculated ε_{zz}^M from the change in axial length of the sandstone specimen relative
 417 to the initial length l_0 , as described in [Section 3.2](#). In addition, to investigate in more minute
 418 detail the dominant failure modes near the onset of yielding in experiment F2, we used DVC
 419 analysis to calculate the incremental strain fields surrounding the yield point, with the highest
 420 possible temporal (i.e., differential stress) resolution.

421 We calculate changes in the local volumetric and shear strain fields using the first
 422 invariant of the incremental strain tensor, $I_1(\Delta \boldsymbol{\varepsilon}) = \Delta \varepsilon_{xx} + \Delta \varepsilon_{yy} + \Delta \varepsilon_{zz}$, and the second
 423 invariant of the incremental strain tensor, $I_2(\Delta \boldsymbol{\varepsilon}) = (\Delta \varepsilon_{xy})^2 + (\Delta \varepsilon_{xz})^2 + (\Delta \varepsilon_{yz})^2 -$
 424 $(\Delta \varepsilon_{xx}\Delta \varepsilon_{yy} + \Delta \varepsilon_{xx}\Delta \varepsilon_{zz} + \Delta \varepsilon_{yy}\Delta \varepsilon_{zz})$, where $\Delta \varepsilon_{ij}$ is a component of the local incremental strain
 425 tensor, i.e. the local strain calculated by comparing two successive tomograms in the DVC
 426 analysis. In this coordinate system, the z-axis is vertical (parallel to σ_1) and x-y plane is
 427 horizontal (perpendicular to σ_1). $I_1(\Delta \boldsymbol{\varepsilon})$ provides information about volumetric strain in
 428 coordinate systems independent of the principle axis system. To characterize the incremental
 429 shear strain, we calculated the second invariant of the incremental deviatoric strain, $J_2(\Delta \boldsymbol{\varepsilon})$.
 430 Since $(3J_2(\boldsymbol{\varepsilon}))^{\frac{1}{2}}$ is the von Mises yield criterion equivalent strain, we refer to $(3J_2(\Delta \boldsymbol{\varepsilon}))^{\frac{1}{2}}$ as the
 431 von Mises incremental strain. The second invariant of the of the incremental deviatoric strain,
 432 $J_2(\Delta \boldsymbol{\varepsilon})$, is related to the first invariant $I_2(\Delta \boldsymbol{\varepsilon})$, and the second invariant, $I_2(\Delta \boldsymbol{\varepsilon})$, of the
 433 incremental strain tensor by $J_2(\Delta \boldsymbol{\varepsilon}) = \frac{1}{3}(I_1(\Delta \boldsymbol{\varepsilon}))^2 - I_2(\Delta \boldsymbol{\varepsilon})$. To be able to compare the values

434 of $I_1(\Delta \boldsymbol{\epsilon})$ (first order in the strain) and $J_2(\Delta \boldsymbol{\epsilon})$ (second order in the strain), we report in the
435 following the values of $(3J_2(\Delta \boldsymbol{\epsilon}))^{1/2}$. In the adopted sign convention, negative values of $I_1(\Delta \boldsymbol{\epsilon})$
436 indicate volumetric dilation, and positive values of $I_1(\Delta \boldsymbol{\epsilon})$ indicate volumetric contraction.

437 The windows used to perform the DVC correlations had a cubic shape with four faces
438 parallel to the main compressive stress σ_1 and two faces perpendicular to it. Following tests on
439 the influence of the correlation window size and node spacing size on the resolution, computation
440 time, and robustness of the resulting displacement fields, we selected a correlation window size
441 of 10 voxels (65 μm) and node spacing size of 20 voxels (130 μm). This choice ensured that each
442 window contained at least a sub-volume of one grain and a sub-volume of one pore, maximizing
443 the contrast for volume correlation. To characterize the resolution of strain values obtained from
444 DVC analysis in these experiments, and thus the lower limit of robust strain values, we
445 performed DVC analysis on: 1) the same tomogram (i.e., autocorrelation), 2) two tomograms that
446 were acquired at the same differential stress, 3) two tomograms that were separated by 1 MPa of
447 differential stress (131 MPa and 132 MPa), and 4) two tomograms that were separated by 36 MPa
448 of differential stress (112 MPa and 148 MPa) (Figure 3a).

449 The resulting strain populations show that the autocorrelation (test 1) produced smaller
450 strain magnitudes than the other tests, as expected (Figure 3b, 3c). The non-zero displacement
451 field produced in this autocorrelation calculation arises from interpolation between integer (pixel)
452 displacements in the sub-pixel resolution method used in the TomoWarp2 software. The strain
453 populations of test 2 and test 3 were similar to each other, indicating that a differential stress
454 increase of 1 MPa did not produce significant deformation of the sandstone in this increment of
455 the experiment. The larger strain magnitudes produced in test 4 indicate that, as expected, more
456 strain accumulated during the 36 MPa of differential stress increase than during the 1 MPa of
457 differential stress increase of test 3.

458 The incremental strain populations produced in the autocorrelation test (test 1) suggest a
459 characteristic error in the calculated strains (Figure 3b, 3c). Twice the standard deviation of the
460 $I_1(\Delta \boldsymbol{\epsilon})$. and $I_2(\Delta \boldsymbol{\epsilon})$. populations produced in test 2 were used as the thresholds for the
461 populations of these two invariants. These thresholds are more conservative than those produced
462 in the autocorrelation of test 1, and so produce strain populations with lower ratios of signal to
463 noise. Invariants with magnitudes below these thresholds, which are on the order of 10^{-3} for
464 $I_1(\Delta \boldsymbol{\epsilon})$ and $I_2(\Delta \boldsymbol{\epsilon})$, were removed from the incremental strain fields.

465

466 **3.5 Fontainebleau sandstone samples**

467 Fontainebleau sandstone is a quartz arenite of Oligocene age outcropping around
468 Fontainebleau city near Paris, France (Bourbie and Zinszner, 1985). This sandstone is considered
469 an ideal reservoir rock because it has a relatively homogeneous mineralogical composition
470 (>99% quartz), well-sorted grain size, an average diameter of 0.25 mm, and a wide range of
471 porosities (3-30%) depending of the degree of quartz cementation. As expected for a clean
472 sandstone with a characteristic grain diameter of 0.25 mm, the high porosity specimens have a
473 high permeability (Bourbie and Zinszner, 1985). In specimens with porosities higher than 5%,
474 most of the pores are connected in three-dimensions (Fredrich et al., 1993). For samples with
475 porosities smaller than 4%, the pore space is less connected, and so the permeability is at least
476 one order of magnitude lower than that of samples with higher porosities (Fredrich et al., 1993).
477 In addition, the P-wave velocity in this sandstone decreases almost linearly from 5500 m·s⁻¹ to
478 3000 m·s⁻¹ as the porosity increases from 3% to 30% (Bourbie and Zinszner, 1985).

479 In the present study, three specimens of diameter 5 mm and length 10 mm were cored
480 perpendicular to bedding from a single 10x10x10 cm Fontainebleau sandstone block with a
481 matrix made of quartz and a minor amount of iron oxides (<1%). The mean grain size was 0.25
482 mm. The porosity derived from the initial three-dimensional data sets following segmentation
483 was in the range 5.5-7%. The porosity was also measured by weighing ten dry specimens before
484 and after imbibition with water, and a value of 6 ±1% (standard deviation) was obtained,
485 consistent with the porosity measured using X-ray tomography. Three-dimensional imaging
486 indicates that the initial pore structure was almost entirely connected in three dimensions. There
487 was almost no microporosity in these specimens (i.e., porosity within grains), except for a few
488 micrometer-size fluid inclusions in the quartz grains that were not connected to the bulk porosity.

489

490 **4. Results**

491 **4.1 Macroscopic and microscopic deformation**

492 In each experiment, the macroscopic axial strain increased faster than a linear trend (i.e. super-
493 linearly) relative to the applied stress until the differential stress reached about 20 MPa. This
494 initial nonlinear phase arose from the closure of the weakest preexisting microfractures and
495 pores, grain reorientation and the deformation of weak intergrain contacts. In experiments on

496 Mount Scott granite, [Katz and Reches \(2004\)](#) observed inconsistencies in the initial, low
497 differential stress behavior (an increase in the deformation modulus, $\partial\sigma_D/\varepsilon_M|_{P_c}$) with increasing
498 strain for some specimens and a decrease in the deformation modulus for others. Consequently,
499 we cannot conclude that the initial softening ($\partial^2\sigma_D/\partial\varepsilon_M^2|_{P_c} < 0$) is generic for low porosity
500 Fontainebleau sandstone under a confining stress of 10 MPa. In addition, the closure of
501 microfractures and pores may cause hardening rather than softening. However, the initial
502 softening was more pronounced in our experiments than those of [Katz and Reches \(2004\)](#). We
503 did not investigate these initial mechanisms in detail because the focus of this work was on the
504 damage preceding macroscopic failure.

505 After the initial nonlinear phase, the macroscopic mechanical behavior transitioned
506 gradually from a quasi-linear phase at intermediate differential stresses (20-80 MPa), to
507 significant deviation from quasi-linear behavior associated with significant dilation, and finally to
508 macroscopic failure ([Figure 1](#)). We chose to define the yield points as the point at which the
509 measured volumetric strain differed by 3% from the strain predicted by linear regression of the
510 mean stress-strain data over the range $20 \leq \sigma_D \leq 80$ MPa (also named point C' in [Brace et al.](#)
511 [1966](#)). After the yield point was reached, the relationship between differential stress and axial
512 strain became increasingly nonlinear until macroscopic failure occurred. The yield point
513 corresponded to an axial strain close to 0.08 for experiments F1 and F3 and to 0.04 for
514 experiment F2 where a fault localized near the yield point. The volumetric strain curves revealed
515 that the sample compacted (volumetric strain increased) preceding the yield point, and then began
516 to dilate (volumetric strain decreased) near the yield point in each experiment ([Figure 1](#)).
517 Following yielding and preceding macroscopic failure, microfractures propagated through grains
518 as well as along grain boundaries ([Figures 2b, 4a, 5a-c, 6c, 7, Videos S1 and S2](#)).

519 Consistent with the similar stress-strain curves of experiments F1 and F3, the evolving
520 microfracture distributions were also similar in these experiments. In particular, damage
521 remained diffuse and distributed throughout much of each experiment ([Figure 4a](#)), and only
522 concentrated into a narrower zone close to failure (~95% of the failure stress), ([Video S2](#)). In
523 contrast, in experiment F2, damage localized early (~75% of the failure stress) along a narrow
524 planar zone that evolved into a fault ([Figure 5, Video S1](#)). These differing behaviors are also
525 revealed in the spatial distribution of high strains from the DVC analyses. The incremental strains
526 above the 95th percentile of each strain population were more spatially diffuse in experiments F1

527 (Video S3) and F3 (Video S5), than in experiment F2 (Video S4). Despite these differences in
528 strain localization preceding failure, the development of conical faults, in which grain
529 comminution and porosity reduction occurred, ultimately caused the macroscopic failure of each
530 sandstone sample (Figures 4b-d, 5d-f). Dilatational shear failure was the main mechanism of
531 faulting, and significant compaction, grain comminution, and porosity reduction was observed in
532 the final fault zone. The fractures developed into conical shapes because the pistons of the rig
533 were not lubricated. This lack of lubrication produced frictional resistance to lateral movement of
534 the sandstone in contact with the pistons, which localized shear strain and so promoted fracture
535 nucleation near the edges of the top and bottom sandstone-piston interfaces (e. g. Peng and
536 Johnson, 1972). Consequently, the boundary condition at the pistons may have controlled the
537 final angle of the faults at failure.

538 In all the experiments, more than 99% of the voids, including the pores and fractures,
539 were connected in three dimensions (Figure 6b), both preceding and following faulting (Figure
540 5). In experiment F2, the gray levels (X-ray attenuation coefficients) of the fractures were slightly
541 higher than those of the pores, enabling segmentation of the fractures from the pore space (Figure
542 6). This segmentation revealed that a core-spanning fracture developed through the linkage of
543 sub-vertical, dilating microcracks (Figure 6b-c). The core spanning fracture did not immediately
544 result in macroscopic failure denoted by a reduction in the axial stress. This segmentation also
545 enabled separation of the contributions to the total porosity increase from the growth of pre-
546 existing pores, and the propagation and opening of new fractures (Figure 6a).

547 Observations of microstructures at the grain scale using scanning electron microscopy
548 (SEM) and visual inspection of the tomograms at the micrometer scale enabled various
549 deformation mechanisms to be differentiated, including intergranular fracturing (i.e. grain
550 boundary opening), transgranular fracturing, and grain comminution (Figure 7). Each of the
551 sandstone cores failed macroscopically through the development of conjugate faults (Figures 4b,
552 5d, 7a). Cracks propagated both within the quartz grains (i.e., transgranular fractures) (Figure 7b,
553 7d), and between grains (i.e., intergranular fractures) (Figure 7c). In some cases, these cracks
554 stopped at a pore interface. In experiment F2, the cracks self-organized into an incipient fault in
555 which shear displacements could be observed (Figure 7 g, h, i).

556 In summary, the three samples have similar macroscopic stress-strain relationships
557 characterized by: 1) initial macroscopic compaction arising from the closing of voids, 2)

558 macroscopic dilation arising from microfracture development and pore opening, 3) transgranular
559 and intergranular propagation of fractures that drives the strain-stress relationship from linear to
560 nonlinear, and 4) macroscopic shear failure due to the coalescence of microscopic fractures.
561 These macroscopic behaviors arose from microscopic deformation processes that the four-
562 dimensional strain tensors revealed. In experiments F1 and F3, damage localized into a narrow
563 deformation zone near macroscopic failure. In contrast, in experiment F2, damage began to
564 localize into a narrow zone near the macroscopic yield point, and the core-spanning fault grew
565 slowly enough to be captured in several tomograms preceding macroscopic failure.

566

567 **4.2 Global evolution of damage toward failure**

568 To characterize the evolution of cumulative damage toward failure, we extracted the voids
569 from the quartz grains in each tomogram. Segmentation into rock matrix and voids (pores and
570 fractures), provided the volume fraction of voids as a function of increasing differential stress
571 (Figures 6, 8, S2). To quantify the accumulation of damage, including opening pores and
572 propagating fractures, Renard et al. (2018) employed a damage index, $D_\phi = \frac{\phi - \phi_i}{1 - \phi_i}$, where ϕ_i is
573 the initial void fraction of the sample under the initial confining pressure, preceding axial
574 loading, and ϕ is the void fraction measured at a given differential stress. In this approach, the
575 normalized distance to failure in stress space is defined as $\Delta = \left(\frac{\sigma_f - \sigma}{\sigma_f} \right)$, where σ_f is the
576 differential stress at which failure occurred, and σ is the differential stress when the tomogram
577 was acquired. When $\Delta = 0$, the rock is at failure, and when $\Delta = 1$, no differential stress is applied
578 to the sample. Renard et al. (2018) applied this normalization to show that damage accelerates as
579 a power law in quartz monzonite ($\partial D_\phi / \partial \sigma \sim \Delta^{-\beta}$), in agreement with mechanical models that
580 consider failure as a critical phenomenon (Dahmen et al., 2009; Girard et al., 2010). This
581 behavior suggests that failure is a critical phenomenon in crystalline rocks such as monzonite.

582 However, in our sandstone experiments, D_ϕ did not increase as a negative power of Δ as
583 failure was approached. In these experiments, the total porosity, ϕ , and the damage index, D_ϕ ,
584 remained near their initial values until the differential stress was several MPa smaller than the
585 differential stress at yield point, and then changed rapidly until failure occurred (Figure 8a). This
586 acceleration of D_ϕ could not be fitted by a power law or an exponential relationship (Figure 8b),
587 and so this relationship differs from the power-law acceleration observed for the crystalline

588 quartz monzonite (Renard et al., 2018). Due to the instability of the confining pressure pump in
589 experiment F1, we focus on the evolving damage index in experiments F2 and F3 (Figure 8).
590 Figure S2 shows the results from all three experiments. We interpret the absence of power law
591 behavior as the pinning of microfractures when they reach pore walls: the presence of pores
592 screens the stress concentration at the fracture tips, which may reduce the long-range elastic
593 interactions necessary to develop a power-law increase of damage as failure is approached
594 (Dahmen et al., 2009; Girard et al., 2010).

595

596 4.3 Evolving statistics of incremental strains

597 To further characterize the evolution of microscopic damage, we performed DVC analysis
598 of sequential pairs of tomograms in each experiment. The resulting series of three-dimensional
599 incremental strain tensor fields, normalized by division by the incremental macroscopic axial
600 strain, $\Delta\varepsilon_{zz}^M$, enabled quantitative assessment of evolving volumetric and shear strain localization
601 (Figure 9). The normalization by $\Delta\varepsilon_{zz}^M$ was performed because the macroscopic strain increase
602 between pairs of tomograms was not constant (Figure 10a, b, g). The top 5% of the negative
603 $\frac{\Delta I_1(\Delta\varepsilon)}{\Delta\varepsilon_{zz}^M}$ (dilatational), positive $\frac{\Delta I_1(\Delta\varepsilon)}{\Delta\varepsilon_{zz}^M}$ (contractive), and $\frac{(3\Delta J_2(\Delta\varepsilon))^{1/2}}{\Delta\varepsilon_{zz}^M}$ (shear) populations in five
604 differential stress increments of each experiment (Figure 9, Videos S3-5) revealed the
605 localization of the core-spanning fracture observed in experiment F2 (Figures 5a-c, 6b-d, Video
606 S2). While segmentation of the tomograms provided information about the evolving dilatational,
607 contractional and shear strain, pore volumes, fractures and their connectivity, DVC analysis
608 provided displacement fields from which strain fields were calculated with a strain resolution
609 close to 10^{-3} for these scans.

610 We calculated the incremental strain fields between pairs of three-dimensional data sets
611 that were separated by an approximately constant change in the macroscopic axial strain, $\Delta\varepsilon_{zz}^M$.
612 For each experiment, ten incremental strain fields that encompassed each complete experiment
613 were calculated (Figure 10). To track the interplay between non-deviatoric and deviatoric strains,
614 we report histograms of the first invariant of the incremental strain, $I_1(\Delta\varepsilon)$, divided by $\Delta\varepsilon_{zz}^M$, and
615 the square-root of the Von Mises incremental strain, $(3J_2(\Delta\varepsilon))^{1/2}$, divided by $\Delta\varepsilon_{zz}^M$ (Figure 10).

616 Because the incremental normalized strain invariants, $\frac{I_1(\Delta\varepsilon)}{\Delta\varepsilon_{zz}^M}$ and $\frac{(3J_2(\Delta\varepsilon))^{1/2}}{\Delta\varepsilon_{zz}^M}$ were calculated at
617 many locations (>50,000 points per tomogram pair), they provided additional information about

618 deformation within the sandstone specimens that global measures such as porosity, macroscopic
 619 axial and radial strains, and the damage index do not capture. In addition, because $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ and
 620 $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ are measures of incremental strain between two tomogram acquisitions, their mean and
 621 median values may not systematically increase as failure is approached.

622 The evolution of the incremental normalized strain invariants were similar in experiments
 623 F2 and F3, but differed from that observed in experiment F1 (Figure 10). In experiments F2
 624 (Figure 10b) and F3 (Figure 10h), the area under the dilatational portion of the $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ histogram
 625 increased by more than 200% as the differential stress increased, while the area under the
 626 contractional portion did not increase as much. The evolution of $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ was similar to that of
 627 the contractional part of $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ in these experiments in that, with increasing differential stress, the
 628 $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ population included a higher proportion of higher magnitude values, increasing the
 629 area under the positive portion of the histogram. In contrast, in experiment F1, the trend of
 630 increasing $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ and $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ was not as consistent as in the other experiment. In particular, the
 631 second to last differential strain increment (yellow in Figure 10a-c), included more high
 632 normalized dilatational strain increments, $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$, than the final differential stress increment
 633 (orange in Figure 10a-c), and so there was a smaller area under the dilatational portion of the
 634 $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ curve. Similarly, the second to last differential strain increment produced a higher area
 635 under the $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ curve than the final differential strain increment, indicating a higher
 636 magnitude of overall shear strain (Figure 10c). The difference in behavior could be related to the
 637 difference of loading path since the confinement was decreased as failure was approached in
 638 experiment F1 but remained constant in experiments F2 and F3 (Figure 1). To summarize the
 639 results of Figure 10: for samples F1 and F3, most of the strain in the sample was accommodated
 640 by local dilation events that we relate to the opening of grain boundaries. For sample F2, the road
 641 to failure was due to a combination of dilation and shear as a fault zone developed.

642 To synthesize these changes in the overall magnitude of each incremental strain field, we
 643 summed the dilatational (negative) and contractive (positive) portions of the $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ populations,

644 and the $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ populations. These sums document the total normalized strain increments in
 645 the sample during loading. We report these sums normalized by the sum calculated for each
 646 population in the first differential stress increment (Figure 11a-c). Figure 11 shows that the
 647 relationships between the sums of the incremental contractions, incremental dilations and von
 648 Mises incremental stresses and the macroscopic axial contraction was complex. The trends in
 649 experiments F1 and F3 were more similar to each other than they were to experiment F2, as
 650 might be expected from the early yielding in experiment F2. In all three experiments, the
 651 dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ sum began to increase when the axial contraction reached about 6×10^{-3} .
 652 However, in experiments F1 and F3, the dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ sum reached a maximum after the yield
 653 point had been reached and decreased before failure occurred whereas in experiment F2 the yield
 654 point was reached before the dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ sum began to increase, and there was no maximum
 655 before failure. The sums of $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ in experiments F1 and F3 did not change much and stayed
 656 around a value of 1. In contrast, in experiment F2, where dilatational and shear strain localized
 657 into a core-spanning fracture, the $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ sum increased by about a factor two.

658 To track the overall strain increments we extracted the number of local incremental strain
 659 values above a threshold (Figure 11d-f), and then normalized by the sum calculated for each
 660 population in the first differential stress increment. The evolution of this quantity is similar to that
 661 of the sum of the increments shown in Figure 11a-c.

662 The evolution of the spatial distributions of the normalized incremental strain invariants
 663 (Figure 9) illuminates the morphology of the coalescing microfractures that produced the trends
 664 observed in the histograms and sums. Consistent with the evolution of the contractive values of
 665 $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ observed in the histograms and sums, high values of contractive $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ did not localize
 666 within or around the fracture. This lack of localization of the contractive $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ values produced
 667 only small changes in the sums and histogram shape, whereas the opening of and slip along
 668 fractures produced fracture localized dilation and shear strain, thus increasing the sums and areas
 669 under the dilatational portion of the $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ histogram and the $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ histogram.

670 To track the interplay between dilatation, contraction and shear strain, we show the
671 dilative part of $I_1(\Delta\epsilon)$, the contractive part of $I_1(\Delta\epsilon)$, and $(3J_2(\Delta\epsilon))^{1/2}$, without normalization by
672 $\Delta\epsilon_{zz}^M$, as a function of each value (Figure 12). The curves outline pairs of strain values with a
673 bivariate kernel density >25% of the maximum kernel density. For the three specimens, these
674 data show that: 1) incremental dilation increased much more than incremental compaction as
675 failure was approached, 2) shear strain increased as failure was approached, and 3) the increase in
676 shear strain acted in concert with dilation and was not strongly correlated with contraction
677 (Figure 12).

678

679 5. Discussion

680 5.1 Micromechanical models of brittle failure

681 Micromechanical models including the wing-crack model, the pore-emanated crack
682 model, the Hertzian fracture concept, and the pore collapse model have linked grain-scale
683 microscopic processes to macroscopic strain in rocks (see Section 2.2). Pore collapse was not
684 observed in the tomograms of these experiments, and so the pore collapse model may not
685 adequately describe the failure of these Fontainebleau sandstone specimens. Development of
686 Mode I cracks and their dilation preceded the onset of strain localization (samples F1 and F3,
687 Figure 11a, 11c) or coincided with this localization (sample F2, Figure 11b), indicating that
688 relatively high magnitudes of dilation began to localize before high magnitudes of shear strain.
689 We found no evidence for wing crack formation. Wing cracks develop through localized shear
690 displacement that then produce regions of high tensile stresses, and so we would expect to
691 observe localized high shear strain regions before localized dilatational regions in the DVC data,
692 rather than the observed opposite trend. In addition, visual inspection of the tomograms, before
693 and after segmentation, provided no evidence for wing cracks, though it is possible that wing
694 cracks with apertures that were too small to detect were formed.

695 The pore-emanated crack model, in which cracks open along grain boundaries could be
696 consistent with the experimental observations of microscale strain due to opening of grain
697 boundaries (Figure 7e-f). However, the Hertzian fracture concept also matches the experimental
698 observations of the development of transgranular cracks that develop within grains, which the
699 pore-emanated crack model does not predict. The Hertzian fracture concept uses the maximum
700 tangential tensile stress within the contact area between spherical grains to predict grain-scale

701 failure that leads to macroscopic failure (Johnson, 1982). Consistent with this concept, we
702 observed that at small differential stresses, a higher number of intergranular fractures than
703 transgranular fractures developed. And then, as the differential stress increased, and particularly
704 after the yield stress was reached, transgranular fracturing began to dominate deformation until
705 extensive grain comminution occurred (Figures 4, 5). During early stage deformation,
706 intergranular fractures may preferentially develop instead of transgranular fractures because the
707 cement that binds sandstone grains may be weaker under tension than the intact grains.
708 Consequently, intergranular fractures may be able to propagate under lower differential stresses
709 than transgranular fractures. In addition, as the differential stress increases, the effect of the
710 difference in strength between the cement and the grains may diminish as preexisting pores close
711 and intergranular contacts are clamped shut under higher normal stresses.

712 The geometry and volume of the pore network play an important role in controlling the
713 deformation and failure of sandstones because the concentration of tensile stress near pore
714 surfaces initiates the growth of fractures. For a spherical cavity under a compressive uniaxial far
715 field stress embedded in homogeneous isotropic linear elastic material (e.g. Goodier, 1933;
716 Sadowsky and Sternberg, 1949; Eshelby, 1957; Mura, 1982), the stress at the surface of the
717 cavity depends only on the far field stress and the polar angle, θ , relative to the direction of σ_1 . As
718 this angle varies from $\pi/2$ at the equator of the pore to 0 or π at the poles, the resulting
719 compressive stress at the pore surface decreases and becomes tensile near the poles. Because
720 mineral grains and cement are weaker under tensile stress than under compressive stress,
721 fractures are expected to nucleate near the poles of a spherical cavity and propagate preferentially
722 along planes that are oriented sub-parallel to σ_1 . Shear fractures may nucleate at cavity walls at
723 orientations parallel to σ_1 , and then propagate at angles that are oblique to σ_1 (Davis et al., 2017).
724 These analytical and modelling approaches have been extended to plastic materials (Monchiet et
725 al., 2008), multiple voids (Tandon and Weng, 1986) and polygonal voids. However, exact results
726 cannot usually be obtained, and the effects of the necessary approximations are challenging to
727 assess. Our experiments provide additional insights because these three-dimensional data show
728 that pores have shapes that differ from the spherical geometry often assumed in numerical models
729 (Figure 7).

730 Numerical methods such as finite element models (e.g. Eggers et al., 2006;
731 Avazmohammadi and Naghdabadi, 2013) and boundary element models (Davis et al., 2017), as

732 well as micromechanical modelling coupled with analogue experiments (Sammis and Ashby,
733 1986), provide additional insights that complement analytical approximations. For example,
734 Nadimi et al. (2015) conducted two-dimensional finite element model simulations of the
735 compression of Fontainebleau sandstone. In these simulations, load-bearing columns developed
736 sub-parallel to the direction of the compressive stress, consistent with finite element simulations
737 performed by Nadimi et al. (2015), and a model of the failure of brittle porous solids proposed by
738 Sammis and Ashby (1986). In our experimental work, we also observed the formation of sub-
739 vertical (sub-axial) columns separated by sub-vertical fractures (Figures 5, 6, 7g-i). However, the
740 columns did not become well-developed until vertical strains were substantially larger than those
741 used in the simulations of Nadimi et al. (2015). The geometric difference between the two-
742 dimensional model and the three-dimensional experiment, the strength of the simulated cement
743 bonds, and the non-detection of fractures with small aperture widths in our X-ray
744 microtomography experiments may have produced this apparent discrepancy between the
745 experiments and the simulations. Features with characteristic scales below the resolution of X-ray
746 microtomography, such as flaws within pore surfaces, mineral grains and cement, play an
747 important role in the nucleation of microfractures.

748

749 **5.2 Dilation and acceleration of damage when approaching failure**

750 In our Fontainebleau sandstone specimens, the total damage, D_ϕ , accelerated toward
751 failure (Figure 8a) and occurred concomitantly with dilation. In a study of crystalline rock, the
752 damage accelerated as a power law of the normalized stress, $\Delta = \frac{\sigma - \sigma_f}{\sigma_f}$, where σ_f is the
753 differential stress at failure, such that $D_\phi \sim \frac{1}{\beta - 1} [\Delta^{-(\beta - 1)} - 1]$ with an exponent β between 1.4 and
754 1.8 (Figure 5 in Renard et al., 2018). In these experiments, the cumulated damage was measured
755 so that the power law exponent is $-(\beta - 1)$, where β is the exponent that characterized the power
756 law divergence of the rate at which the damage increases with increasing differential stress as
757 failure is approached. Based on two-dimensional simulations of uniaxial compression with no
758 confining pressure, Girard et al. (2010) found evidence for power law divergence of the damage
759 correlation length with decreasing Δ as Δ approached failure, thus suggesting that failure is a
760 critical phenomenon. This model of brittle failure indicates that elastic interactions between
761 developing fractures over long-range, system-size, distances can explain the power law

762 divergence of incremental damage as Δ decreases toward failure in low porosity crystalline rock
763 (Girard et al., 2010). A power law acceleration of acoustic emission prior to failure was also
764 observed in the deformation of heterogeneous materials (Vasseur et al., 2015).

765 In contrast, in our experiments on Fontainebleau sandstone, the acceleration of damage
766 occurred at a slower than power law rate (Figure 8b). This slower acceleration of damage may
767 occur in more porous rocks compared to crystalline rocks because when the propagating tip or
768 edge of a microfracture reaches a pore, the local stress concentrations at the fracture tip or edge
769 diminishes. This may prevent further propagation of the fracture until the local stress field
770 increases. Although the rate of damage accumulation differed in our recent monzonite
771 experiments (Renard et al., 2018) and these sandstone experiments, in all five experiments (three
772 sandstone and two monzonite experiments) we observed an acceleration of damage as failure was
773 approached. This acceleration of damage was a precursor to shear failure in our experiments, as
774 rock damage models (Lyakhovsky et al., 1997; Dahmen et al., 2009; Girard et al., 2010) and
775 acoustic emission experiments on sandstones (Cox and Meredith, 1993; Wu et al., 2000;
776 Schubnel et al., 2007; Fortin et al., 2009; Nasserri et al., 2014; Ghaffari et al., 2014; Goodfellow et
777 al., 2015) indicate. Our analysis reveals that this damage increase is slower than the power law
778 divergence predicted by some damage models (Figure 8). Furthermore, this analysis is the first to
779 separate this damage increase into new microfracture development and pore dilation (Figure 6a).
780 Segmentation of X-ray tomograms enabled this distinction because it captures aseismic and
781 seismic strain, whereas acoustic emissions record only seismic strain.

782 Proposed micromechanical models of failure of porous sedimentary rock depend on the
783 processes that produce opening-mode failure and dilation at the grain-scale. In particular, the
784 global evolution of D_ϕ likely differs between porous and crystalline rocks because of the
785 pervasive pore network in more porous rocks. Macroscopic dilation has been observed during the
786 deformation of low porosity crystalline rocks (e.g., Brace, 1978) and porous sedimentary rocks
787 (e.g., Baud et al., 2000). However, the degree of dilation in porous sedimentary rocks differs
788 from that of crystalline rocks because in porous rocks, microfractures may nucleate at grain
789 boundaries, break them, and cause pore collapse (e.g. Handin et al., 1963; Wong et al., 1997). In
790 our Fontainebleau sandstone experiments, the majority of cracks nucleated at grain contacts and
791 opened along grain boundaries (Figure 7), similar to the findings of some other studies on
792 sandstones (Menéndez et al. 1996; Wu et al. 2000). Some cracks also propagated through grains,

793 as observed in other studies (Handin, 1963; Zhang et al., 1990; Wu et al., 2000; El Bied et al.,
794 2012). The tomograms do not reveal clear compaction or pore collapse at the grain scale prior to
795 failure. Although there was a global axial shortening of the sample, the overall porosity increased
796 from the onset of yield to failure. The low initial porosity of this rock (5-7%), may have inhibited
797 pore collapse because the potential maximum tensile stress that may develop at pore surfaces
798 decreases with decreasing pore volume (e.g., Wong and Baud, 2012).

799

800 **5.3 Implications of DVC strain analysis**

801 Videos S3 to S5, show that some high values of strain were detected by DVC analysis
802 inside the sample from the beginning of loading. This could be related to the presence of weak
803 zones inside the samples that deformed even under low differential stress. Such behavior has also
804 been observed when acoustic emissions was used to monitor deformation in porous sandstones
805 (e.g. Fortin et al., 2009).

806 The more diffuse distribution of the high incremental strains in experiments F1 and F3
807 (Figure 9a and 9c, Videos S3 and S5) compared to experiment F2 (Figure 9b, Video S4) suggests
808 why the sums of the incremental Von Mises strains did not change as significantly in experiments
809 F1 and F3 compared to experiment F2 (Figure 11). A core-spanning fracture did not develop
810 prior to macroscopic failure in these experiments as it did in experiment F2. Instead, preceding
811 macroscopic failure, the high values of the incremental strain remained relatively diffuse,
812 although there was some localization of the high strain values into tighter clusters (Figure 9a) and
813 narrower bands (Figure 9c) preceding macroscopic failure. Consequently, much of the high
814 incremental strain population remained outside of these strain localization zones preceding
815 macroscopic failure. However, propagation, opening and sliding along faults produced the final
816 stage of macroscopic failure in all three experiments. This suite of observations is similar to what
817 was observed in experiments that monitored acoustic emissions (e.g. Wu et al., 2000; Fortin et
818 al., 2009) and in which a surge of acoustic emissions occurred from the yield point to failure in
819 sandstones during brittle deformation. The DVC analyses of experiments F1 and F3 show that
820 strain events occurred homogeneously in the volume (Videos S3 and S5), similar to the
821 cataclastic compaction of a porous sandstone (top panel of Figure 5 in Fortin et al., 2009) and
822 was attributed to the opening of microfractures along grain boundaries (Menéndez et al., 1996).
823 The DVC analysis of experiment F2, in which a fault developed, is also similar to observations of

824 shear localization in a porous sandstone (middle panel of Figure 5 in Fortin et al., 2009; Wu et
825 al., 2000). In contrast to Fortin et al. (2009) experiments, we did not observe pore collapse in the
826 tomography data and our DVC results do not show significant volumetric compaction, likely due
827 to the low porosity of the Fontainebleau sandstone that we investigated.

828 The evolving distribution of the higher incremental strain magnitudes revealed by the
829 DVC analyses of experiment F2 indicates that both dilation and shear strain localized along the
830 core-spanning fracture (Figure 9b). The incremental strain fields suggest that for differential
831 stresses of 104 MPa to 151 MPa, incremental dilatational strains dominated the strain field
832 surrounding and within the developing shear fault zone, and above 151 MPa, incremental shear
833 strain became more localized than dilatational strains around the developing fault zone (Figures
834 9b, S3). These observations are consistent with post-mortem scanning electron microscopy
835 images of deformed sandstones that showed shear cracks dominating within the fault zone (e.g.
836 Wu et al., 2000). Initially, dilation, and perhaps tensile failure, localized along the incipient fault
837 zone, and then localized shear strain occurred within the developing fault only after some dilation
838 occurred.

839 To investigate in more detail the dominant failure modes that facilitated the initiation of
840 the fracture observed in F2, we calculated the incremental strain fields following the yield point
841 with the highest possible temporal (differential stress) resolution. Comparing the distribution of
842 the high differential strain magnitudes above the 95th percentile, or the top 5% of values,
843 indicated that the high magnitudes of dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$ began to concentrate around the
844 protofracture before the high magnitudes of $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$ (Figure S3). This observation is consistent
845 with the more localized distribution of dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$ relative to the spatial distribution of
846 $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$ from 104-151 MPa, and the more localized distribution of $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$ relative to
847 dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$ from 151-167 MPa observed in the lower temporal resolution analysis (Figure
848 9). The evolution of the sums above the yield point (insets in Figure 11b, 11e) indicate that the
849 normalized sum of $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$ is generally slightly higher than the normalized sum of the
850 dilatational part of the $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$ field in the differential stress increments immediately preceding the

851 yield point. These properties of the dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$ began to exceed those of $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$ when
852 dilatational strains began to localize along the core-spanning fracture (109-114 MPa differential
853 stress in [Figure 9](#); green lines in [Figure 11](#)).

854 In the two experiments (F2 and F3) in which the incremental strain invariant sums
855 generally increase ([Figure 11](#)), and the areas under the histograms generally increase ([Figure 10](#)),
856 the cumulative strain magnitude distributions generally progressed toward higher strain
857 magnitudes with increasing differential stress ([Figure S4](#)). In experiment F1, this trend was less
858 consistent than in the other experiments. In particular, the magnitude-frequency distributions
859 shifted toward lower magnitudes of strain from the second to last differential stress increment to
860 the final differential stress increment, consistent with the observed decrease in the sums ([Figure](#)
861 [11](#)).

862 To characterize the distribution of strain magnitudes close to the macroscopic shear
863 failure, we searched for scaling relationships within the populations of normalized incremental
864 dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$, contractional $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$ and $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$ strain increments shown in [Figure 10](#). Such
865 scaling relationships may reflect the long range elastic interactions of the stress field produced by
866 individual local microfractures and the presence of initial mechanical heterogeneities (e.g.,
867 [Renard et al., 2018](#)). The cumulative frequency distributions of the incremental strain invariants
868 show a linear trend on a log-log scale covering 1-2 orders of magnitude ([Figure S4](#)). The
869 incremental strain invariant curves flatten toward lower magnitudes near the resolution limit of
870 the incremental strain measurements, below which the values were removed ([Figure S4](#)). The
871 linear trend is indicative of a power-law scaling behavior with numerous small incremental strain
872 values, and fewer large incremental strain values. In log-log space, the slope ($\beta+1$) of the
873 cumulative histogram gives the exponent of the power-law scaling relationship, β , of the strain
874 increments ([Figure S5](#)). We calculated the power law exponents using the maximum likelihood
875 approach of [Clauset et al. \(2009\)](#). These exponents varied during the loading in the three
876 experiments ([Figure S5](#)). However, we observed no significant trend in the exponent value
877 throughout deformation. This relative stability of the exponents could be due to the small range
878 of spatial scales over which effective power-law behavior was observed ([Figure S4](#)).

879

880 **5.4 Transition from microfractures coalescence to localized shear failure**

881 The SEM images revealed that after a distinct shear band formed in experiment F2,
882 extensive grain comminution produced a layer of gouge within the shear zone (Figure 7). The
883 time resolution of the experiments (~1.5 minutes between scans) prevented determination of
884 whether the core-spanning fracture in experiment F2 developed through 1) a localized nucleation
885 and propagation mechanism or 2) the formation of a dense array of microfractures that coalesced
886 and ultimately formed a volume of unconsolidated, comminuted granular material. Tomograms
887 acquired beyond the yield point of experiment F2 showed progressive nucleation, growth and
888 coalescence of an array of opening cracks oriented subparallel to σ_1 (Figures 5, 7g-i).

889 Mechanical breakdown within rupture zones may initially produce a sand-like granular
890 material at these confining stresses. As comminution broadens the grain size distribution within a
891 shear zone, the local shear zone porosity may decrease as small grains fill spaces between larger
892 grains. The rounding and reorientation of large survivor grains (Cladouhos, 1999) may also
893 contribute to porosity reduction in shear zones following larger shear displacements. Such shear
894 zone gouges may also enable local dilation. However, the development of unconsolidated sand
895 grains in the shear zone alone cannot produce the observed macroscopic dilation (negative
896 macroscopic volumetric strain) of about -0.15 following failure. This macroscopic dilation of the
897 specimen suggests that microfractures in the material surrounding the shear zone contributed to
898 the post-failure porosity, in addition to dilation within the shear zone.

899 In experiments in which unconsolidated granular material was subjected to shear
900 deformation with confining stress perpendicular to the shear plane, shear bands developed with
901 enhanced comminution (e.g., Marone and Scholz, 1989). When a broad grain size distribution
902 developed within the fault gouge, the local porosity within the gouge decreased (Marone and
903 Scholz, 1989). In the case of the low porosity Fontainebleau sandstone used in the experiments
904 reported here, Dilation occurred within the incipient shear rupture zone because of the formation
905 of a dense array of sub-vertical microfractures (Figure 6). The opening of these microfractures
906 may lead initially to the formation of a high porosity band of unconsolidated quartz particles that
907 evolves into a low porosity zone of highly comminuted quartz particles with a broad size
908 distribution. However, we did not observe a low porosity zone in these experiments, perhaps
909 because the shear strain within the rupture zone and/or the confining stress perpendicular to the
910 shear plane were too small to reorient, round or crush the grains within the incipient fault gouge,
911 and thereby decrease porosity within the zone. Perhaps if we had continued the experiment

912 beyond macroscopic failure and allowed more evolution of the fault gouge within the shear zone,
913 the local porosity within the gouge would have decreased. In addition, the short (multiple hour)
914 time span and dry conditions in our experiments prevented chemical processes that would further
915 reduce fault zone porosity in the crust.

916 Differences in the rate of fracture coalescence and fault propagation may have caused a
917 core-spanning fracture to localize earlier in experiment F2 (closer to the yield point) than
918 experiments F1 and F3 (immediately before macroscopic failure). For the same rate of
919 deformation, fault propagation tends to progress at a slower rate in more macroscopically brittle
920 rocks than in less brittle rocks that have a higher component of ductile deformation (Ougier-
921 Simonin and Zhu, 2013, 2015). The imposed confining stress of experiment F1 (20 MPa) was
922 larger than that of experiment F2 (10 MPa), and the nominal strain rate of experiment F1 (184
923 scans, each of 1.5 minute duration) was lower than that of experiment F2 (49 scans, each of 1.5
924 minute duration). Higher confining stresses and lower strain rates tend to allow more ductile
925 deformation to accommodate the applied strain, and rocks behave in a less brittle manner with
926 lower fault propagation rates. Consequently, the experimental conditions produced more brittle
927 behavior in experiment F2 than in experiment F1, which promoted slower fault propagation in
928 experiment F2 compared to experiment F1. Similarly, fault propagation may have occurred at
929 slower rates in experiment F2 than experiment F3 because the Young's modulus and failure
930 stress of experiment F3 were lower than those of experiment F2, signaling more brittle behavior
931 during experiment F2 than F3.

932 In each of our experiments, the angle of inclination, θ , between the maximum
933 compression direction and the core-spanning fracture was smaller than that predicted by the Mohr
934 Coulomb failure criterion. We cannot rule out that the possibility that this was caused was the
935 absence of lubrication of the piston-sandstone interface. However, this observation was also
936 reported in previous experiments on Vosges sandstone (22% porosity) in which failure occurred
937 via propagation of sub-axial fractures (e.g. Bésuelle et al., 2000). In these experiments, increasing
938 confining stress increased the orientation of the through-going fractures with respect to σ_1 . At the
939 highest confining pressures, compaction bands developed subperpendicular to σ_1 , thereby
940 following the observed trend. The sub-axial microfractures appeared to control how strain
941 localized into core-spanning fractures. As the density of sub-vertical microfractures increased, the
942 mechanical properties of the rock became increasingly anisotropic, which may have modified the

943 orientation of new fractures and how fractures coalesced. These fractures had orientations
944 between mode I sub-axial fractures when $P_c \ll \sigma^f$, and shear failure consistent with the
945 orientation predicted by the Mohr Coulomb failure criterion when $P_c < \sigma^f$ (i.e., when P_c is a few
946 tens of MPa), where σ^f is the differential stress at failure. As the differential stress increased and
947 the sub-axial fractures increased in size, the unfractured columns between them became more
948 unstable (e.g. Figure 41 in Peng and Johnson, 1972). If one column collapses, the forces acting on
949 it will be transferred to other columns, and a cascade of column collapses may be responsible for
950 strain localization that forms fractures at higher angles than those predicted by the Mohr-
951 Coulomb criteria. Such conceptual description of the failure process is quantified in statistical
952 physics models. As failure is approached, the sizes of the cascades (or avalanches) will increase
953 in size as observed in numerical simulations (e.g., Dahmen et al., 2009; Girard et al., 2010). The
954 preferential orientation of sub-axial microfractures suggests that the long-range function that
955 describes how the rate of damage evolution at one point influences the damage evolution at other
956 points should depend on both the direction and length of the vector between the two points
957 because damage develops preferentially in certain directions, and is thus anisotropic, as proposed
958 for crystalline rocks in the model of Lockner and Madden (1991b).

959 The final core-spanning fault in our experiments was not planar, but curved (Figures 5d,
960 6d). Such curved fault surfaces have also been observed in crystalline rocks (Peng and Johnson,
961 1972). Curved strain concentration zones are not uncommon. For example, Sulem, and
962 Ouffroukh (2006) mentioned measuring the orientation of the shear band on the central part of
963 the specimen because the shear band often warps close to the extremities.

964 The development of a new micromechanical model for sandstone is out of the scope of
965 the present study. A sound understanding of the micromechanics of complex materials should
966 help justify the forms of macroscopic constitutive equations that predict the mechanical and
967 rheological behaviors of rocks. Such macroscopic laws are useful because of their small number
968 of parameters (e.g. Rudnicki and Rice, 1975; see also the discussion in Dresen and Guéguen,
969 2004) and provide a complementary approach to micromechanical modelling. A
970 micromechanical constitutive model should incorporate processes that cause the local increase of
971 damage (e.g. opening of grain boundaries and formation of transgranular microfractures) and the
972 effect of damage on local stress distribution (Cox and Meredith, 1993). It should predict
973 macroscopic properties such as strength, amount of dilation and evolution of elastic parameters

974 and also whether a fault will develop early, near the yield point (like the core spanning fracture in
975 experiment F2) or later near failure (experiment F1 and F3). However, the materials and
976 experimental conditions were essentially the same in experiments F2 and F3, and this suggests
977 that experiments F2 and F3 were conducted very near the boundary between two quite different
978 deformation and failure regimes or that prediction of deformation and failure will be very
979 challenging if not impossible for rocks like low porosity Fontainebleau sandstone.

980

981 **5.5 Limitations of digital volume correlation analysis near failure**

982 Our DVC analyses quantified local strain components (dilation, compaction, shear strain)
983 that may be compared with the evolving pore and fracture network revealed through
984 segmentation of the tomograms, and the macroscopic strain. Histograms of incremental strain
985 components (Figure 10) and the sum of the increments of each strain component (Figure 11) at
986 increasing differential stress steps showed that components of local strain concentrations can be
987 up to one order of magnitude larger than the macroscopic axial strain. The power law scaling
988 analysis of the cumulated distribution of the incremental strain components showed that the
989 exponent did not vary significantly as failure is approached (Figure S5), whereas the total damage
990 increased (Figure 8).

991 During macroscopic failure and the post-failure phase when comminution occurred, the
992 structure of the incipient fault zone changed rapidly. Consequently, DVC analysis of the
993 evolution of the incipient fault zone may not produce meaningful results because changes in the
994 sizes, shapes and orientations of quartz grains, and grain fragmentation during the acquisition
995 time may blur the tomograms. In addition, large differences in microstructures between
996 tomograms may inhibit adequate correlation of sub-volumes across sequential tomograms. Time
997 lapse X-ray microtomography of deformation with small increments of shear strain could reveal
998 the comminution process within the incipient fault zone in more detail than these experiments.
999 However, strain fields obtained from DVC analysis of tomograms separated by these small strain
1000 increments will have lower signal to noise ratios than the strain fields presented in this study
1001 because the signal (incremental strain) decreases the macroscopic strain between scans decreases
1002 (e.g., McBeck et al., 2018).

1003

1004 **5.6 Neutron tomography**

1005 Neutron tomography has been used with DVC analysis to investigate strain localization
1006 and compressive failure of Bentheim sandstone (Tudisco et al., 2015). However, the technology
1007 of neutron tomography is not as far advanced as the technology of X-ray tomography. Neutron
1008 microtomography requires several hours for three-dimensional acquisitions at 30 μm spatial
1009 resolution, whereas the HADES deformation apparatus installed at ID19 beamline at ESRF
1010 enables 1.5 minutes acquisition times at 6.5 μm spatial resolution for cores 1 cm tall and 0.5 cm
1011 wide. Consequently, time lapse neutron tomography has been only rarely used to investigate the
1012 micromechanics of rocks (e.g. Stavropoulou et al., 2018). The attenuation coefficients of neutrons
1013 are not as high as those of X-rays, which is an advantage for triaxial rigs with thick walls that are
1014 required for applying higher stresses than the 200 MPa differential stress limit of the HADES rig.
1015 In addition, neutron and X-ray contrasts are quite different, and in particular, the high neutron
1016 cross-section of hydrogen would be advantageous when studying fluids such as water and
1017 hydrocarbons within rocks.

1018

1019 **5.7 Temperatures and stress limits**

1020 The HADES rig was fabricated from titanium and experiments can be conducted in the rig at
1021 stresses up to 200 MPa and temperatures up to 250 $^{\circ}\text{C}$. This enables processes that occur at
1022 depths up to about 7 km to be investigated. While a very broad range of geological processes and
1023 most geotechnical applications occur under these conditions, there is a clear need to extend the
1024 range of temperatures and stresses. It should be possible to achieve this by using advanced
1025 materials that are strong at high temperatures and have low X-ray attenuation coefficients.
1026 Candidate materials include alloys composed primarily of lithium, beryllium magnesium and
1027 aluminum, metal matrix composites formed from these light alloys and carbon or ceramic fibers,
1028 and carbon fiber reinforced carbon. However, most, if not all, of these and similar materials are
1029 difficult and expensive to fabricate, and few companies have experience with the design and
1030 manufacture of apparatus constructed from these materials.

1031

1032 **6. Conclusion**

1033 Segmentation and digital volume correlation analysis of micrometer-resolution
1034 tomograms acquired through in situ dynamic synchrotron X-ray microtomography during triaxial

1035 compression loading revealed the damage and strain preceding brittle macroscopic failure in
1036 three experiments on Fontainebleau sandstone. In one experiment, a system-size fracture
1037 nucleated at 78% of the differential stress at failure and accommodated an increasing proportion
1038 of incremental shear strain relative to incremental dilatational strain. In the other two
1039 experiments, opening of grain boundaries was the dominant dilation mechanism and localization
1040 occurred late, at 95% of the differential stress at failure. Increases in dilatancy and shear strain at
1041 the micro- and macro-scales, were observed during the loading stage preceding failure for all
1042 three experiments.

1043 Our experiments are consistent with experiments performed on Fontainebleau sandstone
1044 (El Bied et al., 2002; Schubnel et al., 2007; Nasser et al., 2014; Goodfellow et al., 2015) and on
1045 other sandstones (Handin et al., 1963; Menéndez et al., 1996; Wu et al., 2000; Fortin et al., 2009).
1046 Failure of Fontainebleau sandstone did not produce discontinuous shear displacement along an
1047 idealized plane separated by two essentially undamaged sandstone bodies. Instead, progressive
1048 damage accumulated with increasing differential stress as microfractures propagated along grain
1049 boundaries as well as through grains (Videos S1 and S2). After the yield stress was reached,
1050 damage accumulated at increasing rates until macroscopic shear failure occurred. The
1051 microscopic damage developed as sub-axial fractures that propagated, coalesced and became
1052 increasingly concentrated into a high aspect ratio system-spanning volume.

1053 Our experimental approach provided new information on the nucleation of failure and on
1054 the transitions between microfracture nucleation, microfracture coalescence and macroscopic
1055 failure. Incremental strain components revealed by DVC analysis indicated that the high density
1056 sub-axial fracture zone did not form a plane but a curved surface. This progressive damage
1057 enhanced heterogeneity in the stress and strain fields, as well as the local distribution of
1058 mechanical properties. Time lapse X-ray microtomography and DVC analysis provided detailed
1059 information about the evolving microstructure of the Fontainebleau sandstone cylinders as they
1060 deformed and approached macroscopic failure. The dilation of the samples during loading was
1061 due to both crack opening and pore opening, with an almost equal volumetric contribution of
1062 these two processes to the increase of void volume (Figure 6a). The difference in
1063 micromechanical behavior between experiments F1 and F3 on one hand and F2 on the other hand
1064 did not appear clearly in the stress-strain curves (Figure 1), which showed that the ability to “look
1065 inside” rock specimens provided by X-ray tomography is critical to identify the route to failure.

1066 Another important result was obtained by segmenting the voids during loading. We observed a
1067 slow-down of the rate at which damage, characterized by the void volume fraction based damage
1068 index, D_ϕ , increased with increasing differential stress as failure was approached (Figure 8),
1069 which is not observed in crystalline rocks (Renard et al., 2018). We attribute this to the pinning of
1070 microfractures by pores that may reduce the stress concentration at crack tips. These new results
1071 demonstrate that very few, if any, other experimental methods enable such precise correlations
1072 between the macroscopic mechanical behavior of rock specimens and the micromechanical
1073 processes that occur within them.

1074
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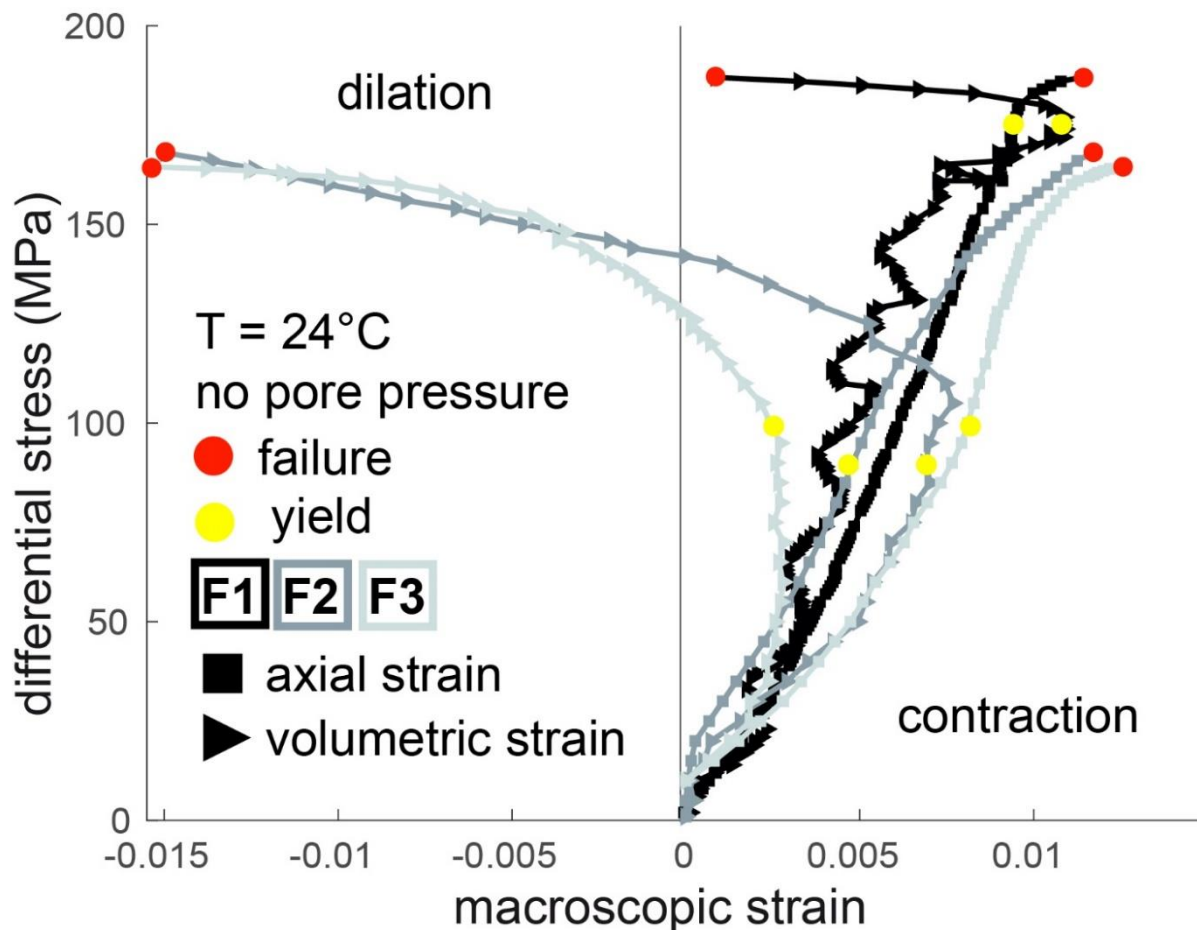
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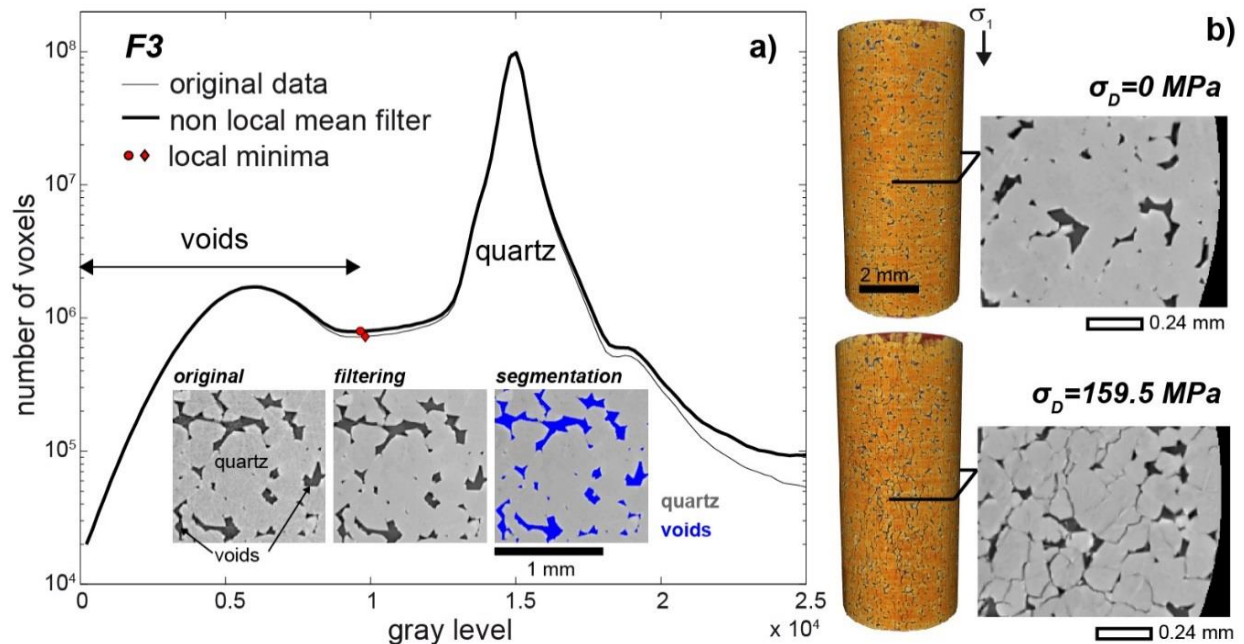
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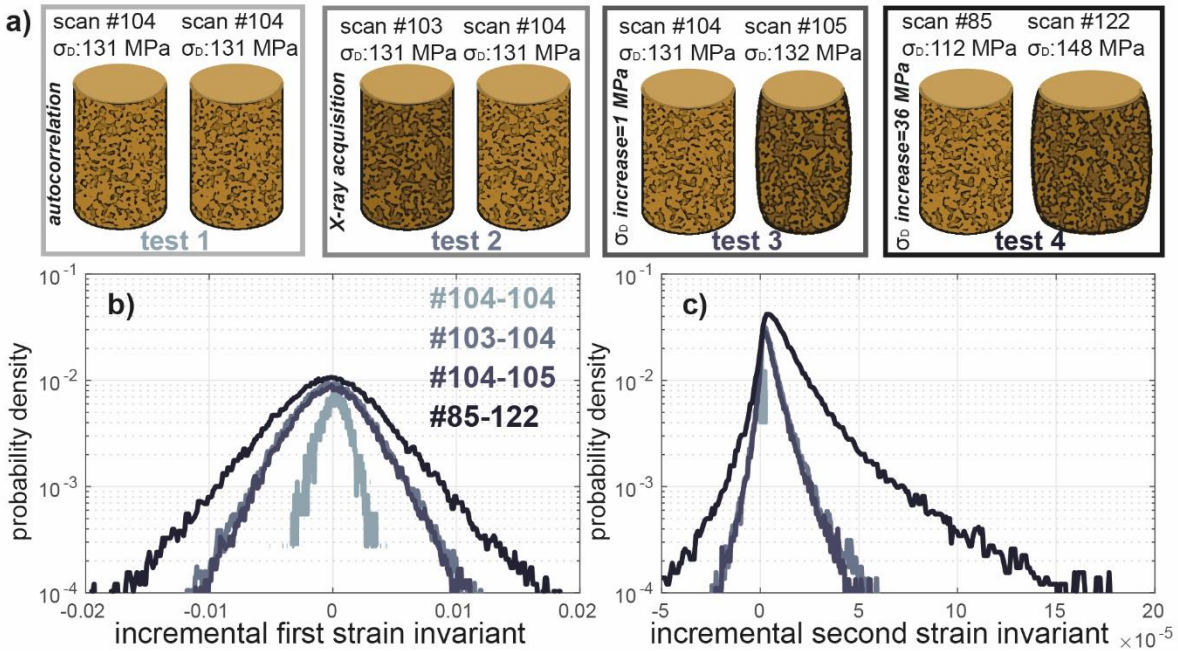


	σ_D at failure	σ_D at yield	σ_2	# scans
F1	185 MPa	165 MPa	20(14) MPa	184
F2	167 MPa	90 MPa	10 MPa	49
F3	159.5 MPa	100 MPa	10 MPa	55

1316
 1317 Figure 1: Experimental axial and volumetric strains as functions of the differential stress for the
 1318 three Fontainebleau sandstone experiments. Each point corresponds to the acquisition of a three-
 1319 dimensional X-ray tomography data set. Yellow and red circles show the yield and failure points.
 1320 The table lists the experimental conditions. In experiment F1, the confining pressure was reduced
 1321 from 20 MPa after the axial stress has reached 199 MPa, and failure occurred when the confining
 1322 pressure was reduced below 15 MPa at a constant axial stress of 199 MPa (i.e., at a differential
 1323 stress of 185 MPa). In experiments F2 and F3, the confining pressure was held constant. In
 1324 experiment F1, the oscillations in the volumetric curve arose from instabilities in the confining
 1325 pressure pump.

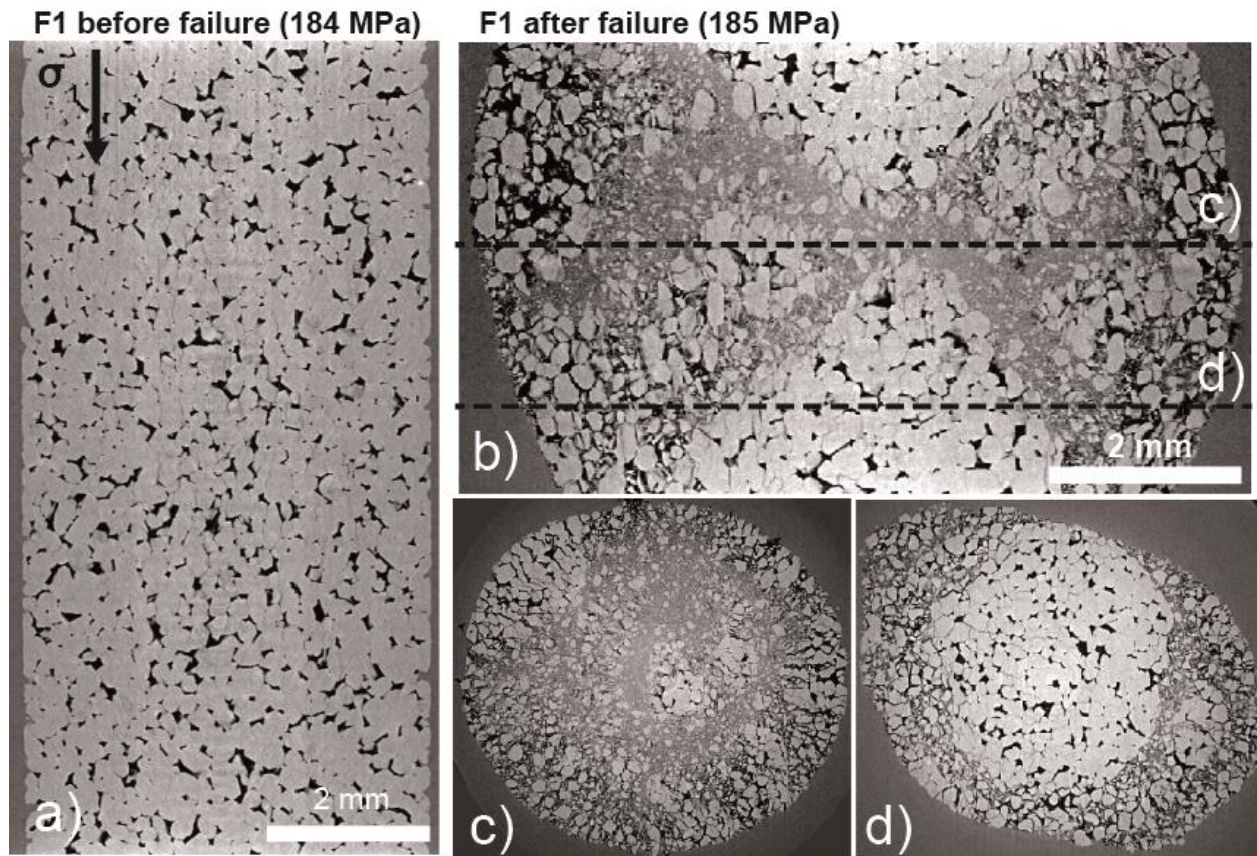


1326
 1327 Figure 2: Thresholding procedure used to segment the three-dimensional data sets into quartz
 1328 matrix and air-filled voids, including pores and fractures. a) Distribution of the gray levels of
 1329 experiment F3 for the original three-dimensional data set (thin line) at a differential stress of 10
 1330 MPa, before and after applying the non-local mean filter (thick line). Insets show two-dimensional
 1331 slices of the sample with the unfiltered data (left), filtered data (middle) and segmented data (right).
 1332 We selected the local minima of the histogram between the peaks arising from voids (left peak)
 1333 and from quartz grains (right peak) to segment the tomograms into quartz grains and voids. The
 1334 red symbols show the minima of the original (diamond) and filtered (circle) data, which do not
 1335 differ significantly. b) Three-dimensional rendered view of the specimen before and after
 1336 experiment, and two-dimensional horizontal cross-sections. [Video S2](#) shows the time-lapse
 1337 evolution of the specimen in this experiment.

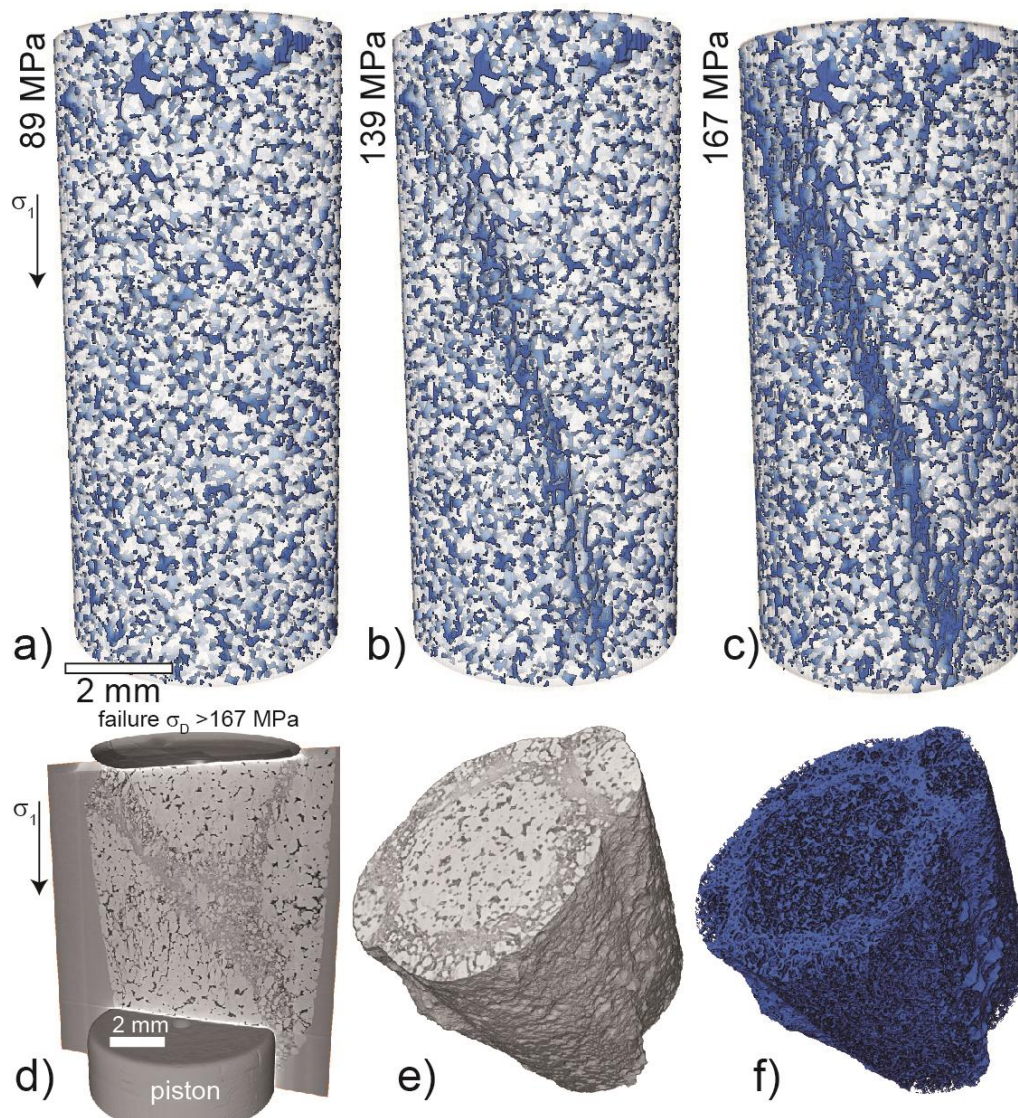


1338

1339 Figure 3: Characterization of the resolution of the digital volume correlation technique using four
 1340 tests. a) Pairs of three-dimensional data sets used in the four tests of digital volume correlation
 1341 analysis. b, c) Results of the tests. The gray level of the boxes surrounding each pair of tomograms
 1342 in (a) matches the gray levels of the probability densities shown in (b) and (c). The strain resolution
 1343 is taken as twice the standard deviation found in test 2 between scans #103-104, a more
 1344 conservative estimate of the error than test 1.

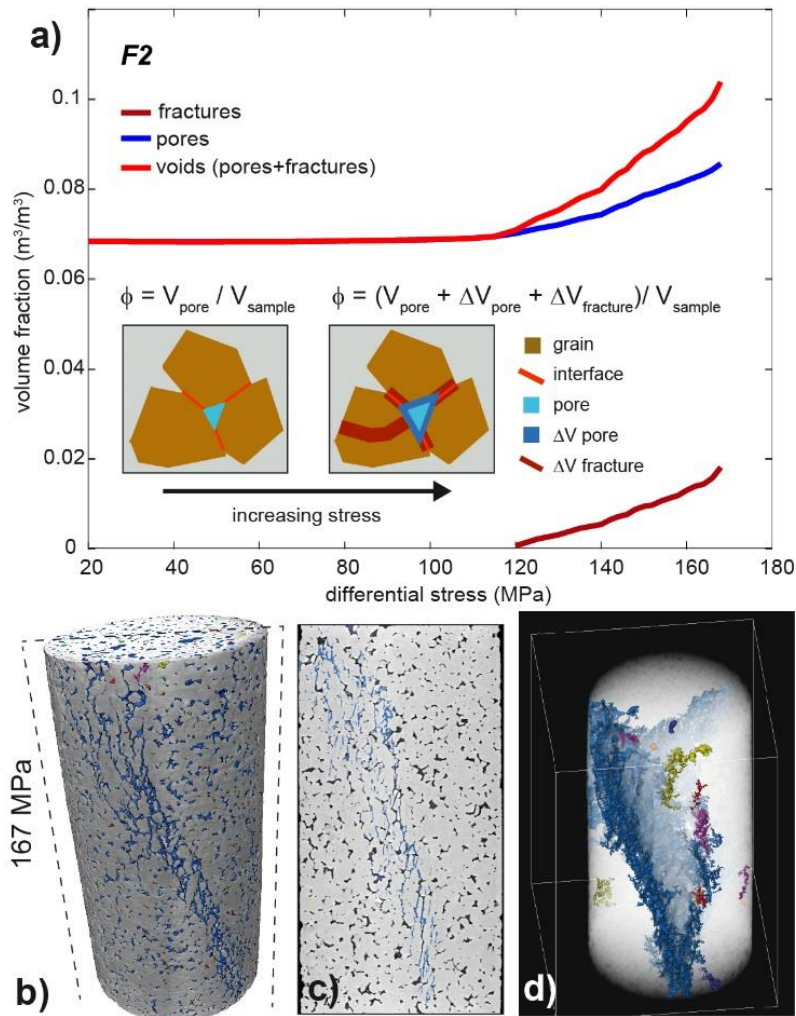


1345
1346 Figure 4: Two-dimensional views of experiment F1 immediately preceding and following
1347 macroscopic failure at differential stresses of 184 and 185 MPa. a) Vertical axial transect at the
1348 onset of failure showing quartz grains, pores, and microfractures oriented sub-parallel to the main
1349 compressive stress direction (vertical). b) Vertical transect of the sample after macroscopic failure.
1350 Compared to a) the sample has shortened and widened significantly because of failure. Dashed
1351 lines indicate the location of the two perpendicular (horizontal) cross-sections shown in c) and d).
1352 The sample failed through the formation of conical fracture zones in which grains were
1353 comminuted.



1354

1355 Figure 5: Evolution of pore space and fracture networks in experiment F2. a-c): Three-
 1356 dimensional rendered views of the specimen at increasing differential stresses. Porosity and
 1357 fractures are shown in blue and the quartz matrix is shown in white. c) The onset of failure (167
 1358 MPa). [Video S1](#) shows a complete time-lapse three-dimensional rendered view of the specimen
 1359 throughout the experiment. d) Sample after failure ($\sigma_D > 167$ MPa) with the two pistons of the
 1360 HADES rig displayed. e) Three-dimensional rendered view of the sample after failure with
 1361 comminuted grains. f) Structure of the porous network, which was 99% connected in three
 1362 dimensions both preceding and following failure. Following failure, the porosity in the fracture
 1363 zones that host comminuted grains was smaller than the porosity in the other parts of the rock,
 1364 where the pore space is similar to that in the rock before loading.



1365

1366 Figure 6: Pores and fractures identified after segmentation of the three-dimensional data sets of

1367 experiment F2. a) Evolution of the pore, fracture and total void (i.e., pore + fracture) volume

1368 fractions as a function of the differential stress. The total void volume fraction (red curve) can be

1369 separated into propagating fractures (blue curve) and expanding pores (dark red curve) in this

1370 experiment. The inset sketch shows how the pore and fracture volumes increased. b-d) Results of

1371 segmentation for experiment F2 at a differential stress of 167 MPa. b) Three-dimensional

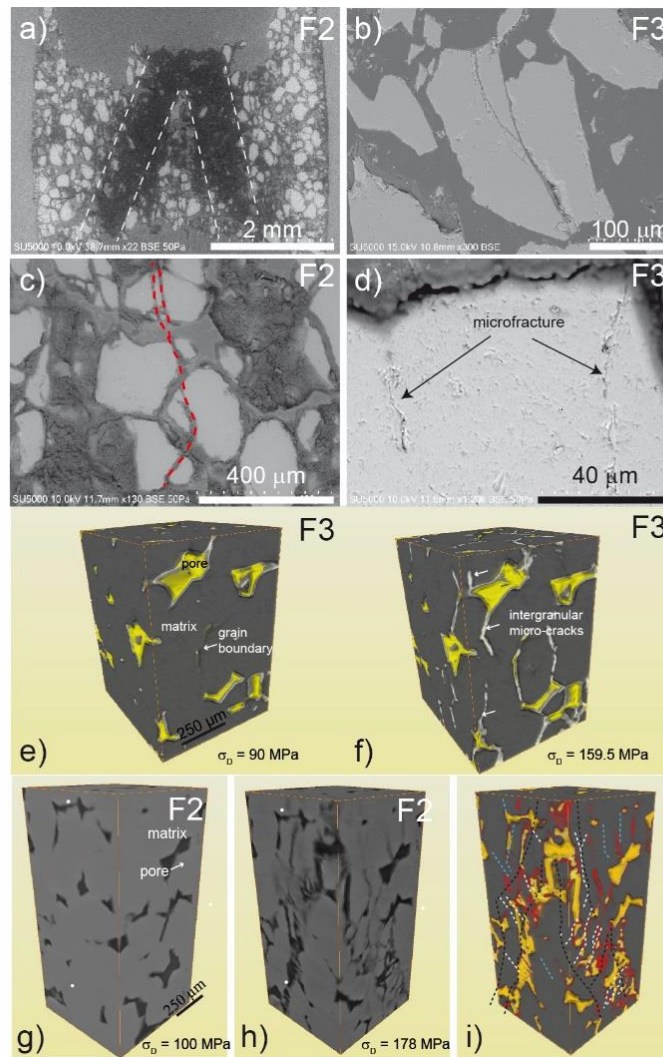
1372 rendered view of the specimen at the onset of failure with quartz shown in light gray, and pores

1373 and fractures shown in colors. c) Vertical transect of the previous image, with fractures shown in

1374 blue, pore space shown in black, and quartz shown in light gray. d) Three-dimensional view of

1375 the microfracture network shown in blue and other colors. Each color shows a unique,

1376 unconnected, fracture network.



1377

1378 Figure 7: Microstructures of deformation. a-d): Scanning electron microscopy images of samples

1379 F2 and F3 after failure. a) Sample F2 with fault zones highlighted by white dashed lines. b)

1380 Transgranular fracture. c) Transgranular fractures that may have been produced by a directed

1381 force network, or chain (dashed red lines). d) Onset of microfractures in a quartz grain. e-f)

1382 Three-dimensional rendering of X-ray microtomograms from experiment F3 (e) before and (f)

1383 after failure showing mostly intergranular fractures. Yellow shows porosity and white shows

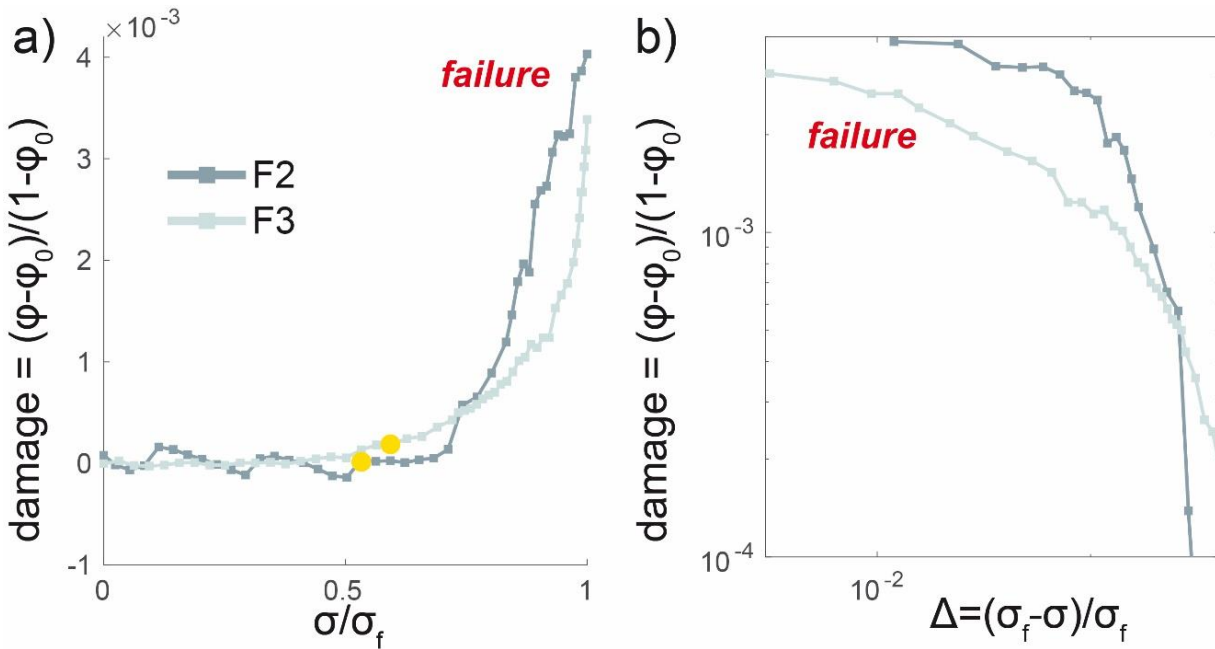
1384 intergranular fractures. Three-dimensional rendered views of experiment F2 (g) before and (h, i)

1385 after failure showing both intergranular and transgranular fractures. i) Red highlights damaged

1386 areas. g-h) White dots show strain markers in both images. (i) White dotted lines highlight shear

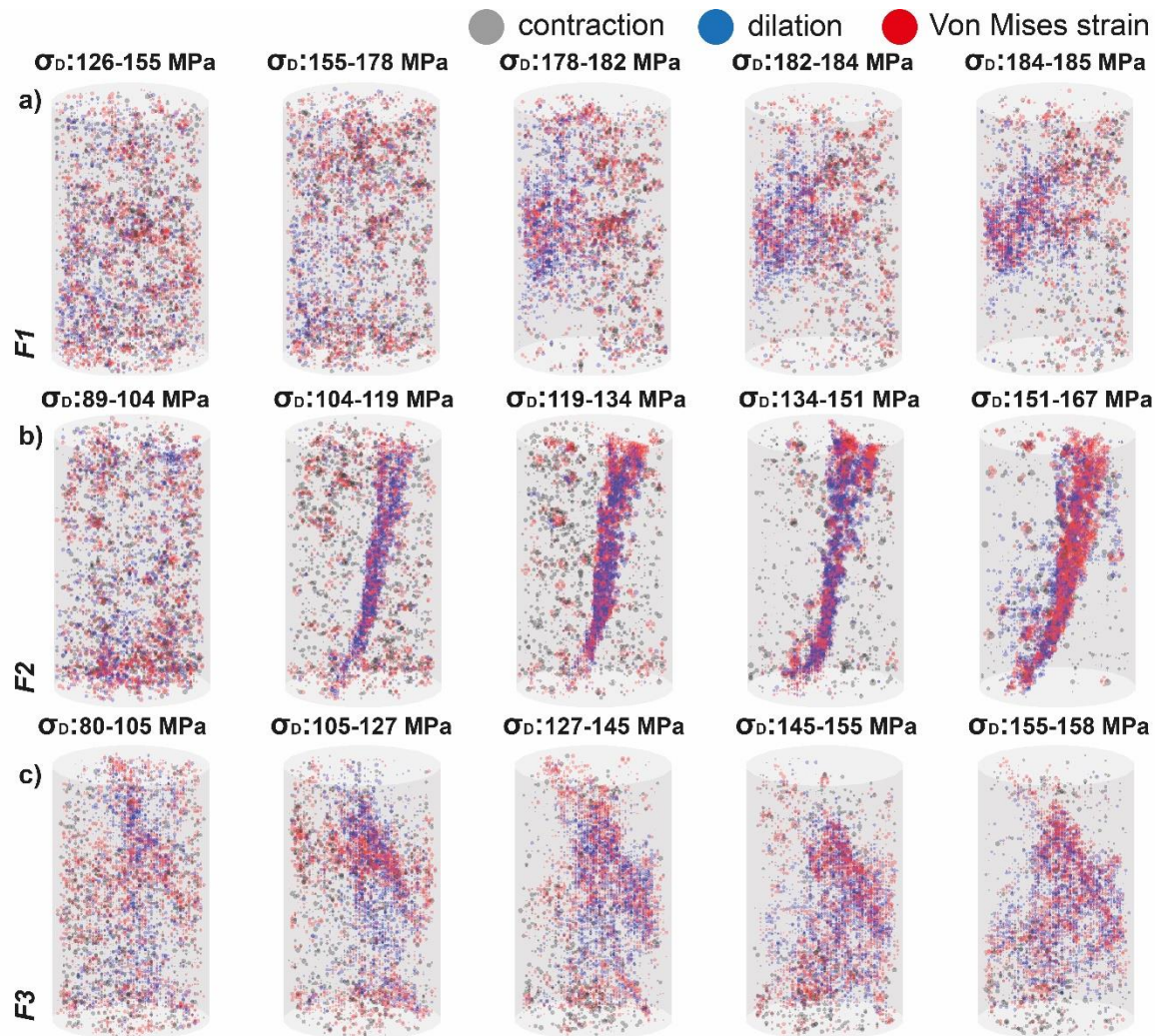
1387 bands. Blue dotted lines show intergranular and transgranular fractures. Dark dotted lines

1388 highlight the fault zones.

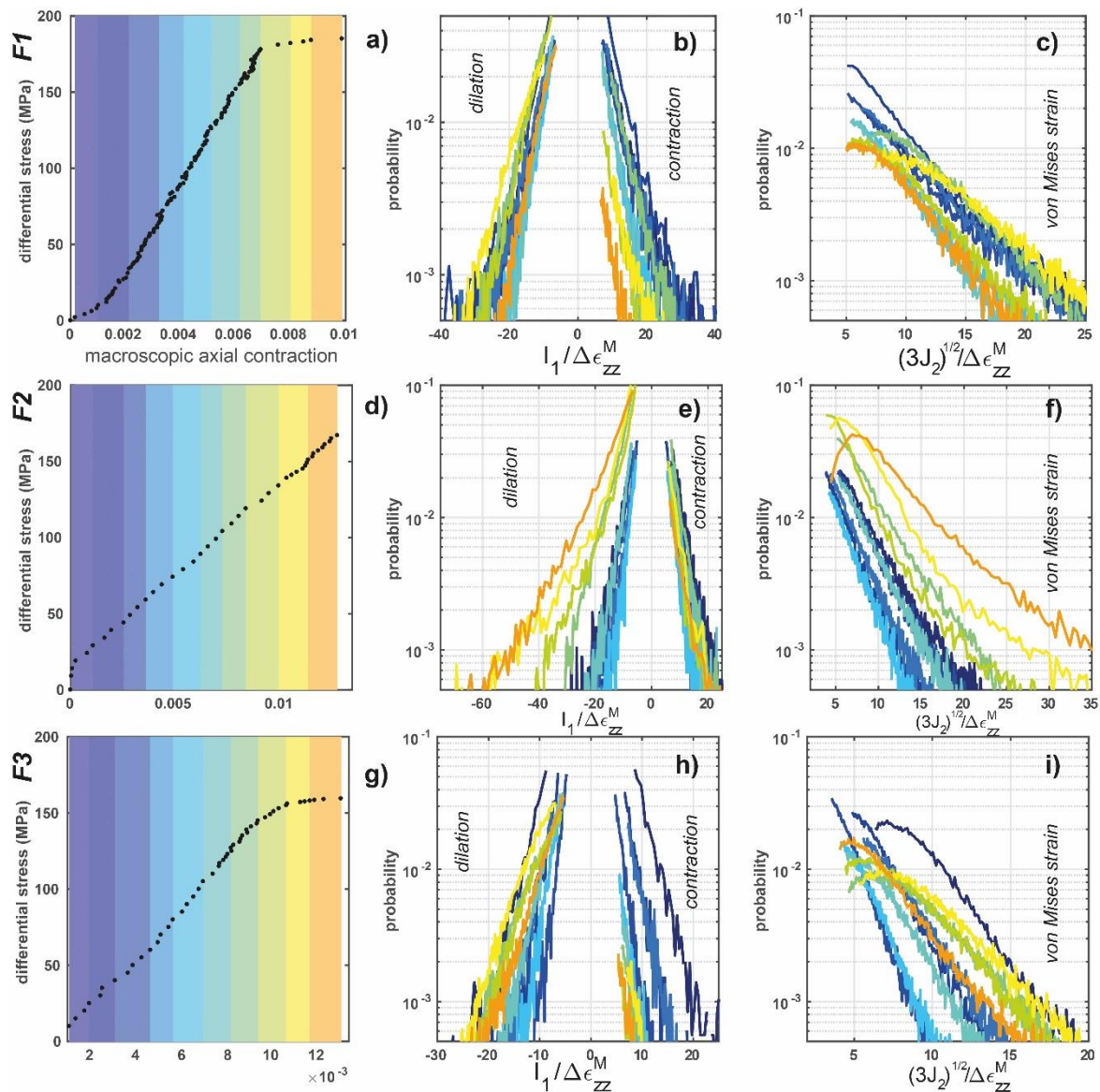


1389

1390 Figure 8: Evolution of the damage index as the sandstone specimens approached failure in
 1391 experiments F2 and F3. [Figure S2](#) shows data from experiment F1. a) Evolution of the damage
 1392 index as a function of the differential stress normalized by the differential stress at failure, σ / σ_f .
 1393 The porosity and damage index increased as the differential stress approached the differential
 1394 stress at failure. b) Evolution of the damage index as a function of the normalized distance to
 1395 failure, $\Delta = (\sigma_f - \sigma) / \sigma_f$, in log-log space. Failure occurred when $\sigma / \sigma_f = 1$ in a) and when $\Delta =$
 1396 0 in b). Yellow circles show the yield points.

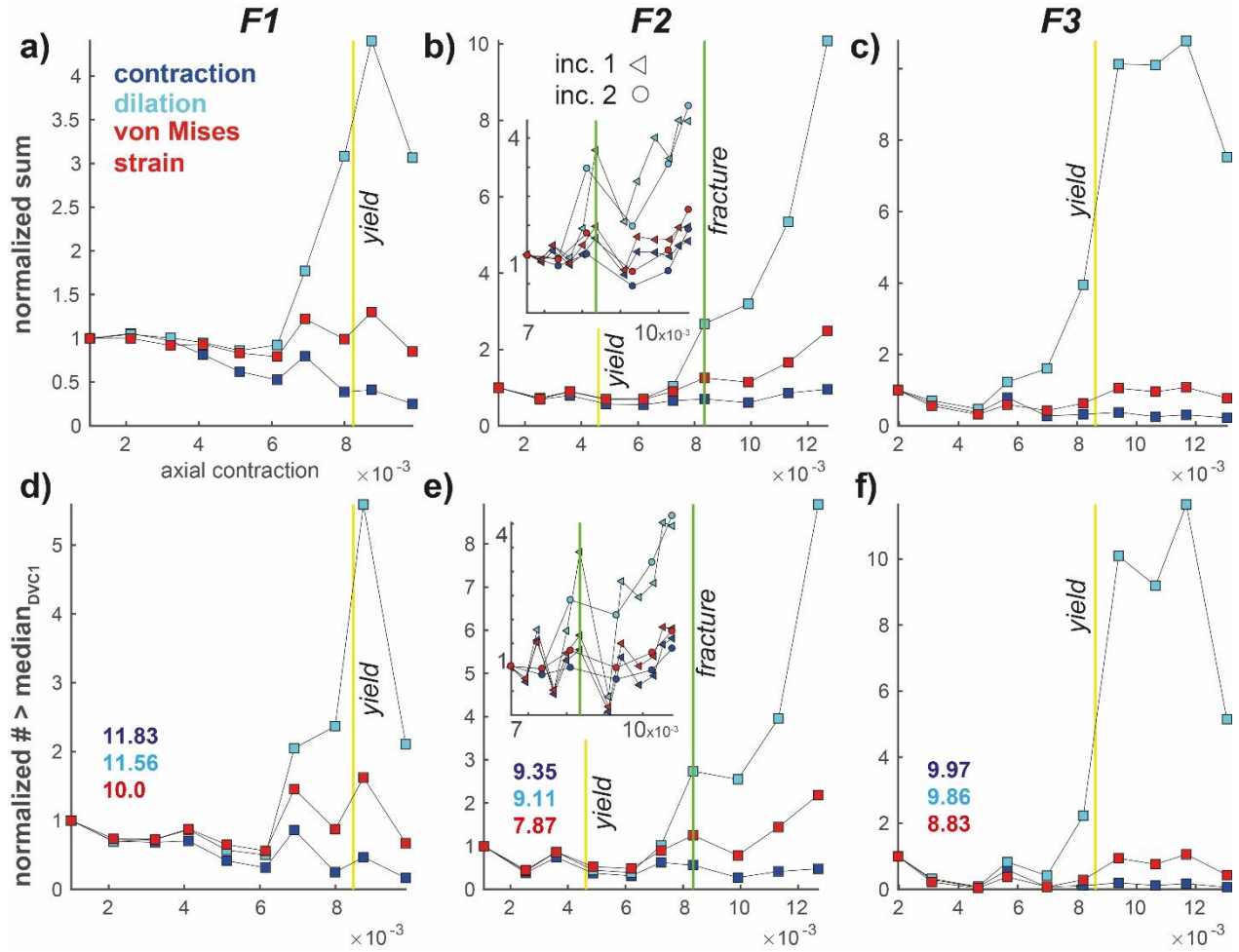


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 1398 Figure 9: Spatial distribution of incremental strain invariant magnitudes above the 95th percentile
 1399 of each incremental strain population for experiments F1 (a), F2 (b), and F3 (c). The sizes of the
 1400 points are proportional to the magnitude of the incremental strain invariants. Gray dots show the
 1401 contractive $\frac{I_1(\Delta\epsilon)}{\Delta\varepsilon_{ZZ}^M}$, blue dots show dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\varepsilon_{ZZ}^M}$, and red dots show the Von Mises incremental
 1402 strain, $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\varepsilon_{ZZ}^M}$, used here to characterize shear strain. The differential stresses above each
 1403 figure indicate the stresses at which the pairs of tomograms used in the DVC analysis were
 1404 acquired. [Videos S3-S5](#) show the spatial distribution of high strains throughout each experiment.



1405

1406 Figure 10: Histograms of incremental normalized strain invariants determined by DVC analysis
 1407 of pairs of three-dimensional data sets for experiments F1 (a-c), F2 (d-f) and F3 (g-i). a, d, g
 1408 Differential stress-axial strain curves. The rectangles show the strain intervals between which we
 1409 performed digital image correlation. b, e, h) Histograms of $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$. c, f, i) Histograms of $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$.
 1410 The color of each histogram corresponds to the strain interval displayed in the first column. The
 1411 gap in the histogram curves at small magnitudes arises because strain values below the identified
 1412 threshold (Figure 3) were removed.



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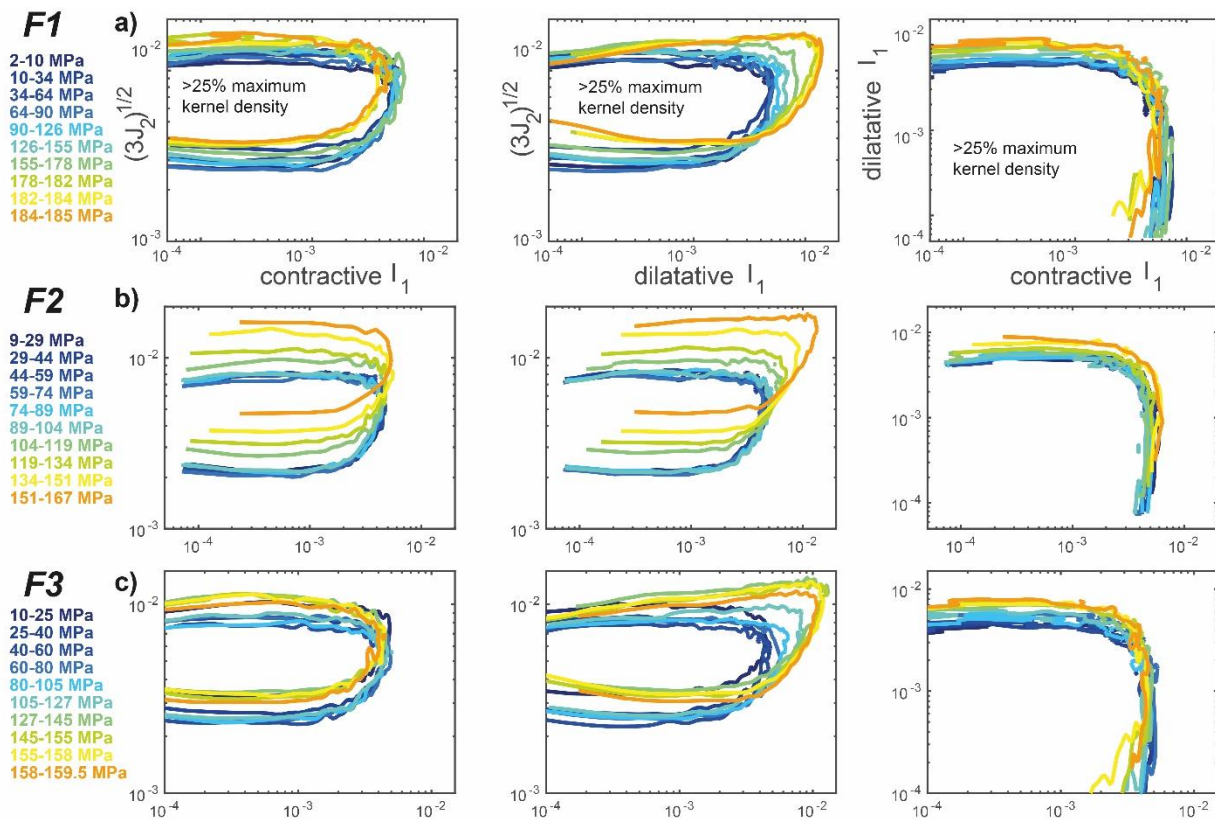
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Figure 11: Evolution of sums of incremental strain invariants (a-c) and number of values above the median identified in the first increment, $\text{median}_{\text{DVC1}}$, (d-f) in experiments F1 (a, d), F2 (b, e), and F3 (c, f). Dark blue, light blue, and red squares show values of contractive $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$, dilatational $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{zz}^M}$, and $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{zz}^M}$ (shear strain). Each sum is normalized by the sum calculated in the first increment. Each number of strain values above $\text{median}_{\text{DVC1}}$ is normalized by that number in the first increment. Inset of (b) shows sums near the yield point of experiment F2 where DVC analyses were performed on twelve pairs of tomograms with successive stress step increase. Yellow lines show the yield point for each experiment. The green line shows the onset of fracture development in sample F2. The value of $\text{median}_{\text{DVC1}}$ for each strain invariant population is shown in each of the plots, corresponding to the normalized incremental strain invariant. The dark blue, light blue and red numbers indicate the median values of contraction, dilation and Von Mises incremental strains, respectively.



1426

1427 Figure 12: Evolution of deviatoric and normal incremental strain invariants for experiments F1

1428 (a), F2 (b), and F3 (c). For each incremental strain field, we calculated the dilative $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$,

1429 contractive $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$, and $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ value at each point. The bivariate kernel density of each pair of

1430 strain values was estimated assuming a Gaussian distribution: contractive $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ vs. $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$

1431 (left), dilative $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ vs. $\frac{(3J_2(\Delta\epsilon))^{1/2}}{\Delta\epsilon_{ZZ}^M}$ (middle), and contractive $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ vs. dilative $\frac{I_1(\Delta\epsilon)}{\Delta\epsilon_{ZZ}^M}$ (right). Lines

1432 outline the strain values with a kernel density >25% of the maximum kernel density.