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# 1 Strain localisation and grain breakage in sand under shearing 2 at high mean stress: insights from *in-situ* x-ray tomography

3 Alikarami R.<sup>1,2</sup>, Andò, E.<sup>3</sup>, Gkiousas-Kapnisis M.<sup>3</sup>, Torabi A.<sup>1</sup>, Viggiani G.<sup>3</sup>

4<sup>1</sup> Uni CIPR, Uni Research , P.O. Box 7810, N-5020 Bergen, Norway

5<sup>2</sup> Earth Science Departments, University of Bergen, P.O. Box 7800, N-5020 Bergen, Norway

6<sup>3</sup> Grenoble-INP / UJF-Grenoble 1 / CNRS UMR 5521, Laboratoire 3SR, 38041 Grenoble, France

7 Corresponding author: Reza Alikarami, Uni CIPR, Uni Research , P.O. Box 7810, N-5020 Bergen, Norway  
8 ([reza.alikarami@uni.no](mailto:reza.alikarami@uni.no))

9

## 10 Abstract

11 This work presents results from a series of triaxial compression tests on two quartz sands  
12 (differing principally in grain shape), at confining pressures high enough to cause grain breakage  
13 during shearing. Tests are performed inside an x-ray scanner, which allows specimens to be  
14 imaged non-destructively as they deform. Observation of the acquired images clearly shows  
15 different mechanisms of deformation, including shearing, dilation, compaction and grain  
16 breakage. These mechanisms are investigated quantitatively through 3D measurements of local  
17 porosity, as well as strain (obtained by 3D Digital Image Correlation), which is analysed in terms  
18 of volumetric and shear components. These tools allow the transition between macroscopically  
19 dilative (typically of a dense sand at low mean stress) and compactive behaviour to be  
20 investigated. The analysis reveals that at the high end of the confining pressure range studied  
21 (100 to 7000 kPa) the more rounded sand deforms with highly localised shear and volumetric  
22 strain – the porosity fields show a dilative band within which a compactive region (due to grain  
23 crushing) grows. The more angular material shows shear strain localisation, however its faster  
24 transition to compactive behaviour (due to a higher propensity for individual grains to crush)  
25 translates to much more distributed compactive volumetric strain.

## 26 Keywords

27 Deformation band, Grain breakage, Grain shape (angularity), Triaxial compression test, In-situ x-  
28 ray microtomography, 3D Digital Image Correlation

29

## 301. Introduction

31 Weakly cemented and poorly lithified sandstones are important class of geological reservoirs  
32for hydrocarbon production and are good candidates for geological carbon dioxide sequestration  
33due to their high porosity and permeability. These highly porous rocks undergo different modes  
34of deformation depending on their stress history as well as their lithological and petrophysical  
35properties. Strain in such sandstones may localise into thin (mm-scale) planar structures often  
36referred to as deformation bands [1-3]. The kinematics of deformation bands always includes  
37shear, while the volumetric response ranges from dilative to compactive strain (e.g. [1, 4]). Both  
38porosity and permeability are reduced within compaction bands and compactive shear bands, as a  
39result of pore-collapse and possible grain breakage [5-7]. In dilative shear bands, porosity  
40increases and (depending on pore tortuosity and changes in specific surface area) permeability  
41may decrease or increase [8-10]. Extensive experimental work has been carried out to study the  
42effects of variables such as stress level, porosity and grain size on localised deformation in well  
43lithified and cemented sandstone (e.g. [11-15]).

44 At the micro-scale (i.e. the scale of the grain, for a sandstone), strain corresponds to  
45combinations of different mechanisms including grains rearrangement (sometimes referred to as  
46“particulate flow”), grain breakage (cataclasis) and cement breakage. A full understanding of  
47strain localisation in sandstones requires investigation of these mechanisms, all of which strongly  
48depend on grain shape and angularity. Experience and knowledge from our previous work on  
49natural compactive shear bands formed in very porous and friable sandstone (e.g., [16, 17]) were  
50the motivation to explore the behaviour of sand at high mean stress. In this paper, sand is adopted  
51as a model material that allows the investigation (at substantially lower mean stresses than that

52needed for sandstone) of all deformation mechanisms mentioned above at the grain scale – with  
53the exception of cement breakage.

54 X-ray tomography is proving to be an ideal tool for the investigation of the micro-  
55mechanisms of deformation in sand – for example recent work from Laboratoire 3SR, Grenoble,  
56has clearly demonstrated the effect of grain angularity on the macroscopic mechanical behaviour  
57of sand as well its effect on the formation and development of shear bands in different types of  
58sand [18-20], all this at relatively low mean stress, which rules out grain breakage.

59 A series of dry triaxial compression tests on two different sands has been performed *in-situ*  
60(*i.e.* performing x-ray scans at various points throughout loading) inside the x-ray scanner in  
61Laboratoire 3SR (Grenoble), at mean stresses up to 7000 kPa – which proves to be high enough  
62to cause grain breakage. The two sands tested are both quartz sands, with similar grain size  
63distributions but different angularities: Ottawa 50-70 sand (rounded) and Hostun HN31 sand  
64(angular).

65 The structure of the paper is as follows: the materials and testing method are first described;  
66this is followed by a description of the tests performed. The typical, macroscopic measurements  
67obtained from triaxial testing are detailed for all tests. The image processing tools used to make  
68micro-scale measurements on the acquired x-ray images are then briefly described. These  
69measurements are further used to explain the differences in the macroscopic responses observed.  
70The paper closes with a discussion of the results obtained.

## 712. Experiments

### 722.1 Materials tested

73 The two different types of sand tested in this work are Hostun HN31 sand and Ottawa 50-70  
74sand. Hostun sand is angular siliceous sand (see SEM image in Fig. 1) produced in a quarry close  
75to the commune of Hostun in the Rhône-Alpes region of France. The Hostun HN31 sand studied  
76in this work [21] is mechanically identical to the “Hostun S28” and “Hostun RF” varieties that  
77have been thoroughly studied (e.g. [22]). The Ottawa sand is a rounded siliceous sand (see SEM  
78image in Fig. 1), that comes from sedimentary deposits in Ottawa, Illinois (U.S.A.). A particle  
79size distribution for both sands is presented in Fig. 2; the values of  $D_{50}$  of Hostun and Ottawa  
80sands are 338  $\mu\text{m}$  and 310  $\mu\text{m}$ , respectively.

### 812.2 Experimental setup and testing campaign

82 A series of triaxial tests on small specimens of dry Hostun and Ottawa sands at confining  
83pressures ranging from 100 kPa to 7000 kPa have been carried out at Laboratoire 3SR with *in-*  
84*situ* x-ray scanning. The specimens used in this work are cylinders of approximately 22 mm  
85height and 11 mm diameter, with non-lubricated end platens. Note that from a mechanical  
86standpoint, the response obtained with such small specimens compares well to that obtained with  
87larger (standard size) specimens (in [23]), as discussed in detail in [24].

88 The triaxial cell has no steel tie-bars to take the return force from the axial compression of  
89the specimen; this is instead taken by the cell in tension (see Fig. 3). The absence of steel tie-bars  
90allows the specimen to be imaged by x-rays, without disturbance, inside the cell. Two triaxial  
91cells were used in this experimental campaign: a high and a low pressure cell, which differ  
92principally in the thickness of the wall of the cell, as well as how the confining pressure is  
93applied. Both cells are made of PMMA (Plexiglas) which is transparent to visible light as well as

94to x-rays. In the lower pressure cell shown in Fig. 3 (designed for confinements up to 1000 kPa),  
95the confining fluid is water, and confining pressure is applied by compressed air (coming from a  
96compressor) with a pressure controller, allowing a fine control of the confining pressure. For the  
97higher pressure cell, confinement by air is no longer feasible, and so a pump is used; for  
98compatibility with the pump, the confining fluid is oil.

99 Given the different confining pressures and fluids, two different membranes have been used  
100in this experimental campaign. In the lower pressure tests, a 300  $\mu\text{m}$  thick latex membrane (very  
101transparent to x-rays) is used. In the higher pressure tests, however, a 500  $\mu\text{m}$  thick neoprene  
102membrane is used – primarily to avoid piercing of the membrane, but also for compatibility with  
103the confining fluid. The x-ray absorption of this membrane is considerably higher than the latex  
104one, so it is clearly visible in the images coming from x-ray tomography (see Fig. 6).

105 All specimens are prepared by air pluviation into the membrane stretched in a mould, aiming  
106for a dense initial packing (this specimen preparation technique has been selected for its  
107reproducibility). Once prepared, samples are installed into the triaxial cell with vacuum applied to  
108the sample. The cell is then filled with the relevant confining fluid, which is slowly pressurised to  
109an initial isotropic state, while the vacuum is released.

110 Each specimen is loaded isotropically until the desired confining pressure is reached, at  
111which point deviatoric loading is applied under strain control by shortening the sample axially  
112using a domed ram driven up by the loading system. The force required to advance the ram is  
113recorded with a load cell, and the displacement of the ram is recorded with an LVDT. The  
114specimen is shortened with a displacement rate of 21  $\mu\text{m}$  per minute (corresponding to a nominal  
115strain rate of just under 0.1% per minute).

116 At various key points during a test, loading is halted, and a tomographic scan is performed  
 117by acquiring 1024 x-ray radiographs of the sample as it is rotated 360° around its vertical axis by  
 118the rotation stage visible in Fig. 3. Since the specimens scanned are relatively small, the  
 119geometrical zoom provided by the x-ray cone-beam coming from the generator gives a pixel size  
 120of 15.6 µm/pixel, meaning that each grain within the specimen (we recall the  $D_{50}$  is bigger than  
 121300 µm for both sands) is clearly represented. The set of radiographs acquired is then  
 122reconstructed (using DigiCT 2.4.2 from Digisens) into a 3D field of the x-ray attenuation inside  
 123the specimen.

### 1243. Macroscopic results of triaxial testing

125 This section presents the macroscopic results coming from the triaxial compression tests  
 126analysed in this work (see Table 1).

127**Table 1. Summary of tests analysed in this paper**

<b>Material</b>	<b>Test Name</b>	<b>Confining Pressure</b>	<b>Initial Porosity (before shearing)</b>
Ottawa	OUEA06	100 kPa	32.1%
Ottawa	OHEA03	4000 kPa	29.1%
Ottawa	OHEA01	7000 kPa	27.3%
Ottawa	OHEA02	7000 kPa	28.9%
Hostun	HNEA01	100 kPa	37.7%
Hostun	HHEA03	1000 kPa	33.7%
Hostun	HHEA05	1000 kPa	34.8%
Hostun	HHEA04	2000 kPa	35.1%
Hostun	HHEA06	3000 kPa	34.3%
Hostun	HHEA02	4000 kPa	34.6%
Hostun	HHEA01	7000 kPa	33.8%

128 In some tests, a few scans are performed during the isotropic loading of the specimen,  
 129however most are during deviatoric loading. When deviatoric loading is halted to perform a scan,  
 130some axial stress relaxation occurs, which can be seen as small drops of deviator stress

$$p = \frac{1}{3}(\sigma_a + 2\sigma_r)$$

131 throughout the  $q/p$  vs.  $\epsilon_a$  responses of both sands (see Figs. 4 and 5 noting that

132 and  $q = (\sigma_a - \sigma_r)$  with  $\sigma_a$  and  $\sigma_r$  being the axial and radial stresses respectively).

133 Figures 4 and 5 show the deviator stress response normalised by the mean normal stress ( $q/p$ )  
134 and volumetric strain response (in %) both against axial shortening (normalised by the initial  
135 height) for all the tests on Ottawa and Hostun sands. Since samples are tested dry, the  
136 macroscopic measurement of bulk specimen volume that is used to measure volumetric strain is  
137 actually derived from the images of the specimen – the technique for making this measurement is  
138 detailed in Section 4.1.

139 It is clear from the responses shown for both sands that there is a significant, but progressive  
140 change between the macroscopic material response at 100 kPa and 7000 kPa confinements. The  
141 triaxial compression behaviour of Ottawa sand (shown in Fig. 4) with increasing mean stress  
142 reveals a progressive retardation (with respect to axial shortening) of the peak deviatoric stress as  
143 well as a progressive reduction of its value. The difference between the peak deviator stress and  
144 the plateau is large at low confinement (test OUEA06), and the difference reduces with  
145 increasing confining pressure – as does the normalised  $q/p$  value of the plateau. The considerable  
146 dilation undergone by the specimen at 100 kPa confinement progressively reduces from 4000 to  
147 7000 kPa. Although the volumetric strain curves obtained for the two tests performed at 7000 kPa  
148 confinement differ, the response can be seen to be slightly dilatant (compared to test OUEA06 at  
149 100 kPa) in both cases.



150 The evolution of the mechanical response of Hostun sand (Fig. 5) with increasing confining  
151 pressure has the same sort of evolution as with the Ottawa sand described above (less clear peak  
152 stress and increasingly less dilatant behaviour), however, over the same range of confining  
153 pressures this evolution appears to be considerably more rapid: a peak in deviator stress is only  
154 clearly visible until 3000 kPa confinement – at higher confinement there is no peak, only strain  
155 hardening. The volumetric response also displays a good deal more change than for Ottawa sand:  
156 the specimen tested at 100 kPa confinement shows clear dilatancy, whereas the one tested at 7000  
157 kPa confinement is contractant throughout. The other tests at 1000, 2000, 3000 and 4000 kPa fall  
158 in between these two cases. The stress response of HHEA05 (1000 kPa confinement) also reveals  
159 a clear peak, yet with a lower dilatancy than HNEA01 at 100 kPa confinement. Stress-strain  
160 responses of HHEA04 at 2000 kPa and HHEA06 at 3000 kPa confinement also show peaks but  
161 not as clearly marked as for HNEA01 and HHEA05. Their volumetric responses are very slightly  
162 dilatant in the case of HHEA04, and slightly contractant for HHEA06. Stress-strain response of  
163 sample HHEA02 tested at 4000 kPa confinement reveals no peak, and shows contraction, but to a  
164 lesser extent than test HHEA01 at 7000 kPa.

165 During the post-test removal of specimens tested at high pressures, the production of fines  
166 was noted when the contents of the membrane was emptied for weighing. The production of fines  
167 by grain breakage may well explain the change from dilation to compaction in the macro-scale  
168 volumetric responses of these sands (see Figs. 4 and 5).

#### 169 **4. Image analysis**

170 This section briefly outlines the image analysis techniques used to make micro-scale  
171 measurements on the various 3D images coming from the tomographic scans during loading. The  
172 objective is to use these tools to explain the differences in the macroscopic responses detailed

173above. The 3D images coming from tomography are 16-bit greyscale measuring  
1741250x1250x1600 pixels, with a pixel size of 15.6  $\mu\text{m}$ .

#### 175**4.1 Preparation of images for analysis**

176 In order to make micro-scale measurements from the acquired images, the first step is to  
177define the domain (within the image) of the solid skeleton to be analysed, so that measurements  
178are made only on this part of each image, and that other objects (such as the top and bottom  
179platens, as well as the neoprene membranes) are not taken into account. The specimen-platen  
180interface is considered to be a plane, which can be tilted with respect to the specimen's axis.  
181Given that the platens have a long extent of flat greyscale (as opposed to the sample, which is  
182made of grains and pores), this information is incorporated into an automatic procedure which  
183automatically detects points on this interface and fits a plane to the two specimen-platen  
184interfaces.

185 The shape of the membrane is rendered more complex by the fact that it does not necessarily  
186have a uniform thickness (especially once the sample starts to undergo localised deformation);  
187consequently, there is no question of manually removing the membrane, especially taking into  
188account the large number of 3D images acquired. A specific image processing technique is  
189therefore developed to recognise the inner surface of the membrane: for each horizontal slice, 300  
190equally-spaced radial profiles are made, centred on the approximate axis of the specimen. As  
191illustrated in Fig. 7, the plot of the variance of this profile reveals sharp peaks, corresponding to  
192the rapidly changing greyscale values at material interfaces. Each greyscale profile is variance  
193filtered and the characteristic peaks are used to identify the border points: a closed border  
194defining the inside of the membrane is then created by connecting these 300 individual points by  
195linear segments. Finally, a 3D median filter (of radius 5 pixels) is used to smooth out this border.

196The full technique is shown in Fig. 7, and its effectiveness can be seen from the way in which the  
197membrane visible in Fig. 6b is removed in Fig. 6c.

198 After each individual stage has been processed, the inside of the specimen is defined with a  
199geometrically complex border around the inside of the membrane bounded by two planes. The  
200number of voxels (3D pixels) within this space can be counted, using the pixel size, and used to  
201calculate the bulk volume of the specimen as imaged.

## 2024.2 Local measurement of porosity

203 Porosity is a key measurement for granular materials, and its spatial distribution is of  
204particular interest given the different volumetric strain responses seen with macro-scale  
205measurements. Porosity is defined as the ratio of the volume of voids to the total volume. This  
206can be measured locally, by defining a 3D subvolume within a 3D image and measuring the  
207volume of the voids within the subvolume. In previous work [26], voids have been counted by  
208*binarising* the greyscale 3D image into a black and white image of the solid and void phases by  
209applying a threshold. Observation of the images obtained in this work, particularly for Hostun  
210sand at high confining pressure and after shearing shows that grain breakage does indeed occur  
211(see Fig. 15 for example), and that the finer grains produced can be small compared to the pixel  
212size of the images. This smaller grain phase partially fills voxels, and consequently looks like a  
213homogeneous material of x-ray attenuation in between grain and void, thus invalidating the  
214inherent two-phase hypothesis for binarisation. In this work, the volume of voids in each  
215subvolume is calculated on the greyscale (as opposed to binary) 3D images; values of pore and  
216grain greyscale are measured manually on a small selection of pores and grains throughout the  
217specimen. Any voxel having a greyscale value equal to, or higher than, the grain greyscale value  
218identified is 100% solid, and any voxel having a greyscale value equal to, or lower than, the pore

219greyscale value identified is 100% pore. Greyscale values between these two greyscale limits are  
220interpolated with a linear relationship.

221 Local measurements of porosity are therefore made by defining local subvolumes, centred on  
222a number of regularly spaced nodes. The size of the cubic subvolumes is selected as a reasonable  
223trade-off between sensitivity and representativity of the measurement. Subvolume sizes of  
224620x620x620  $\mu\text{m}$  and 470x470x470  $\mu\text{m}$  are used to make measurements on Hostun and Ottawa  
225sand respectively.

### 2264.3 Digital Image Correlation (DIC)

227 In order to make measurements of local kinematics between two 3D images of a deforming  
228specimen, Continuum Digital Image Correlation, as implemented in Tomowarp by Hall [27], has  
229been used to follow regularly-distributed cubic subvolumes between two different greyscale  
230images. The method relies on image correlation and attempts to find a pattern (i.e. the greyscale  
231inside a given subvolume) extracted in the reference configuration, and looks for this pattern in  
232the deformed configuration. When the best matching pattern is found (by optimizing a correlation  
233coefficient mapping one image to the other), the displacement of the subvolume is a natural  
234output, and is measured to subpixel precision. This gives the displacement of a series of points  
235spread throughout the reference configuration, giving therefore, a full-field measurement of the  
236kinematics between the two states. By deriving this displacement field, the 3D strain tensor can  
237be obtained. The first two invariants of the strain tensor (representing volumetric and shear strain)  
238are chosen for display in the results shown in Section 5.

239 Image correlation has an increased probability of making matching errors when a  
240considerable amount of grain breakage occurs between steps. This is due to the considerably

241different patterns from one image to another that naturally make the patterns harder to match. In  
242order to minimize these potential errors, small increments are analyzed in this work. It is worth  
243noting that DIC is used incrementally, i.e. to measure the displacement field from image i-1 to  
244image i, rather than from the initial configuration up to image i. This is different from the “total”  
245porosity measurements presented in Section 5.

## 246**5. Measurements from image analysis**

### 247**5.1 Tests on Ottawa sand**

248 This section uses the 3D images acquired during the different tests on Ottawa sand to  
249investigate the micro-mechanisms at play during triaxial shearing of the specimens tested at 100,  
2504000 and 7000 kPa confining pressure.

251 Figure 8 shows vertical slices from the final states imaged in each test on Ottawa sand  
252analysed in this work. The slices are oriented to contain the axis of the sample and the normal to  
253the single shear band that is apparent in all cases. In all images acquired at high pressure, the  
254membrane and platens have been removed using the technique described in Section 4.1. These  
255images reveal that in OUEA06 a clear dilatant shear band crosses the sample, whereas for the two  
256samples tested at 7000 kPa confinement, a narrow compactive shear band is also visible,  
257containing crushed material. OHEA03 is an intermediate case, i.e. with no change or slight  
258change in porosity inside the shear band.

259 Figure 9 shows the porosity maps obtained for some key steps during the triaxial shearing of  
260three of the specimens. Prior to shearing, all specimens have relatively uniform distribution of  
261porosities ranging from  $n=27\%$  to  $n=32\%$ . Specimen OUEA06 (tested at 100 kPa confinement)  
262presents a clear, dilatant shear band that develops early in the test – at 3.9% axial shortening

263(around the position of the peak stress) localised dilation is visible, with a porosity in the band of  
264around  $n=46\%$  by the end of the test. OHEA03 (4000 kPa) does not seem to show localised  
265changes in porosity at 3.9% axial strain, however around its peak (at 7.7% shortening, which is  
266considerably later than OUEA06), localised dilation is visible. This dilative band continues to  
267develop with increasing shearing, and becomes more pronounced by the end of the test (reaching  
268a value of  $n=39\%$ ). The sample tested at 7000 kPa confinement (OHEA01) has a considerably  
269different behaviour, which is captured well by looking at the porosity maps: by the end of the  
270test, a band of *reduced* porosity is noticeable, reaching  $n=33\%$  (2% lower than the surrounding  
271material). Looking back towards the beginning of the test, two mechanisms can be seen: there is a  
272clear and relatively uniform densification of the sample between 0 and 4.3% shortening.  
273Thereafter, a dilatant shear band is visible at 12.9% shortening (achieving a porosity of  $n=36\%$ ),  
274and at 14.3% shortening, *within* this *dilatant* band, a zone of contraction starts to develop at the  
275bottom of the band (on the right side of the specimen), and appears to progressively develop  
276within the dilatant band as shearing continues: by 17.2% shortening it crosses the entire  
277specimen. This contractive band is consistent in space with the zone of crushed material visible in  
278Fig. 8.

279 Figure 10 shows the DIC results (vertical sections of the calculated fields of volumetric and  
280shear strain) for some selected increments of the three tests analysed. Interestingly, incremental  
281volumetric strain fields can reveal mechanisms that changes in total porosity simply are not  
282sensitive enough to pick up. While over the peak the volumetric strain fields from DIC merely  
283confirm the dilating bands shown by the porosity fields, the increments analysed at the end of the  
284tests reveal that as shearing continues, the volumetric strain in the bands of localised shear strain  
285either disappears (for the test at lowest confinement) or becomes compactive (in all the other

286tests), likely due to grain breakage. The shear strain fields show that in all tests the shear band  
287gets thinner after the peak.

## 288**5.2 Tests on Hostun sand**

289 Figure 11 shows vertical slices from the final states imaged in each test on Hostun sand  
290analysed in this work. The slices are oriented to contain the axis of the sample and the normal to  
291the single shear band (when this is apparent).

292 As for Ottawa, in all images acquired at high pressure, the membrane and platens have been  
293removed using the technique described in Section 4.1. These images reveal a range of final states  
294that go from a high porosity band (at 100 kPa and to a lesser extent at 1000 kPa confinement) to a  
295wide band with clear evidence of crushed grains (in the tests at 4000 and 7000 kPa). The tests at  
2962000 kPa and 3000 kPa confinement are intermediate cases, with no striking changes in porosity  
297throughout the specimen.

298 Figure 12 shows the porosity maps obtained for some key steps during the triaxial shearing  
299of the specimens of Hostun sand. Prior to shearing, all specimens have relatively uniform  
300distribution of porosities ranging from  $n=27\%$  to  $n=32\%$ , with initial porosity generally  
301decreasing with increasing confining pressure, as expected. Specimens HNEA01 and HHEA05  
302both exhibit a clear shear band that becomes increasingly dilatant with increasing shortening –  
303with the out-of-band material remaining at almost constant porosity. Specimen HHEA04 tested at  
3042000 kPa confinement seems to deform with essentially no porosity change. A progressive and  
305distributed reduction in porosity is evident in all specimens tested at higher cell pressure.  
306Specimen HHEA02 shows some degree of localisation in porosity reduction at the end of the test.

307 Figure 13 shows the DIC results (vertical sections of the calculated fields of volumetric and  
308 shear strain) for some selected increments of the three lower pressure tests. At 100 kPa and 1000  
309 kPa confinement, (shear and volumetric) strain localisation starts in a wide band and then  
310 progressively concentrates with increasing axial shortening. At 2000 kPa, the shear strain  
311 concentrates in a similar fashion, while the maps of incremental volumetric strain confirm that  
312 little or no volume changes occur during (progressively localised) shearing. The DIC results for  
313 the other three tests analysed (shown in Fig. 14) show that a shear band eventually forms in all  
314 tests – which was not evident from either x-ray images or the porosity maps. The reason for this  
315 can be seen in the volumetric strain increments presented, which show no localisation. It is also  
316 important to note that at these levels of confinement, volumetric strain is essentially compactive  
317 rather than dilative – consistent with the global volumetric strains shown in Fig. 5.

## 318 6. Discussion and conclusions

319 The range of cell pressures used in this experimental campaign has resulted in a significant  
320 evolution of the mechanical response of both tested sands, at the macroscopic level. The  
321 tendency for increasing confinement is for the volumetric response to change from dilative to  
322 contractive, and for the peak of the deviator stress to become less pronounced. This is entirely  
323 consistent with previous experimental findings (e.g. [28-30]), all of which attribute this change in  
324 macroscopic behaviour to the appearance of grain crushing with increasing mean stress – which  
325 is supported by post-mortem sieve analyses.

326 Grain crushing is generally studied through its numerous effects at the macro scale (e.g. its  
327 effect on compressibility, shear strength, permeability, etc.). In this work, the process of grain  
328 crushing is approached experimentally at the scale of the grain. X-ray micro tomography allows  
329 imaging of this process, which is a major mechanism of inelastic deformation in sand at high



330pressure – along with granular rearrangement. Furthermore, the comparison of successive pairs of  
331x-ray images (with 3D DIC) yields 3D (incremental) strain fields, which reveal the nature of the  
332strain occurring between the two states.

333 In all the tests performed on Ottawa sand, shear strain and volumetric strain are observed to  
334localise into a band a few grains thick. At high pressure, grain crushing only occurs in the shear  
335band when a sufficiently high porosity is reached. It should be mentioned that this evolution is  
336well portrayed by the first three stages (dilation, pore collapse and grain size reduction) in the  
337sequence suggested by Lothe *et al.* [30] to describe the evolution of deformation bands in  
338sandstones.

339 DIC measurements in Hostun sand show that while shear strains localise in a shear band at  
340all pressures, volumetric strain is localised when dilatant (at low pressures) and becomes  
341increasing less localised in the contractive regime. X-ray images show diffuse breakage at high  
342pressure, with the relatively wide regions of porosity reduction corresponding in space to regions  
343of crushed grains (see Fig. 15).

344 Since both Ottawa and Hostun are quartz sands with similar grain size distribution, the cause  
345of their different responses can be mainly ascribed to the shape of their grains (rounded in the  
346former and angular in the latter). One may hypothesise that in the more angular material the  
347amount of energy required to break a grain is lower due to the presence of sharper contacts that  
348concentrate stresses on the grain. Therefore, for a given stress level one would expect more  
349crushing in the angular material – which is exactly what is observed. The massive grain crushing  
350observed in Hostun sand at 7000 kPa confinement suggests that the material is close to the stress  
351conditions required for distributed grain breakage. In Ottawa sand, even at 7000 kPa confinement

352breakage only occurs after a strong reduction of porosity, which for a given stress increases  
353interparticle forces – this happens only inside the narrow compactive shear band of localised  
354strain (see Fig. 15). These differences manifest themselves in the macroscopic response of the  
355two sands, most evidently in their volumetric response – with the specimens of Ottawa sand  
356never showing overall compaction whereas Hostun crosses this threshold between 2000 and 3000  
357kPa confinement.

358 The findings of this study on sand highlight the central role played by grain shape on both  
359the stress level needed to break grains as well as the patterns of deformation (localised vs.  
360diffuse). It is the authors' contention that these findings are also largely applicable to poorly  
361lithified sandstones. These results are of particular interest for hydrocarbon production and CO<sub>2</sub>  
362storage in sandstone, where the overall reservoir permeability and its evolution is key for the rate  
363at which extraction/injection processes can occur. In the triaxial compression tests analysed in  
364this work, similar materials differing only by the shape of their grains exhibited very different  
365distributions of particle breakage, diffuse in the angular material and highly localised in the  
366rounded one – with obvious implications for the permeability and its directional variation. The  
367stress paths encountered in a natural reservoir are clearly quite different from triaxial  
368compression, however one might expect grain shape to play an equally important role under any  
369deviatoric stress path, such as those induced by oil production or CO<sub>2</sub> injection.

## 370**Acknowledgments**

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374quantitative analysis and interpretation of the test results.

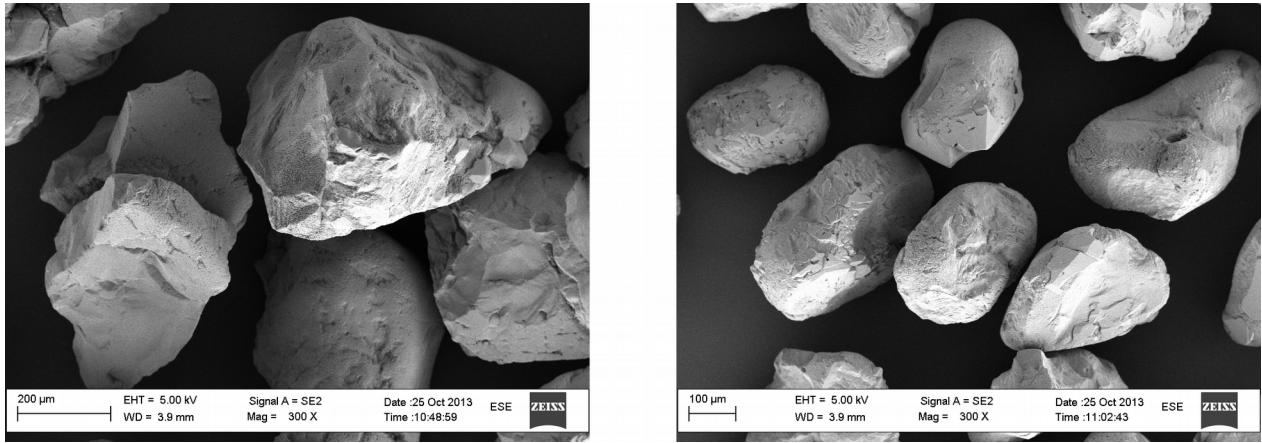
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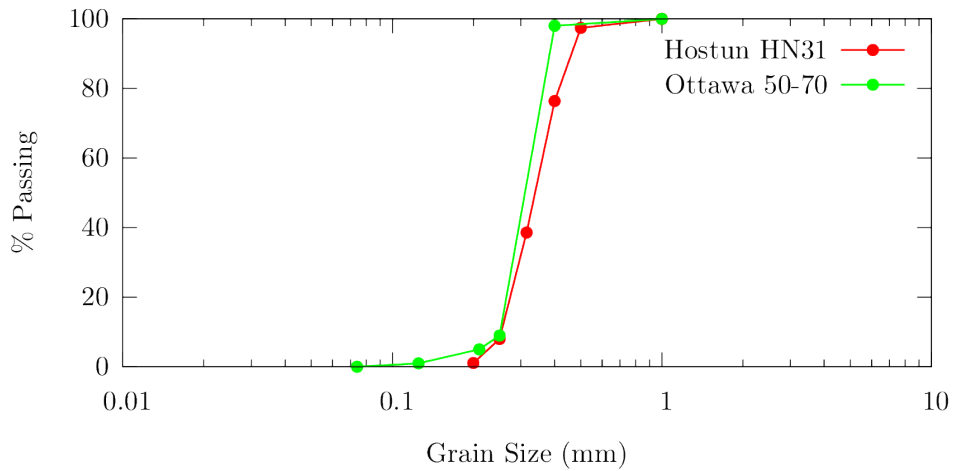
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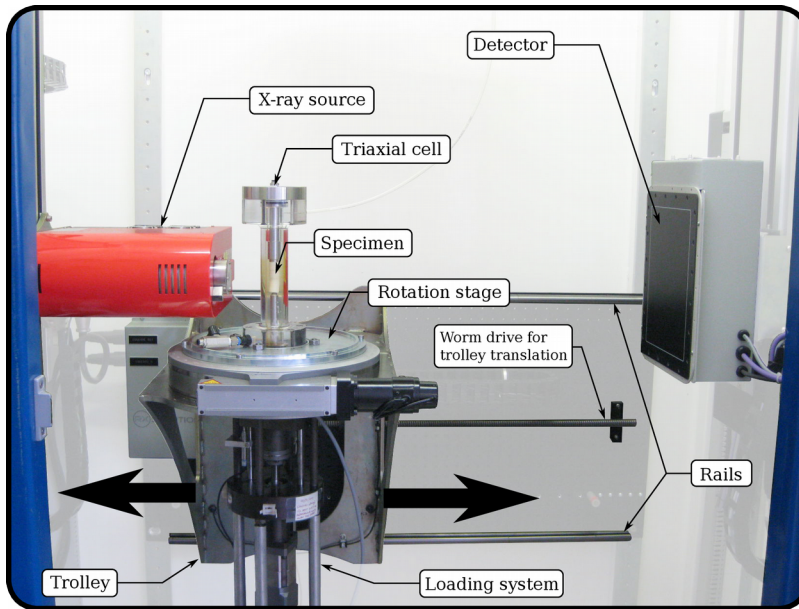
451**Figures**



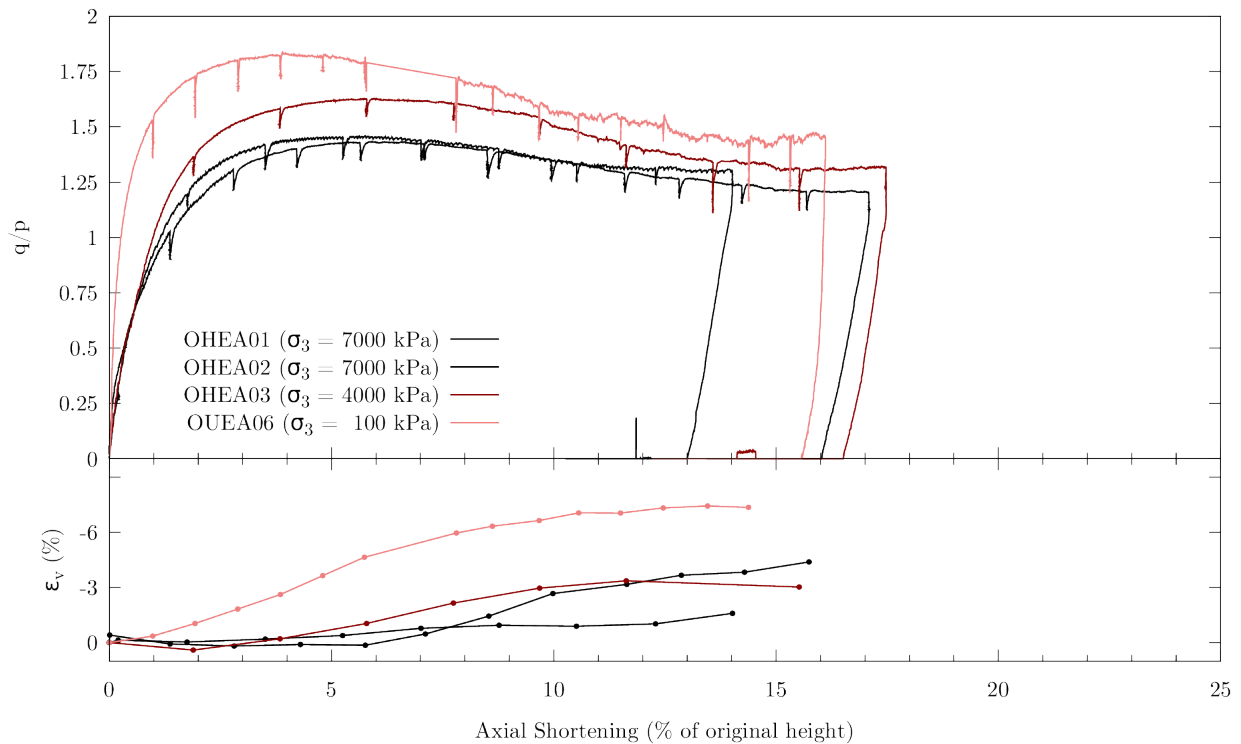
452**Fig. 1** Scanning Electron Microscope (SEM) images of: left) angular Hostun sand grains, and  
 453right) rounded Ottawa sand grains



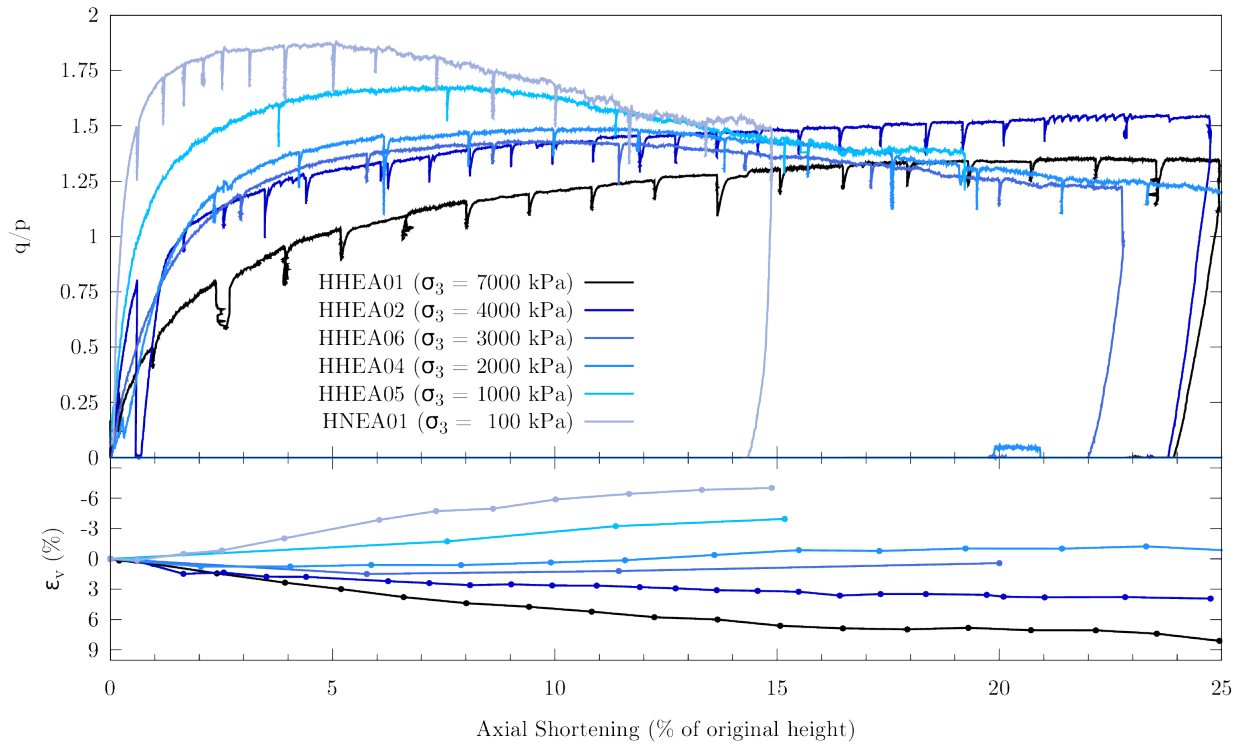
454**Fig. 2** Grain size distribution curves for the two sands studied in this work. Data for Hostun sand  
 455comes from the manufacturer [21], and data for Ottawa sand from Kim and Santamarina [25]



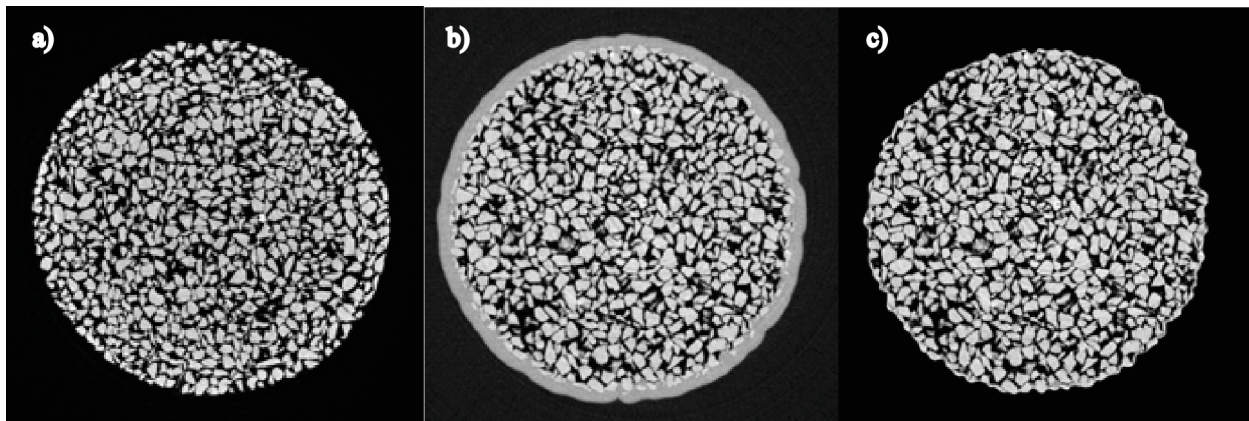
456**Fig. 3** Labelled photograph of the Laboratoire 3SR x-ray scanner, with background faded out for  
 457clarity



458**Fig. 4** Deviatoric stress normalised by the mean stress ( $q/p$ ) vs. axial shortening (top) and  
 459volumetric strain vs. axial shortening (bottom) for triaxial compressions tests on Ottawa sand

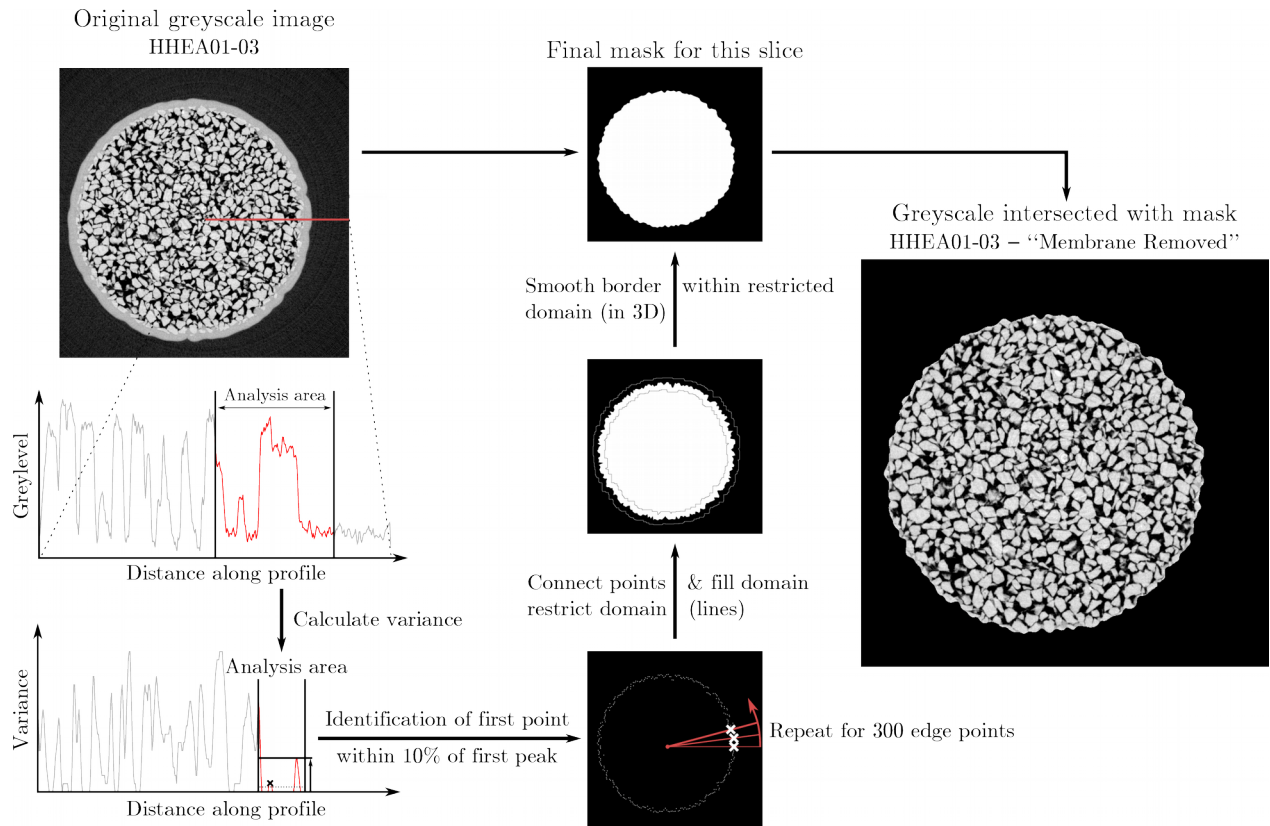


460**Fig. 5** Deviatoric stress normalised by the mean stress ( $q/p$ ) vs. axial shortening (top) and  
 461volumetric strain vs. axial shortening (bottom) for triaxial compressions tests on Hostun sand

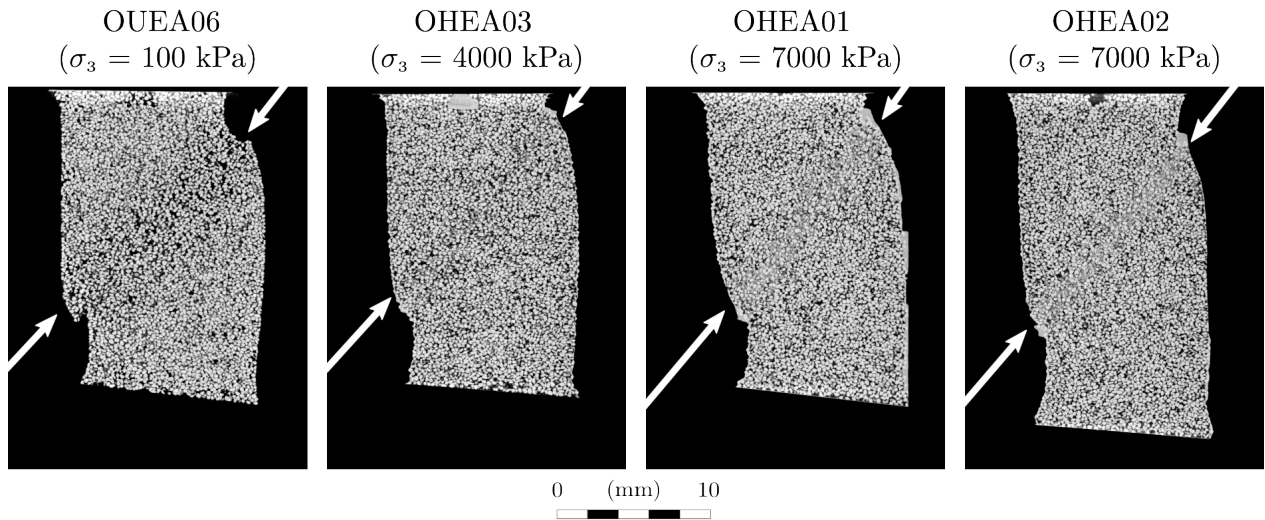


462**Fig. 6** Horizontal slice of a specimen of Hostun sand a) under low confining pressure, b) and c)  
 463under high confining pressure but before and after membrane removal, respectively



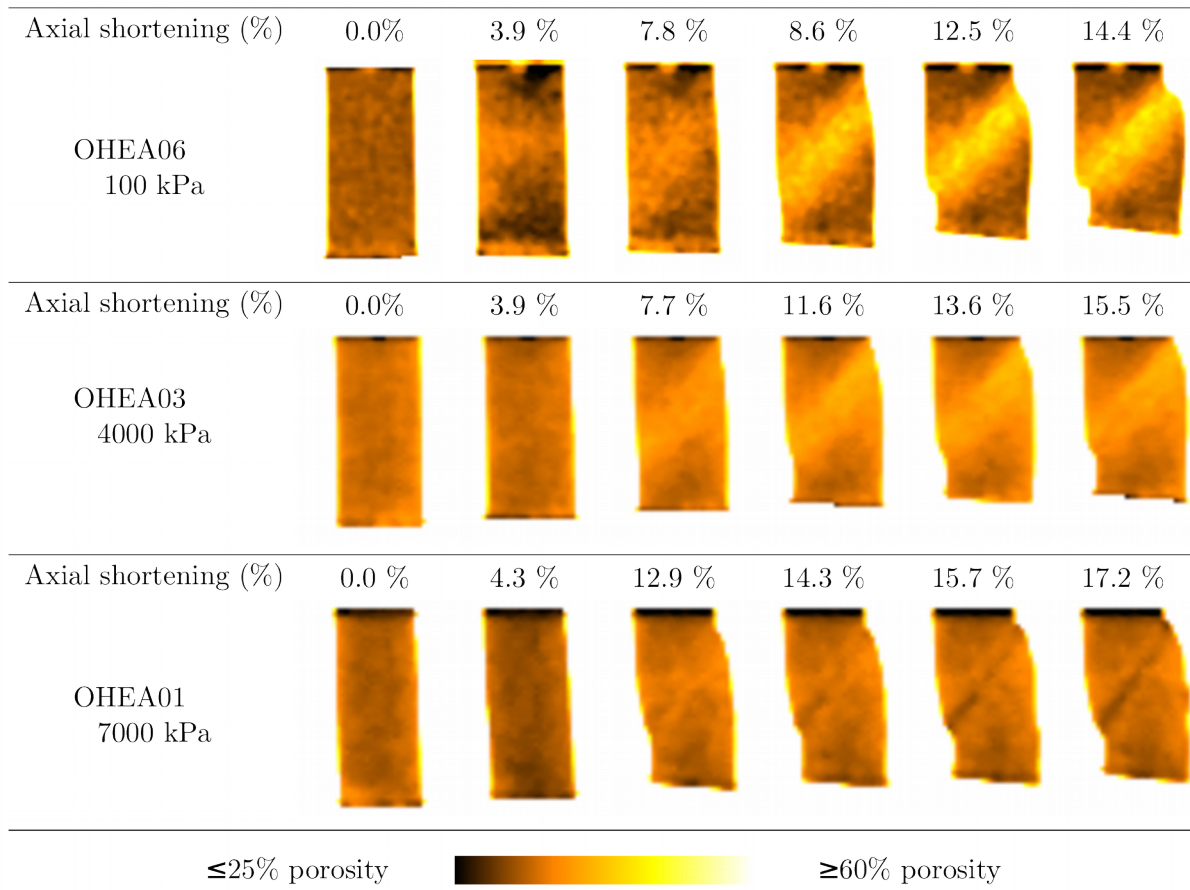


464**Fig. 7** Illustration of the technique developed for the identification and removal of the membrane  
 465from the 3D images acquired with the high pressure cell setup

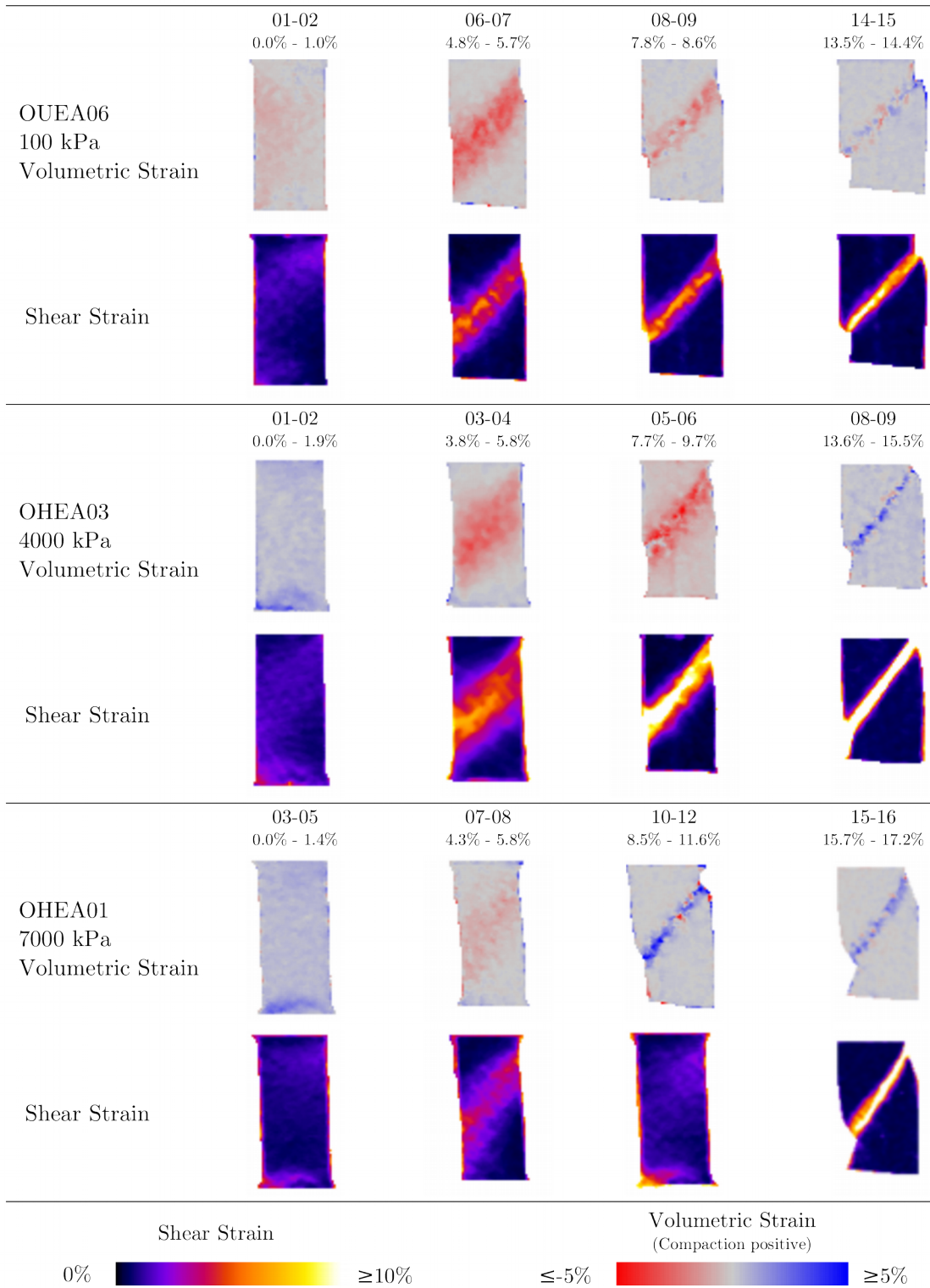


466**Fig. 8** Vertical slices through the last image acquired in each test on Ottawa sand

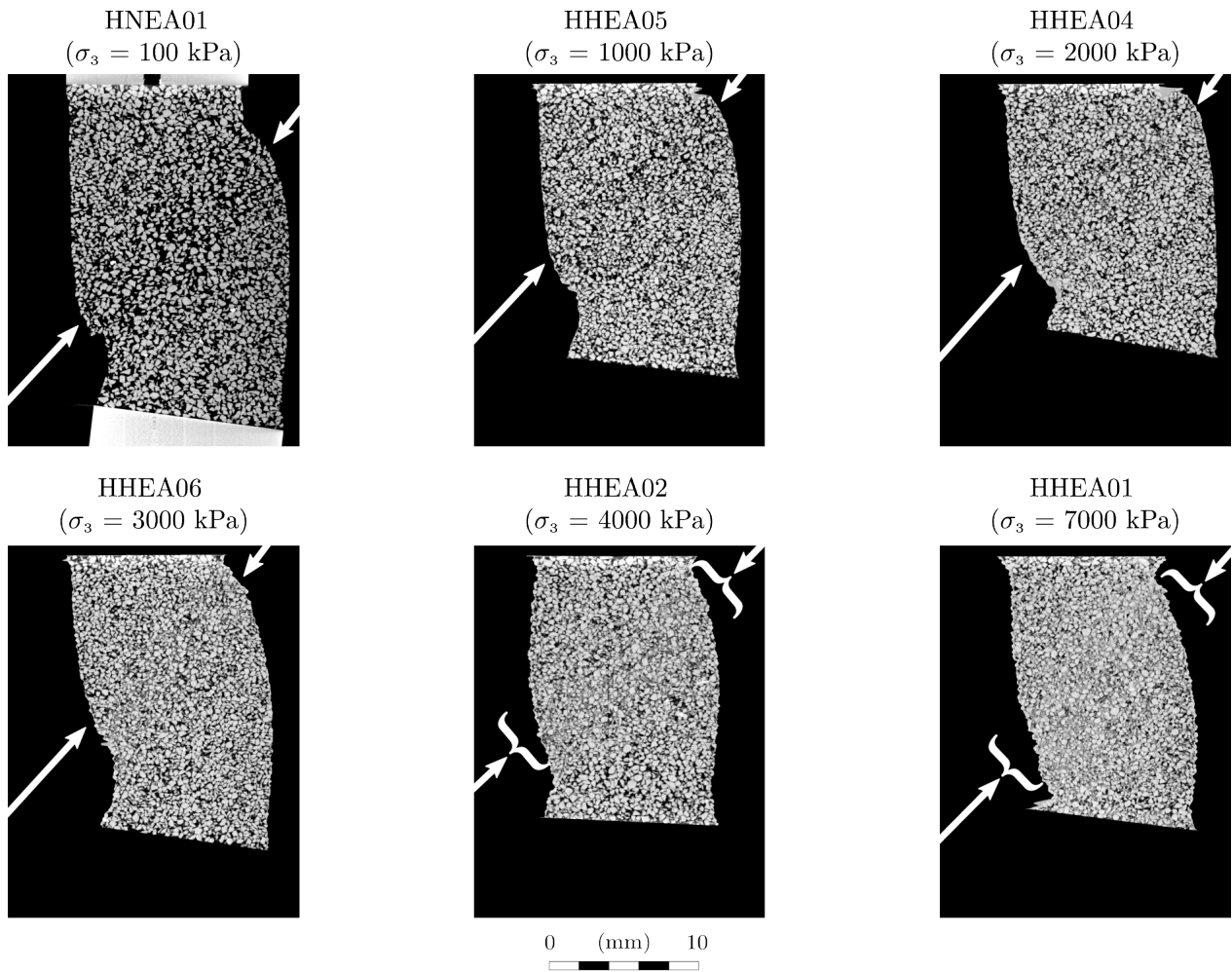




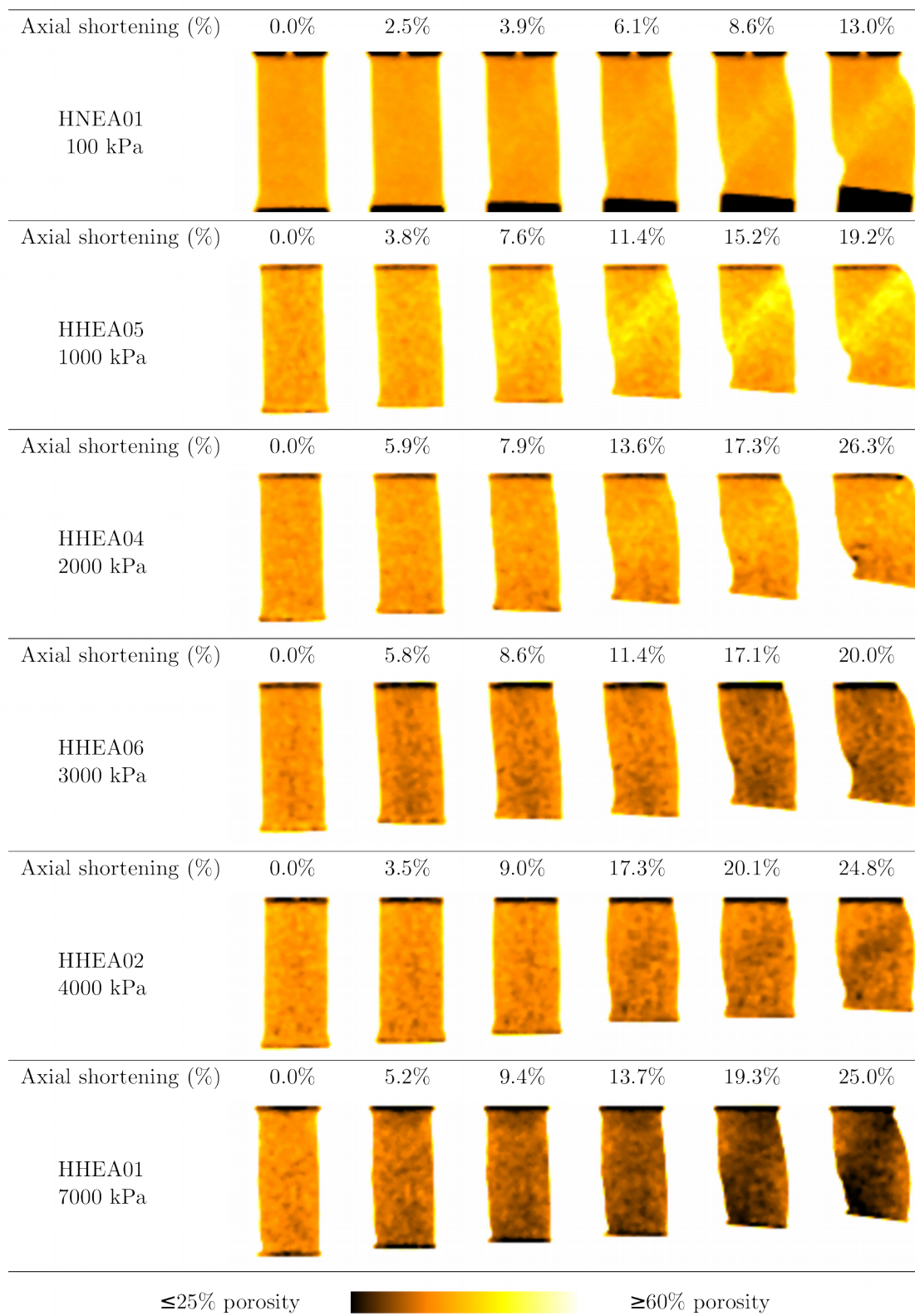
468**Fig. 9** Porosity maps of some selected states of the specimens of Ottawa sand during shearing



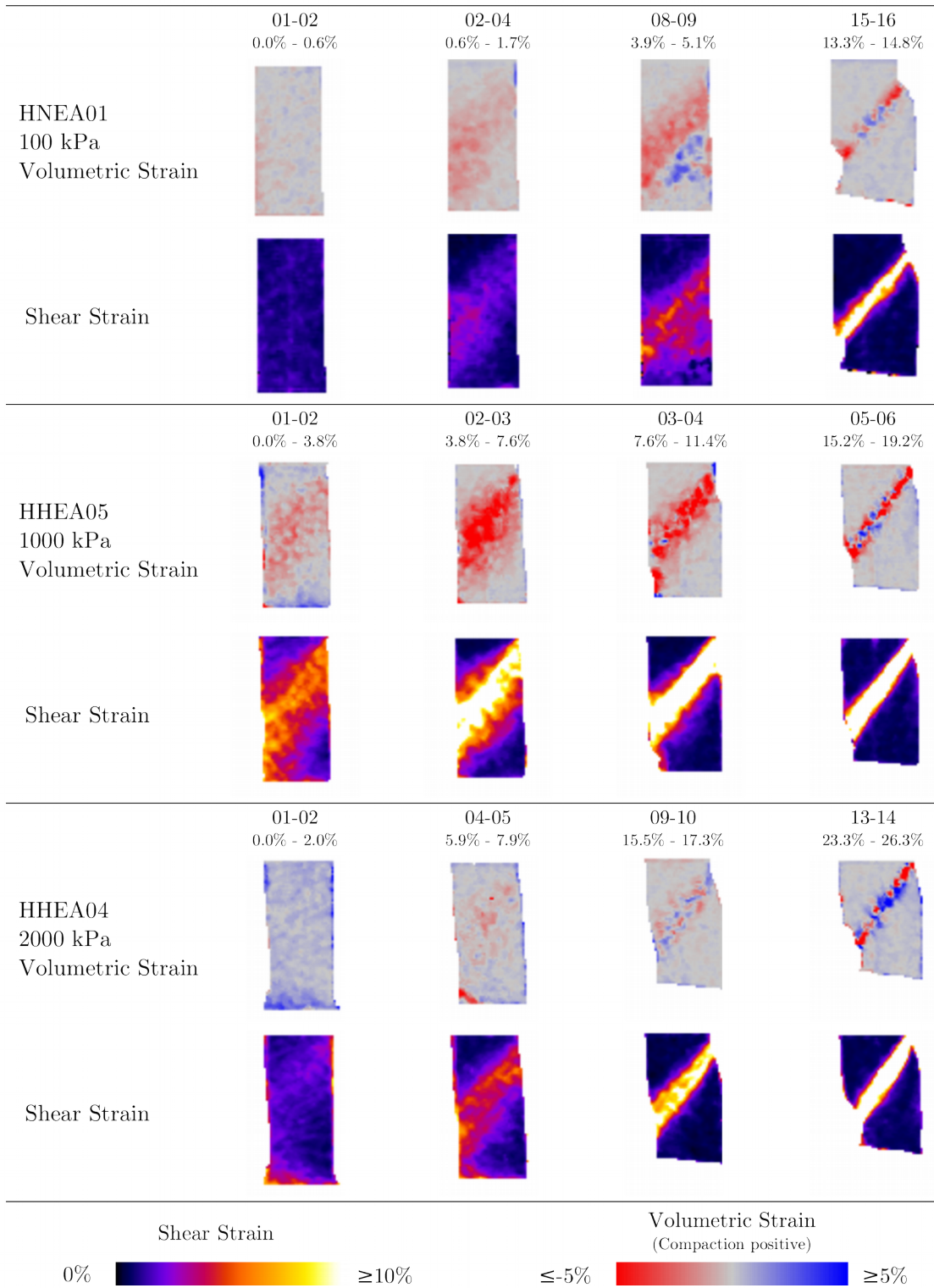
469**Fig. 10** DIC Results for OUEA06, OHEA03 and OHEA01



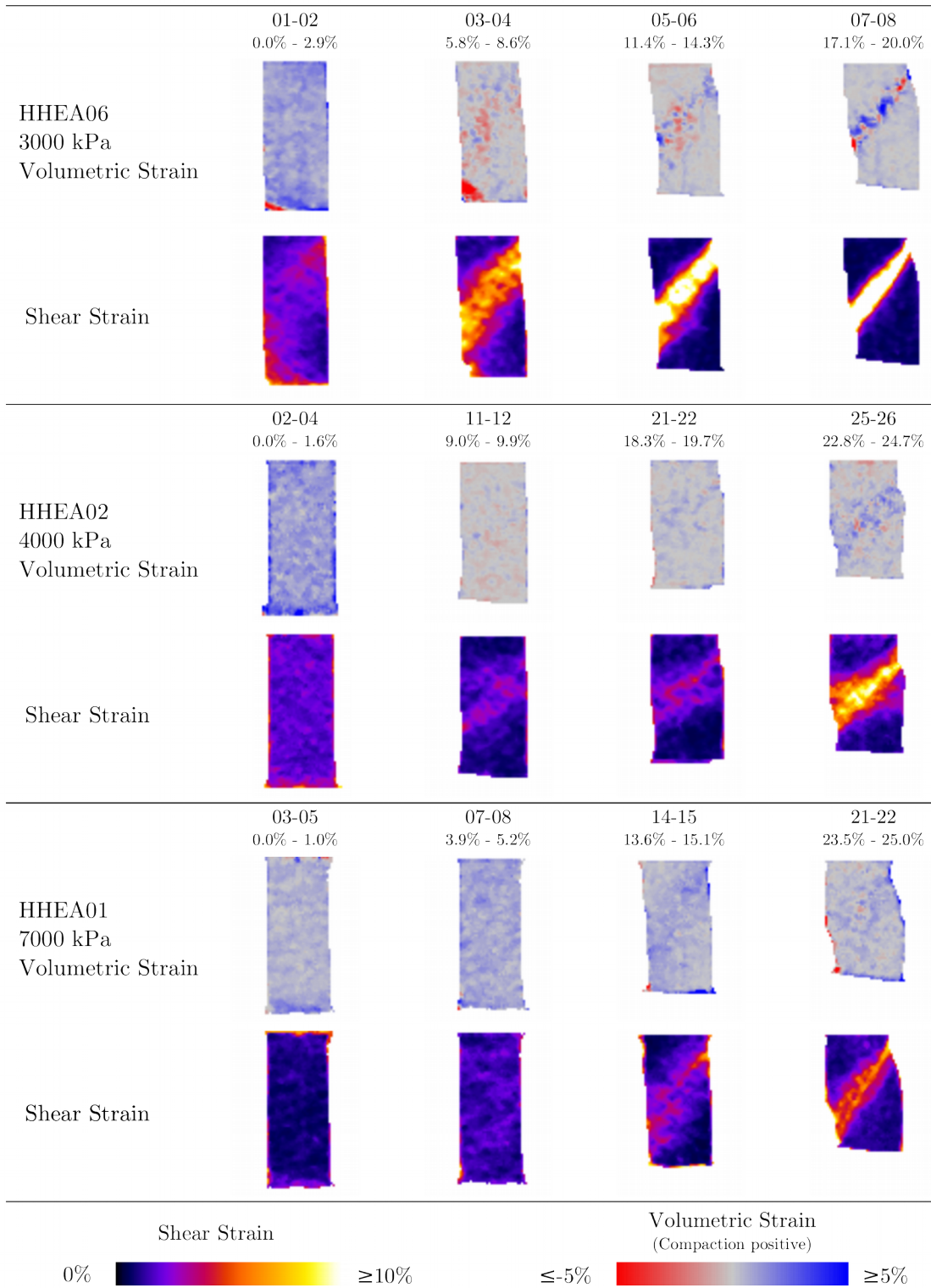
470**Fig. 11** Vertical slices through the last image acquired in each test on Hostun sand



471Fig. 12 Porosity maps of some selected states of the specimens of Hostun sand during shearing

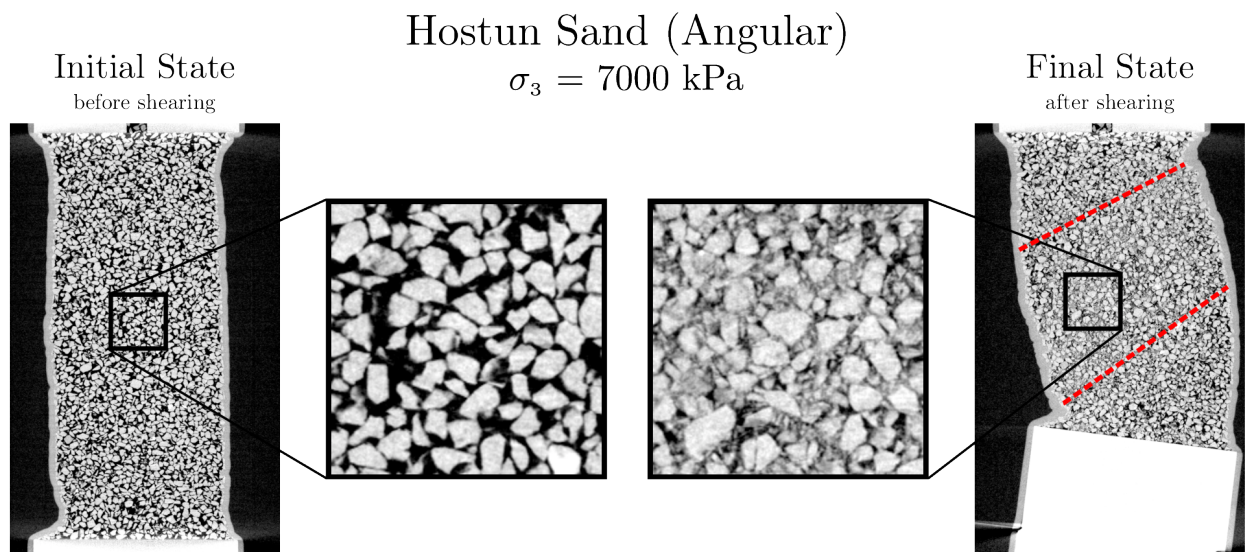
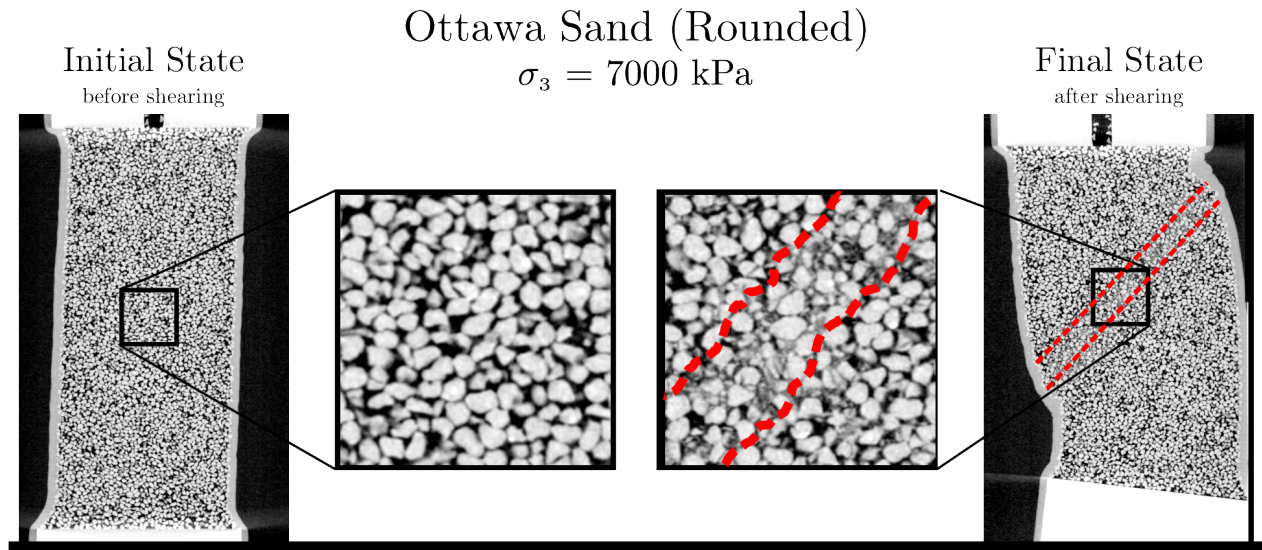


472**Fig. 13** DIC Results for tests HNEA01, HHEA05 and HHEA04



473**Fig. 14** DIC Results for tests HHEA06, HHEA02 and HHEA01





474**Fig. 15** Vertical sections of Hostun and Ottawa sands (under 7000 kPa confinement) with  
 475highlighted zones of intense grain crushing.