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# Fast 4D imaging of fluid flow in rock by high-speed neutron tomography

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## Key Points:

- Full 3D fluid front velocity map can be obtained from high-speed (1 minute/tomography) neutron tomography during water invasion into air-filled samples
- Quantitative measurements are validated by comparing experimental results to 1D analytical solution of pressure-driven flow
- During imbibition, compactant shear bands result in locally higher fluid-flow velocity due to decreased pore size in the bands

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18 **Abstract**

19 High-speed neutron tomographies (one minute acquisition) have been acquired during  
 20 water invasion into air-filled samples of both intact and deformed (ex-situ) Vosges sand-  
 21 stone. 3D volume images have been processed to detect and track the evolution of the  
 22 waterfront and to calculate full-field measurement of its speed of advance. The flow pro-  
 23 cess correlates well with known rock properties, and is especially sensitive to the distri-  
 24 bution of the altered properties associated with observed localised deformation, which  
 25 is independently characterised by Digital Volume Correlation (DVC) of x-ray tomogra-  
 26phies acquired before and after the mechanical test. The successful results presented herein  
 27 open the possibility of in-situ analysis of the local evolution of hydraulic properties of  
 28 rocks due to mechanical deformation.

29 **1 Introduction**

30 Deformation in rocks is typically not homogeneous and is often localised into fea-  
 31 tures such as shear or compaction bands. Such deformation can have significant influ-  
 32 ence on the hydraulic properties of the rock because it locally alters the rock structure  
 33 in ways that are expected to change the local flow properties. Standard experimental  
 34 approaches to assess deformation and changes in fluid flow in rocks are based on mea-  
 35 surements of fluxes and pressures taken at the boundaries of a tested sample. These mea-  
 36 surements, however, cannot provide information on internal heterogeneities, which likely  
 37 exert a strong control on the mechanical and fluid flow behaviour. Therefore, understand-  
 38 ing how rock deformation influences the hydraulic properties of rocks requires local ob-  
 39 servations that can provide a direct link between the mechanical deformation-induced  
 40 changes and the altered fluid flow responses. This necessitates 3D observations of flow  
 41 and measurements over time, since the fluid flow is a dynamic process that can only be  
 42 assessed as it occurs, and deformation features can have a complex 3D arrangement. Thus,  
 43 time-resolved 3D (i.e., 4D) measurements and observations are required.

44 X-ray imaging has become a common method to characterize the internal struc-  
 45 ture of bulk objects (e.g., Maire and Withers (2014)), including geomaterials, such as  
 46 rocks and soils (e.g., Cnudde and Boone (2013)). Such 3D imaging has been extended  
 47 to look at the evolution of localised deformation in rocks using time-lapse imaging and  
 48 Digital Volume Correlation (DVC) (e.g., Charalampidou et al. (2013)). Water imbibi-  
 49 tion in rock specimens with compaction bands has also been studied using medical x-  
 50 ray tomography (David et al., 2008; Pons et al., 2011) and pore-scale fluid flow processes  
 51 have been imaged in small rock samples using 4D x-ray micro-tomography (Berg et al.,  
 52 2014; Youssef et al., 2013; Pak et al., 2015). X-rays, however, have a strong interaction  
 53 with the rock material and a much smaller interaction with the pore fluids of interest.  
 54 Neutron imaging provides an alternative method that is much better adapted to the imag-  
 55 ing of hydrous fluids in rocks. Neutrons have a different interaction mechanism with mat-  
 56 ter to x-rays. In particular, neutrons are absorbed or scattered by the nucleus of the atoms  
 57 while x-rays interact with the electrons shells. Therefore, x-rays absorption is mainly pro-  
 58 portional to material density whilst neutrons absorption is linked to the capacity of the  
 59 nucleus to host an additional neutron. This leads to a strong interaction of neutrons with  
 60 hydrogen, whose nucleus is formed by only a proton and it is keen to receive a neutron,  
 61 and thus with the hydrogen-rich fluids of interest in rocks. In contrast, deuterium does  
 62 not have such a capture site (it is already filled), and thus the D<sub>2</sub>O form of water is sub-  
 63 stantially transparent to neutrons, much like the solid components of the rock and the  
 64 metals used in the experimental configuration. (e.g., Perfect et al. (2014); Kaestner et  
 65 al. (2016)). Therefore, much higher contrasts of fluid, compared to the rock, can be achieved  
 66 with neutron imaging than with x-ray imaging, which enables much lower saturations  
 67 to be detected. Furthermore, with a higher contrast, a sufficient signal to noise ratio can  
 68 be obtained with a shorter acquisition time leading, potentially, to faster imaging.

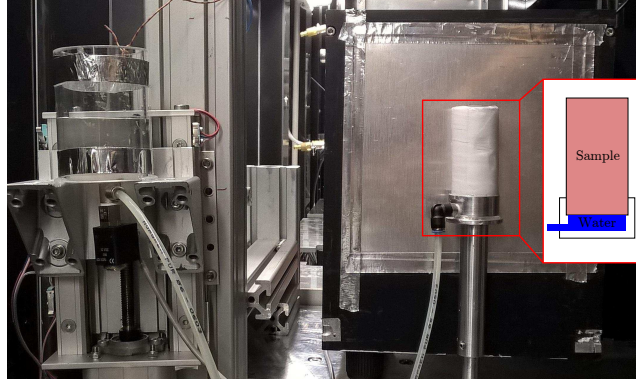
69 Previous work (e.g., Masschaele, Dierick, Cnudde, et al. (2004), Masschaele, Di-  
70 erick, Van Hoorebeke, et al. (2004), Hall (2013), Tötzke et al. (2017), Tudisco, Hall, et  
71 al. (2017)) demonstrated the potential of neutron imaging to follow internal fluid flow  
72 in geomaterials. In Masschaele, Dierick, Cnudde, et al. (2004) and Masschaele, Dierick,  
73 Van Hoorebeke, et al. (2004) an example of 3D visualisation of fluid advanced is presented.  
74 However, the dimensions of the sample and the resolution were limited and no informa-  
75 tion on acquisition time is provided. The authors express the need for a quantitative anal-  
76 ysis of the water front motion. In deformed rock samples, this analysis was restricted to  
77 2D (radiography) imaging due to limitations with the possible acquisition speed. Here,  
78 the restriction to only 2D imaging of dynamic flow processes is overcome with advances  
79 in high-speed neutron tomography, to provide full 3D observations of flow processes that  
80 operate over a finite time interval. These measurements are analysed to provide full-field  
81 analysis of fluid-front velocity distributions, which is linked to hydraulic conductivity  
82 and the boundary conditions, for; (i) an intact rock sample subjected to pressure-driven  
83 flow; (ii) a sample exhibiting localised deformation subjected to a water-imbibition pro-  
84 cess. In the second case, the fluid flow analysis is compared with 3D measures of defor-  
85 mation from DVC.

## 86 2 Experimental approach

87 The neutron tomographies employed in this work were acquired with a high-speed  
88 imaging set-up at the CONRADII beamline at Helmholtz Zentrum Berlin (HZB) (Kardjilov  
89 et al., 2011), as described in the following. High-speed neutron imaging (1 minute per  
90 tomography) is possible at CONRADII using a white beam at the experimental posi-  
91 tion closest to the neutron guide, where the neutron flux is maximum (i.e.,  $2 \times 10^8$  n/cm<sup>2</sup>),  
92 which enables the exposure time to be minimized. However, at this position, the distance,  
93  $L$ , between the pinhole aperture (diameter,  $D$ ), which defines the source, and the sam-  
94 ple is small. Since the resolution for neutron imaging is defined by the ratio  $L/D$ , this  
95 implies a reduced spatial resolution. For the experiments presented here, a pinhole di-  
96 ameter of 30 mm was used, which gives an  $L/D$  ratio of 167. In addition to the  $L/D$  ra-  
97 tio, the distance,  $l$ , between the sample and the detector also influences the image res-  
98 olution according to the equation  $d = l * D / L$ , where  $d$  is the maximum blur (e.g., Banhart  
99 (2008)). Therefore, samples should be placed as close as possible to the detector; here,  
100 due to the sample diameter, this distance was 20 mm, giving a theoretical resolution of  
101 120  $\mu\text{m}$ .

102 The neutron tomography acquisitions involved 300 radiographic projections acquired  
103 during rotation of the sample over  $180^\circ$ . Acquisitions were made with consecutive, con-  
104 tinuous positive and negative rotations over  $180^\circ$ , as full rotation over  $360^\circ$  was not pos-  
105 sible due to the possibility of collision between the detector and the water inlet tube (see  
106 Fig. 1). To minimise the exposure time for each projection the camera was used with  
107 a pixel binning of 4x4 pixels. This allowed fast exposures (0.2 s per projection) with suf-  
108 ficient signal:noise, which, in turn, permitted continuous rotation, without deadtime for  
109 stopping for each projection. With these settings, the full tomography acquisition of 300  
110 projections could be acquired in just 1 minute and with a pixel size of 100  $\mu\text{m}$  over a field  
111 of view (FoV) of about  $60 \times 73$  mm<sup>2</sup>, which is slightly smaller than the height of the sam-  
112 ples. (Note that, due to the high attenuation by the silicone used to seal the base of the  
113 sample cup, the useable FoV was slightly smaller vertically).

114 Two samples have been studied in this work to illustrate the experimental approach.  
115 These were both 38 mm diameter cylindrical samples of arkosic Vosges sandstone (22%  
116 porosity) selected from the materials used in Charalampidou et al. (2011, 2013). One  
117 sample (IGSN: IEETV003) was “as-cored” and the other (IGSN: IEETV002) had been  
118 loaded under triaxial conditions at 30 MPa confining pressure such that it contained shear  
119 bands. A notch was cut at about 2/3 of the height of the sample and covering 1/4 of the  
120 circumference to encourage the expected localised deformation to occur in the middle

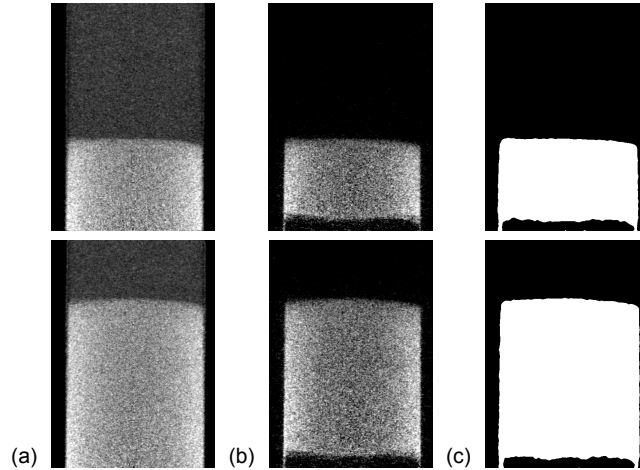


**Figure 1.** Photograph of the experimental set-up and a sketch of the inside of the cup. The water reservoir is to the left and the sample, mounted on the rotation stage, is to the right, in front of the scintillator of the detector. The electro-valve is visible below where the tube exits the reservoir. The metal post below the sample is attached to the rotation stage (out of sight, below).

121 region of the sample, similarly to Charalampidou et al. (2011)). This deformed sample  
 122 had been imaged before and after the triaxial testing using both x-ray and neutron to-  
 123 mography. DVC was performed on the x-ray images using the code TomoWarp2 (Tudisco,  
 124 Andò, et al., 2017) to provide 3D volumetric and shear strain maps throughout the sam-  
 125 ple that are used here to compare to the flow measurements. This analysis indicated that  
 126 two main shear bands had developed extending from the notch, one reaching the top of the  
 127 sample and the second extending diagonally downward for about 25 mm. Additional,  
 128 mainly smaller (shorter), bands occur in complex patterns that are associated with the  
 129 larger bands, or they may be located away from it. Although the orientations of these  
 130 smaller bands are mainly similar to the main bands, there are differences that lead to  
 131 a reluctance to describe the entire array as having variations only in two axes.

132 The intact sample had parts of the cylindrical sides removed to create two, diametrically-  
 133 opposite flat faces on which two notches had been cut on the surfaces at different heights.  
 134 This particular shape enables further advanced measurements (i.e., ultrasonic tomog-  
 135 raphy) to fully characterise the sample before and after deformation. For the neutron  
 136 experiments (to ensure a good fluid sealing and optimal imaging), specially machined  
 137 teflon inserts were placed on the flat surfaces to recover a cylindrical shape. Teflon tape  
 138 was wrapped around the sample before the inserts were positioned to prevent fluid from  
 139 being able to flow out of the sides of the sample. The sample, plus teflon inserts, was  
 140 confined in a heat-shrunk Fluorinated Ethylene Propylene (FEP) membrane to seal the  
 141 ensemble. The complete sample assembly was mounted and sealed with silicone onto a  
 142 specially-designed end-cup, which allows water to contact the entire sample base. This  
 143 cup was connected by flexible tubing to a small reservoir on a table whose height could  
 144 be remotely adjusted (see Figure 1). An electro-valve, controlled from outside the ex-  
 145 perimental area, allowed the fluid supply from the reservoir to the end-cup to be turned  
 146 on or off.

147 During the first test, on the intact sample, the level of the water was kept at a con-  
 148 stant height, above the top of the sample, by moving the reservoir upwards to compen-  
 149 sate for the fluid leaving the reservoir and entering the sample. In this way, an almost-  
 150 constant water pressure was applied at the bottom of the sample. For the initial stage  
 151 of the second test (sample with shear bands), the level of the water was kept constant  
 152 and level with the top of the sample to accelerate the advance of the fluid until the wa-



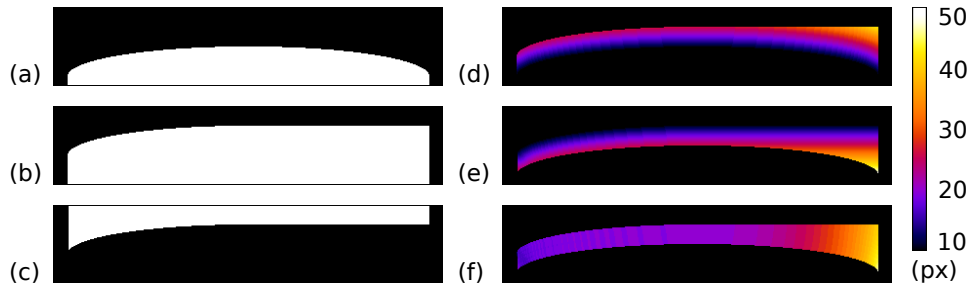
**Figure 2.** (a) Two vertical cross-section slices through the high-speed neutron tomography images of the intact sample at different times in the fluid flow process. (b) The same slices reconstructed after subtracting the initial (dry) images, as described in the text. (c) Corresponding slices through the binarisation of (b). Note that the first (dry) tomography was made when water was already in the field of view, so the region of the sample where water was already present appears as a dark area at the bottom of the image in (b) and (c).

153 ter front reached about 1/3 of the sample height. Subsequently, the reservoir was low-  
 154 ered to maintain the water level a few mm above the sample base, which allowed imbibition-  
 155 dominated flow to be studied in the area of the sample that presented localised deformation  
 156 (middle-top part). The flow experiments lasted for about 2.5 hours for the intact  
 157 sample and 5.5 hours for the deformed one, which resulted in 149 and 384 tomo-  
 158 graphsies, respectively. A technical problem occurred in the middle of the second test such  
 159 that no images were acquired for about 40 minutes, which resulted in a gap in the imag-  
 160 ing of the imbibition process.

### 161 3 Image analysis

162 The tomographic reconstructions were performed using an in-house Python code  
 163 based on the ASTRA tomography toolbox (van Aarle et al., 2015; W. J. Palenstijn et  
 164 al., 2017) assuming a parallel beam and using the 3D SIRT GPU-based algorithm (W. Palen-  
 165 stijn et al., 2011). Figure 2(a) presents two vertical slices extracted from the centre 3D  
 166 images of the intact sample at two different times. These images clearly show the ad-  
 167 vance of the fluid front and the imaged texture of the rock in the dry region above the  
 168 advancing front. In the water-filled area, the neutron attenuation is high and the tex-  
 169 tural information is obscured. Moreover, a beam hardening effect is evident in the sat-  
 170 urated region. To facilitate the thresholding of the images described above, the tomo-  
 171 graphic reconstruction was repeated using radiographic projections normalised with the  
 172 corresponding projections from the initial tomographic scan of the dry sample. This pro-  
 173 cedure allowed only the water to be reconstructed, as shown in Figure 2(b). It can be  
 174 seen that this process removes the rock sample texture from the images, which provides  
 175 further support that the sample texture visible in Figure 2(a) is real and not noise.

176 The focus of these experiments is to demonstrate the capability to achieve a quan-  
 177 tification of the advance of the fluid front, which is clearly visible in the images in Fig-  
 178 ure 2. The contrast between the fluid-filled and the dry regions of the samples is much  
 179 greater than the noise level or the effect of beam hardening, especially after removing

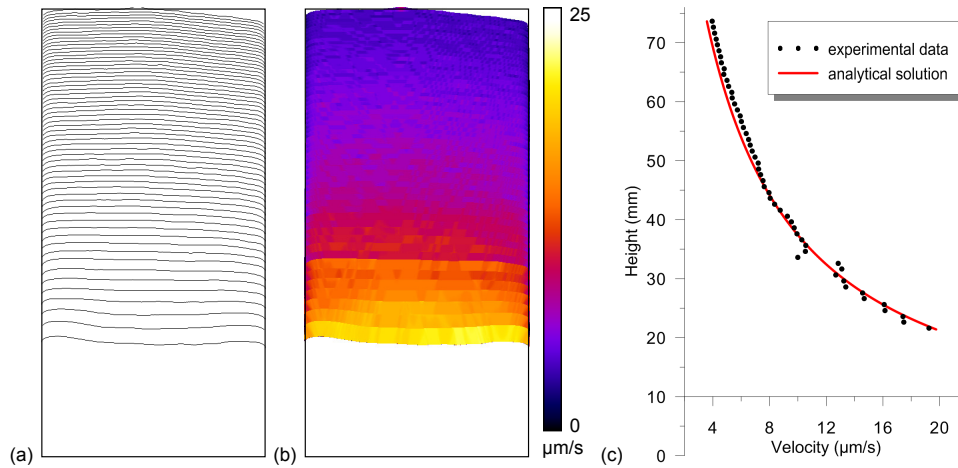


**Figure 3.** Speed field determination steps: binary image of (a) the initial front (Front 1) and (b) the manually-adjusted next front (Front 2), (c) negative of Front 2, distance map from (d) Front 1 and (e) from Front 2 masked to show only the area of interest, (f) sum of the distance maps.

180 the tomographic images of the dry sample (Figure 2(b)). By binarisation of the 3D  
 181 images it is possible to separate the dry and wet volumes to provide a series of binary 3D  
 182 images that represent a time series of the water front progression. From each consecu-  
 183 tive pair of binary image series, a 3D map of the speed of advance of the water front has  
 184 been calculated, as described in the following.

185 Figure 3 illustrates the speed map determination approach for an artificial 2D case  
 186 with a front manually moved up 20 pixels in the vertical direction and flattened on one  
 187 side so that the maximum displacement there is 48 pixels. The first step in the analy-  
 188 sis procedure involves calculation of the euclidean distance of each pixel in the image from  
 189 the nearest black pixel to give distance maps. This is performed for the first binary image  
 190 in each consecutive image pair and is repeated for the second image in the pair, but  
 191 with a negative of the binarised image to retrieve the distance in the correct direction  
 192 from the second front (i.e., downward). To reduce artefacts at the boundaries of the sam-  
 193 ple, the outside of the sample in the images was set to zero (see Figure 3(c)). For the  
 194 real data, the shape of the sample is determined from the last tomography image, when  
 195 the sample is full of water. The two distance maps are subsequently masked to retain  
 196 only the image of the sample region between the two fronts. These masked distance maps  
 197 are summed together to provide a new image that represents, for each point in the im-  
 198 age, the shortest-path distance that water crossing the point travels to reach the second  
 199 front from the first. Knowing the time elapsed between the acquisition of the two im-  
 200 ages, this distance can be directly converted to a speed of the front advance for each point  
 201 between the fronts in the pair of images. Figure 3(f) shows that the calculated distance,  
 202 in the artificial example, is constant and equal to 20 px where the front has moved uni-  
 203 formly, whereas the distance increases where the front is distorted. Curvature of the front  
 204 at the boundaries results in an inclined path being shorter than the vertical one. Whilst  
 205 this does not correctly represent the imposed constant advance of the front and indicates  
 206 caution should be applied when interpreting the boundary areas, this might be consid-  
 207 ered as a reasonable representation of a real flow where there is no reason for the wa-  
 208 ter to advance only in the vertical direction.

209 The described procedure provides the “instantaneous” flow speed field of the ad-  
 210 vance of the water-front between two tomographies. Repeating the process for each con-  
 211 secutive pair of images and assembling the results provides a full speed map volume cov-  
 212 ering the entire imaged sample. The resolution of the speed map is determined by the  
 213 frequency of the front reconstructions (each tomography) and it is limited by the noise,  
 214 which affects the smoothness of the surface. To obtain reasonable results, the movement  
 215 between two fronts has to be larger than the front roughness.



**Figure 4.** (a) A series of water fronts in the central vertical slice, (b) corresponding vertical slice of the water speed map for the intact sample, and (c) an analytical solution, as in equation (1), fitted with experimental data.

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## 4 Results

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Figure 4 shows, for the intact sample, the positions of the water-fronts (at 2 minute intervals) in a vertical central slice through the imaged volume and the corresponding slice through the 3D speed map (the former determined by binarisation of the tomography images during the water advance, and the latter calculated using the method described above). The speed map shows a decrease in the speed of the front advance with increasing height of the front. This is consistent with a decreasing pressure gradient with increased distance of the front from the constant-pressure condition at the sample base. Furthermore, the flow-speed field does not show any significant local variations, which is as expected from the assumption of a nominally intact and quasi-homogeneous sample. Moreover, closer inspection of the fluid front in the tomography images indicates there is no significant gradient in saturation at the front. Therefore, the role of capillarity effects can be neglected in the analysis: i.e., the flow is predominantly pressure driven.

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The second test differs from the one described above in two respects: it contains an array of experimentally-created deformation bands; and the flow experiment is conducted so that, in the top part of the sample, imbibition of water is the only operative driving force. Figure 5 shows horizontal and vertical slices through: (a) the neutron tomography image at the end of the flow experiment; (b) the water-front speed volume; (c-d) the maximum shear- and volumetric-strain volumes derived from DVC applied to x-ray tomographies acquired before and after triaxial deformation. Strain localisation bands, and their related flow effects, are clearly visible in all the presented images, including the smaller scale features of the localised deformation in the upper part of the sample. Figure 6 presents the 3D rendering of the water speed map and the maximum shear strain, in which low values are displayed as transparent to allow the visualisation of the deformation band. The images show the variability of the deformation along the sample in both shape and intensity. Such variability would be lost in a 2D analysis, where only an average projected value in the plane of the deformation can be measured.

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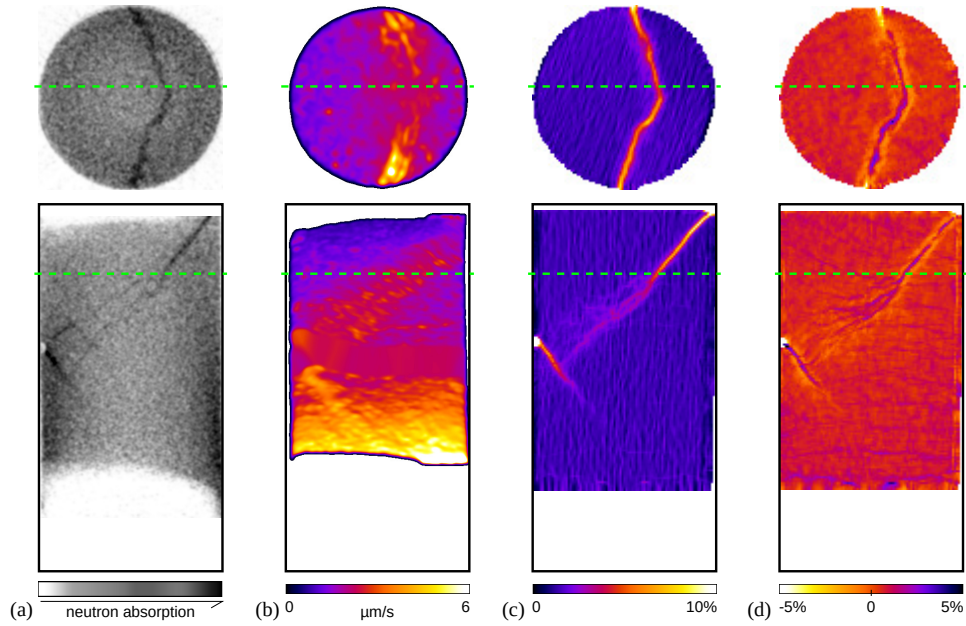
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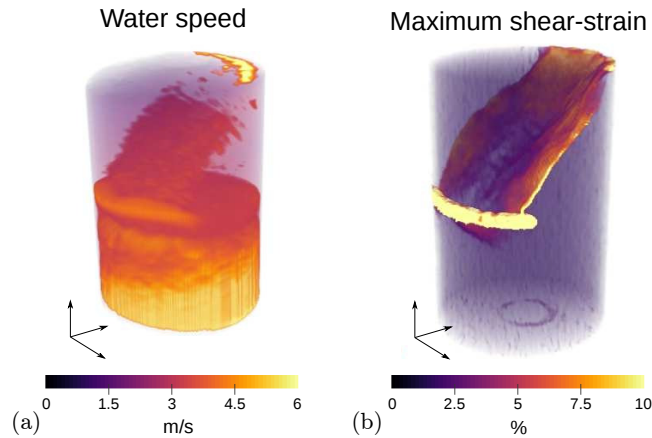
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The higher attenuation of neutrons seen in the region of the deformation bands in Figure 5(a) indicates higher water saturation, and the water-front speed map (Figure 5(b)) shows a higher value inside the bands. This experiment was conducted so that imbibition of water is the only operative driving force, therefore the higher speed in the localised deformation bands can be interpreted as being related to higher air-water cap-





**Figure 5.** horizontal and vertical slices through: (a) the neutron tomography image at the end of the flow experiment; (b) the water speed volume; (c) the maximum shear-strain volume; (d) volumetric-strain volume (positive values indicate compression). The strain fields were derived from DVC applied to x-ray tomographies acquired before and after triaxial deformation. To better highlight the variations in the water speed map, a Gaussian filter with radius 5 pixels was applied twice to enhance the signal:noise) this also generates the dark borders visible in (b)).



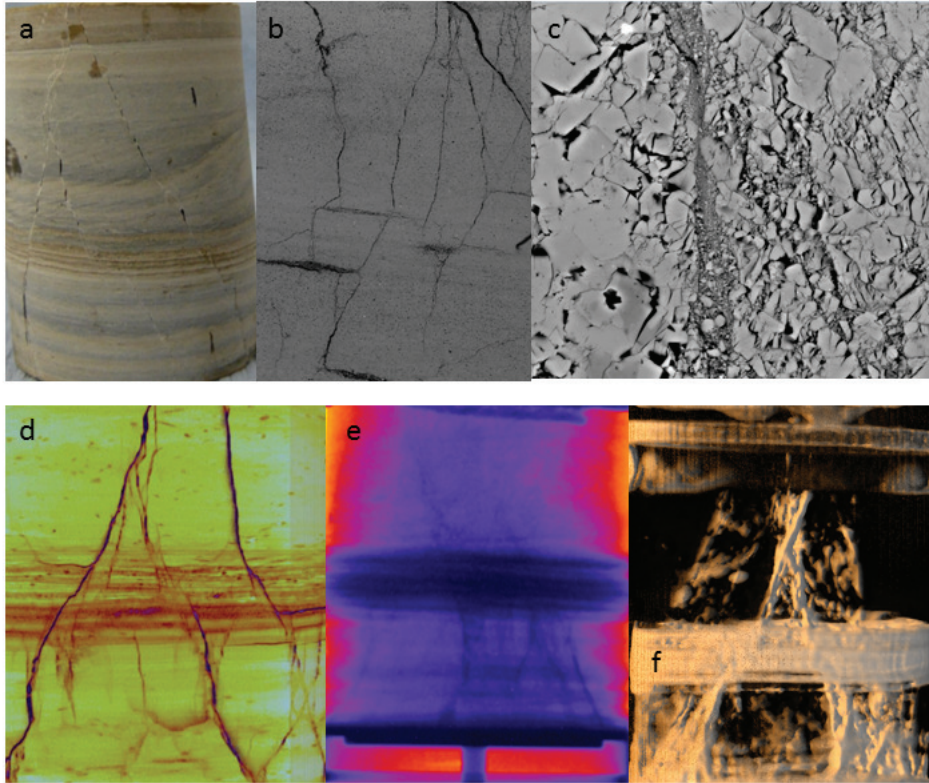
**Figure 6.** 3D rendering of (a) the water speed map and (b) the maximum shear strain. Low values are displayed as transparent to allow the visualisation of the deformation band.

248 illary pressure, which suggests smaller pore sizes in the band than in the surrounding  
 249 region. This hypothesis is consistent with the strain maps from the DVC, which reveal  
 250 that the shear bands are predominantly compactant (although some regions of dilation  
 251 can also be seen, especially close to the boundary of the sample). The shear band ex-  
 252 tending downwards from the notch shows higher water-front speeds, which suggests that  
 253 the capillary properties in this part of the band array are strongly affected by deforma-  
 254 tion, even though this localisation zone is less visible in the DVC results. In contrast,  
 255 the band continuing from the notch towards the top of the sample appears to have a lesser  
 256 impact on the water speed, compared to the surrounding region. This could be due to  
 257 the much lower velocity of the water at this height (which can only advance as quickly  
 258 as more water can arrive from below), which would make the difference between the ve-  
 259 locity in the band and in the surrounding area too small to be appreciated. Another pos-  
 260 sible explanation is the dilatant character of this part of the shear band, which might  
 261 reduce the capillary effects. In the central height region, where no information is avail-  
 262 able on flow because of the break in the image acquisition described above, it is inter-  
 263 esting to note that the method is still able to capture the average speed and the higher  
 264 speed in the bands despite the long distance between the two water-fronts.

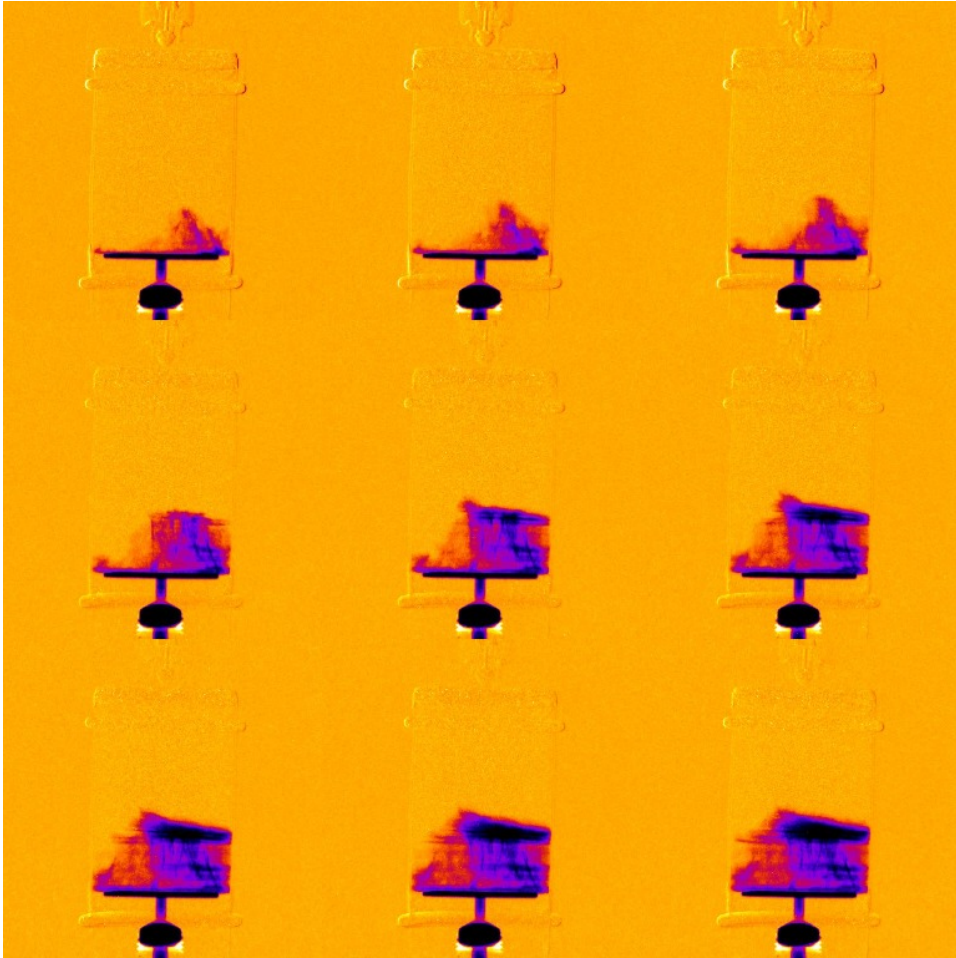
265 An additional neutron experiment is briefly described here to emphasise the rel-  
 266 evance of the three-dimensional nature of tomography. This experiment is performed on  
 267 a carbonate (limestone) rock comprised of very thin layers (called laminae, <1mm thick)  
 268 oriented orthogonal to the cylinder axis. These laminae are composed of aggregates of  
 269 micro-crystals (2-5  $\mu\text{m}$  diameter), with alternating laminae exhibiting variations in inter-  
 270 crystalline pore space such that local porosity ranges from <15% to >45% (Buckman  
 271 et al., 2018). The sample was pre-deformed (dry) to just past the peak stress at a con-  
 272 fining pressure of 20 MPa. Instead of developing through-going deformation bands like  
 273 the samples described above, it developed a complex array of shorter, planar and pre-  
 274 dominantly steep features (Figure 7(a)) that exhibit a dilational (lower density) char-  
 275 acter on x-ray tomography examination (Figure 7(d)). On subsequent inspection by SEM  
 276 (Scanning Electron Microscopy), they are seen to include a dilation-dominant mixture  
 277 but with some compactional zones (Figure 7(b),(c)). Nominally, at macro-scale, these  
 278 features look like partly-open fractures, with shear movement indications as well as open-  
 279 ings like those seen at smaller scale in the SEM images. They are typically steeply in-  
 280 clined with multiple orientations arrayed around the sample axis, and altogether, they  
 281 create a complex network of partially-intersecting discontinuities at a wide variety of ori-  
 282 entations that cannot be captured in a 2D representation (Figure 7(e),(f)). They also  
 283 exhibit a clear relationship with the laminae, with terminations typically but not exclu-  
 284 sively occurring at lamina boundaries.

285 The neutron experiment on this sample (performed at the Institut Laue-Langevin  
 286 (ILL), using the then newly-commissioned NeXT facility, which at the time could not  
 287 acquire high-speed neutron tomographic images due to constraints, since overcome, with  
 288 the control software for the rotation stage) reveals considerable complexity in the flow  
 289 patterns. The initially air-filled sample was subjected to slow water injection at its base,  
 290 using a cup analogous the one described above. The invasion of the initial fluid ( $\text{D}_2\text{O}$ )  
 291 was extremely non-uniform. The following description is based on neutron radiography  
 292 images (Figure 8), so, while the observations reveal clear patterns of flow behaviour, the  
 293 fixed viewpoint inevitably results in significant uncertainty except when the view is aligned  
 294 directly along a planar feature. In particular it is impossible to identify if a tabular zone,  
 295 or an irregular zone with finite length and width, as seen on the radiography is actually  
 296 water in porous rock or if it is a fracture oriented normal to the viewing direction.

297 Initially, the water moved up along a few of the fractures, readily identifiable in the  
 298 radiography as such (Figure 8(a)) because of their suitable orientation. These fractures  
 299 were known to intersect the base. As well as moving up the fractures the  $\text{D}_2\text{O}$  moved  
 300 horizontally creating a set of typically approximately tabular zones tabular zones. These



**Figure 7.** (a) Photo of deformed sample, showing laminated character of this rock, and the expression of deformation features as they intersect the sample exterior. Sample is 38mm diameter. (b) SEM image of a small ( $\sim 2$ mm field of view) part of the deformed sample. Image courtesy of Jim Buckman. (c) High-resolution SEM image of part of one fracture, showing the comminution of grains forming a groundmass of ultra-fine particles with nano-porosity. Note also the presence of disconnected microfractures within the band. Field of view  $\sim 20$  microns. Image courtesy of Jim Buckman. (d) Vertical slice from post-deformation x-ray tomography scan, showing that the fractures are dark (less dense). Note how some of the deformation features terminate against a depositional interface, and others transect larger regions. (e) Radiograph of the sample after significant water has entered the sample. Note how the fractures are dark, indicating higher water saturations. (f) Part of a 45-minute tomography scan obtained after  $H_2O$  replaces  $D_2O$ . Note how the  $H_2O$  is mainly located within the deformation features, but has invaded the unusual set of more porous laminations in the middle of the sample (compare also with (a) and (d)).



**Figure 8.** Sequential false colour radiographic images of the laminite sample.

301 semi-tabular zones show water ingress into laminae (Figure 8(b),(c)) but it was not possible to determine from the radiography if these zones were actually indicating D<sub>2</sub>O occupying the porespace of the undeformed rock or if it was occupying a fracture oriented at right angles to the direction of view. But the tomographic images (Figure 7(f)) show no open fractures in this position. Significant D<sub>2</sub>O saturation was normally achieved at least the right side of the sample as viewed in Figure 8 before the process moved to a progressively higher lamina and repeated. Generally filling moved from right to left, which was also away from the well-connected fracture network horizontally along the laminae, as well as from the base upwards. Some laminae were not invaded, while a few laminae were filled by cross-layer movement from an adjacent or nearby lower lamina: again this discrimination between lamina and fracture D<sub>2</sub>O ingress required the tomographic images. Some suitably oriented fractures can be seen in the radiography to start participating in the flow, and delivered D<sub>2</sub>O to a higher laminae, with the D<sub>2</sub>O then moving downwards into a dry lamina, typically along a fracture (compare Figure 8(c) and (d)): again the unsuitably oriented fractures needed the tomographic image to confirm their identity. This filling process was modified as the height of the D<sub>2</sub>O invasion increased, first encountering a zone of more porous laminae, with no suitably positioned open fractures, which filled (probably radially) lamina by lamina. The early stages of this process are seen in Figure 8(d). Above this region of more porous laminae, the sample has fewer but longer and less-well-connected fractures (Figure 7(f)). However the same pattern of water ingress along fractures and laminae was observed and confirmed by the combination of radiography and tomographic images as needed. When the invasion achieved a quasi-static state (i.e., no discernible changes in saturation), the injection fluid was switched to H<sub>2</sub>O. Due to higher neutron attenuation of H<sub>2</sub>O, the flow pattern in this semi-saturated condition was readily apparent (Figure 7(c)). During this phase of the experiment, the flow was almost exclusively concentrated within the fractures, and water exited the top of the sample very quickly.

328 Despite the high neutron flux ( $8.6 \times 10^7$  n/cm<sup>2</sup>/s), the initial setup on the NeXT facility (at the time this sample was used in the experiment) did not permit the rotation stage to operate at high-speed, so slower, higher quality tomographies (typically 45 minutes) were acquired. Fluid injection was stopped while individual tomographies were acquired, with a limited degree of diffusion taking place during the suspended flow. For the majority of the experiment, the process was only followed through radiography (with an exposure time of 0.6 s). However, at the more interesting points, the sample was remotely rotated to allow the experimental team to construct a mental picture of the 3D character of the flow being observed. For both tomography and radiography a pinhole of 20 mm was used, which gave an L/D of 500. The high-speed neutron tomographic method described here is now operational at the ILL, and achieves tomography acquisition in less than 1 minute, thanks to the uniquely high neutron flux. Subsequent experiments, using samples similar to this one, along with other carbonate rocks, have since been undertaken, and the full analysis of that suite of experiments will be reported separately. The key point, in relation to this paper, is that flow processes is intrinsically three-dimensional and can exhibit major complexity depending on the nature of the rock and its loading history. High-speed neutron tomography is in this respect an essential tool which enables new investigations of fluid flow in complex geomaterials.

## 346 5 Discussion

347 The results presented in the previous section demonstrate the ability to capture the time-dependent evolution of the fluid flow front in 3D via high-speed neutron tomography, both under pressure- and capillary-driven flow, and for a nominally homogeneous sample and a heterogeneous one that contains localised deformation features. Based on these results some clear questions might be posed, such as: (i) are the speed measure-

352 ments truly quantitative and reliable; (ii) how fast can the flow be and still be captured  
353 by this method?

354 To answer the first of the above questions, a comparison against an analytical so-  
355 lution can be made for the first test on the intact sample, as, in this case the water fronts  
356 are sub-horizontal, which allows the system to be considered as 1-dimensional. On this  
357 basis, Darcy's law defines a linear relationship  $q = -Ki$ , where  $q$  is the specific discharge,  
358  $K$  is the hydraulic conductivity,  $i$  is the hydraulic gradient, with  $i = (h_1 - h_2)/z$  where  
359  $z$  is taken as the height of the wetted area of the experiment, and  $h_1$  and  $h_2$  are the hy-  
360 draulic head values at the bottom and top of that region. The pressure (head) at the bot-  
361 tom of the sample is constant and given by the water-tank level ( $z_w$ ). Setting  $z = 0$   
362 at the bottom of the sample, the hydraulic head is  $h_1 = z_w = 160\text{mm}$ . The pressure  
363 at the water-front is zero and the hydraulic head is  $h_2 = z$ . The hydraulic gradient changes  
364 with time because the height of the wetted region increases and the specific discharge,  
365 which is equivalent to a flow speed, is given by:

$$q(z) = -K(1 - zw/z). \quad (1)$$

366 Given the flow speed determined for each height in the sample, a value for the pa-  
367 rameter  $K$  can be determined, from which the permeability of the sample,  $k$ , can be de-  
368 rived by  $k = K\mu(\rho g)$ , where  $\mu$  and  $\rho$  are the water viscosity and density, respectively.  
369 The analytical equation (1) has been fitted to the experimental points obtained by av-  
370 eraging the water speed values over square horizontal slices in the middle of the sam-  
371 ple, avoiding the edge effects, see Figure 4(c). Fitting of (1) to these data provides a best-  
372 fit value for  $K$  of  $3.3 \cdot 10^{-6}$  m/s, which corresponds to an apparent permeability of  $3.3 \cdot$   
373  $10^{-9}$  cm<sup>2</sup>, which compares well with lab-measured values of 1–2 Darcy obtained with  
374 other samples of this sandstone. The good fitting of the curve and the realistic value found  
375 for the permeability suggests that the hypothesis of pressure driven flow is correct and  
376 that the results from this imaging method can be used for quantitative analysis.

377 With respect to the second question above, accepted wisdom suggests that, in gen-  
378 eral, changes in a sample being imaged for tomographic reconstruction should not ex-  
379 ceed one pixel during the scan to allow a good reconstruction of the volume. In this case,  
380 the analysis provided reasonable results even where the water-front was moving around  
381 1 mm per minute (i.e., 10 pixels advance of the front during the time taken for the to-  
382 mography data to be acquired). The movement of the water, however, causes a distor-  
383 tion in the reconstructed volume since the front is lower in the first projection than in  
384 the last one. For this reason, measurements of the frontal advance speed are best made  
385 between even numbers of tomographies (i.e., with the same rotation direction) to have  
386 the same kind of distortion in all the fronts. This issue can be overcome, in part, by analysing  
387 the data as a time-series of projections and not as reconstructed tomographic volumes,  
388 as has, for example, been described by Jailin et al. (2018).

389 In other tests not shown in this paper, it appears that, during imbibition, the wa-  
390 ter is always faster inside the deformation bands regardless of the confining pressure ap-  
391 plied during the prior triaxial loading (which might be considered to control the char-  
392 acteristics of the resulting deformation). It is, however, crucial to carry out pressure-controlled  
393 flow tests with pre-saturated samples to study the effect of deformation on the local in-  
394 trinsic permeability. Such tests have already been performed, but with only radiographic  
395 (2D) imaging of the sample during flow by Tudisco, Hall, et al. (2017), using a D<sub>2</sub>O (heavy  
396 water) saturated sample with pressure-driven H<sub>2</sub>O to replace the D<sub>2</sub>O. As D<sub>2</sub>O and H<sub>2</sub>O  
397 have very similar physical properties, but quite different neutron interactions (D<sub>2</sub>O at-  
398 tenuates neutrons much less than H<sub>2</sub>O), this can be considered as a single phase flow sys-  
399 tem where, as demonstrated by Tudisco, Hall, et al. (2017), it is possible to distinguish  
400 an advancing front (between H<sub>2</sub>O and D<sub>2</sub>O). Moreover, the tests of Tudisco, Hall, et al.  
401 (2017) were performed in-situ (i.e., during loading), placing a triaxial cell in the beam-  
402 line, which enabled the study of the effects of evolving deformation and without unload-

403 ing or desaturation of the sample for the imaging. The results in this current work have  
404 shown that it is now possible to perform the 3D imaging sufficiently fast and to control  
405 the flow to be able to follow the flow processes in full 3D.

406 The fluid flow conditions used for these experiments, although not ideal to study  
407 intrinsic permeability, provide opportunities to make observations leading to important  
408 insights. An example is the situation depicted in Figure 6, where the water has prefer-  
409 entially moved upwards, above the general level of water and beyond the position of the  
410 notch, along the inclined shear band. The water front is still advancing along the band,  
411 while the water located within the band, but behind the front, is also continuing to move  
412 at high speed. The rock texture inside the band (based on examination of other Vosges  
413 samples deformed in the same campaign, and destructively sampled for thin sections and  
414 SEM studies) is believed to consist of a zone of broken grains, with smaller pore spaces  
415 between the fragments as compared against the texture of the intact rock. That tex-  
416 ture is expected to result in high capillary pressure values within the band. The fast move-  
417 ment of water, as observed, is consistent with the inferred zone of higher capillary pres-  
418 sure. But why does the water behind the front continue to move at a rapid rate? Cap-  
419 illary pressure is an extremely local phenomenon, and is rooted at the pore scale, where  
420 the curvature of the water/air interface (in this example) provides the fluid energy that  
421 becomes expressed as an equivalent (negative or suction) pressure. Is it sensible to in-  
422 fer that the frontal advance pulls water along behind it? The answer is no, of course, and  
423 the physics of water movement in the already partly-saturated band has to be examined  
424 for another explanation.

425 It is possible to erect a working hypothesis that remains focused on capillary forces.  
426 The quasi-planar (but finite-thickness) band may well have a distribution of textures across  
427 its area. Some local spots could have smaller pore sizes, and thus higher effective cap-  
428 illary pressures. These would locally imbibe any water available from nearby regions, seek-  
429 ing to achieve a higher water saturation that would reduce the local energy differentials.  
430 So, there are plausible mechanisms for flow within the band. But the real question is not  
431 answered, because if local regions imbibe water from adjacent local regions, those source  
432 regions experience de-saturation, which introduces new energy differentials that counter  
433 the imbibition flow. Thus, the observed continuity of water in the partly-saturated band,  
434 and the persistence of rapid flow within it, implies that the band becomes a favored flow  
435 route. This reasoning leads to the hypothesis that the band also possesses a higher-permeability,  
436 as well as its higher capillary pressure. Higher permeability is usually thought to anti-  
437 correlate with higher capillary pressure, but the detailed flow observations provide a ba-  
438 sis for supposing that the two characteristics exhibit a positive correlation. The same  
439 implications arise from consideration of the flow behaviour of the complexly fractured  
440 sample depicted in Figure 7, which will be treated in a subsequent paper focused on that  
441 sample suite.

442 The value of time-resolved 3D images of fluid flow, in rock samples with internal  
443 heterogeneities, is clear. The images provide evidence of flow processes that reveal com-  
444 plexity in the way that invading fluids move into the rocks, and the way that fluid moves  
445 in a saturated sample. In the case of the limestone sample, the experiment involved in-  
446 troduction of a contrasting fluid ( $\text{H}_2\text{O}$  replacing  $\text{D}_2\text{O}$ ), and the resulting flow pattern  
447 shows that the later fluid movement is focused in the planar deformation features that  
448 were previously thought to be simple opening-mode fractures. Current work is exam-  
449 ining the local textures of these planar features, via destructive sampling that has en-  
450 abled thin sections to be made and studied (e.g., as in Figure 7 (d)). Digital-rock meth-  
451 ods are being applied to the textural images to derive local flow properties (Jiang et al.,  
452 2017, 2018). These will be assembled into whole-sample models that should provide a  
453 basis for developing a full process explanation of the fluid flow processes that can be ob-  
454 served via high-speed neutron tomography.

## 6 Conclusions

This paper has presented a new method, involving high-speed neutron imaging and image analysis, for 3D monitoring of fluid advance in rock specimens, with unprecedented spatio-temporal resolution. The new analysis method allows the determination of fluid-front advance speed at each voxel position in the 3D volume.

Quantitative full-field determinations of fluid-front speeds have been made in two samples of a sandstone, a nominally-intact specimen and one containing localised deformation features resulting from laboratory triaxial loading, for flow under pressure- and capillary-driven conditions, respectively. Verification of quantitative speed measurements has been performed by comparison to a 1D analytical model of pressure-driven flow for the intact sample, for which an hypothesis of homogeneity is reasonable. This verification provides confidence in the local measurements of capillary-driven fluid-front advance in the sample containing localised deformation features. The resultant fluid-front speed map for the deformed sample has been compared to full-field strain measurements from DVC analysis of x-ray tomography images acquired before and after the laboratory triaxial loading. This comparison reveals that the localisation of fluid-front speed correlates well with localisation of deformation and that the fluid-front advances faster in the more deformed regions of the samples (i.e., in the shear-bands). The apparent 'pull' of water in these bands likely relates to increased capillary drive relating to reduced pore-sizes, which is consistent with the general compactant nature of the localised shear features.

Based on the methods presented herein, it is now possible to perform 3D imaging sufficiently fast and to control the flow to be able to follow flow processes in full 3D and in-situ in a neutron imaging station. The next step is to combine this with in-situ triaxial loading and high spatial resolution time-lapse tomography to enable DVC analysis, which, combined with the 3D flow quantification of this paper, will enable studies of the evolution of both the deformation and the fluid flow, thus enabling research in hydro-mechanical coupling.

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Data supporting the conclusions can be obtained at [https://figshare.com/projects/Mechanical\\_deformation\\_and\\_fluid\\_flow\\_analysis\\_in\\_geomaterial/37097](https://figshare.com/projects/Mechanical_deformation_and_fluid_flow_analysis_in_geomaterial/37097)

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