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Studies on resaturation processes and structural characteristics of the Callovo-Oxfordian claystone using X-ray microtomography and digital volume correlation

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Abstract

Claystone formations in their natural state are favorable for geological disposal of radioactive waste. Nevertheless, disturbances and water loss generated around the excavated areas might change the favorable properties of formations. Similarly, water loss and resaturation during sample preparation can also cause variations in the mechanical properties. To investigate the influences produced by these actions, resaturation processes in a claystone were studied using X-ray microtomography and Digital Volume Correlation (DVC). Structural characteristics of Callovo-Oxfordian (COx) claystone inside the sample during resaturation processes are visualized in the form of images. Strain fields demonstrate the non-uniform deformation behavior of the sample during the resaturation processes, as well as details of local deformations. The results indicate that the effects of resaturation processes on the claystone cannot be ignored for the laboratory testing at the sample scale.

Keywords: X-ray microtomography; Digital volume correlation; Claystone; Structural characteristics; Strain field

1. Introduction

Claystone formations, due to their extremely low permeability and good self-healing capacity, are considered by many European countries as geological disposal of radioactive waste [1]. Permanent safety is the primary objective for geological repositories of radioactive waste. One of the major issues associated with the safety assessment is excavation-induced disturbance. And the drying and wetting cycles may cause more effects on the excavation disturbance zone. Conventional tests are mainly performed by measuring

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26 the permeability of samples to determine the degree of damage and self-sealing [2]. In addition, the pro-
27 cesses in the preparation of laboratory samples will unavoidably lead to water loss in saturated claystones.
28 For these cases, it is unable to visualize the variations in samples only with standard macroscopic measure-
29 ments and more difficult to investigate the mechanistic correlation. In recent years, industrial micro X-ray
30 computed tomography has been widely used in various fields for the internal characterization of materi-
31 als [3, 4]. To better understand the variations of the COx claystone during resaturation processes, X-ray
32 microtomography and digital volume correlation were applied to this study. Structural characteristics and
33 non-uniform strain fields of the claystone sample during the resaturation processes are visually demon-
34 strated by image processing and visualization softwares. In particular, the local deformation behaviour in
35 the sample is investigated (e.g. development and closure of tensile cracks).

36 **2. Material and methods**

37 *2.1. Material characterization*

38 The tested sample was produced from a core (Ref. EST57926) of COx claystone at Bure, France. Available
39 mineralogical studies have shown that the average compositions of minerals in the formation are: 50-55%
40 clay minerals, 20-25% carbonates, 20% tectosilicates and 3% heavy metal minerals. The components of
41 the clay minerals are relatively constant at 55% illite-smectite interstratified minerals, 30% illite and 15%
42 kaolinite and chlorite. Its average porosity varies between 14% to 21% and natural water content ranges
43 from 5% to 8% [5].

44 *2.2. Sample processing*

45 A sample of about 4.0 mm in diameter and 7.5 mm in length was produced by a complex multi-step process
46 [6], and its central axis is perpendicular to the bedding planes of the formation. The initial water content of
47 the core from the formation is about 6.9%. The complicated processes of sample preparation resulted in a
48 final sample with a water content around 4.0%.

49 *2.3. Experimental setup*

50 In order to meet the requirements of the test, a new device suitable for the X-ray microtomography platform
51 was designed and made, the sketch is shown in Fig.1. The cell is made of Polyetheretherketone and the

52 attenuation coefficient of this carbon-based material is small (low atomic number and low density compared
53 to the constituents of argillite). The thickness of the cell is 7 mm. This device can withstand maximum
54 radial and axial compressive stresses of 12 MPa and 60 MPa, respectively, for a sample diameter up to 5
55 mm. Images were acquired at the ISIS4D X-Ray platform.

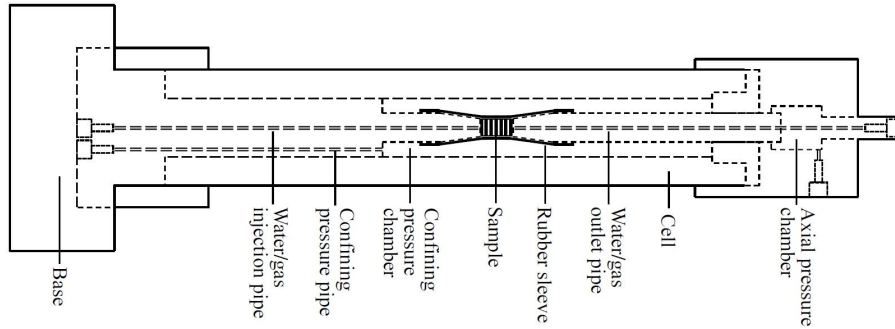


Fig. 1. Sketch of experimental device for realisation of water resaturation tests

56 2.4. Methods

57 A sample with a saturation of about 50% was set up in the device. Then an axial stress of 14 MPa and
58 a radial stress of 12 MPa were applied simultaneously. Axial and radial stress values are set close to the
59 in-situ stress state of the CO_x formation. A water injection pressure of 2 MPa was applied to the sample
60 and it was maintained for about 2 months. Seven scans were performed during the test with a voxel size of
61 6 μm . The parameters of acquisition are always kept identical between acquisitions (current, voltage and
62 filters). This would limit the variation in grey levels between acquisitions. To ensure the relative positions
63 between acquisitions, images were registered through Avizo software. Here DVC calculations were carried
64 out using the YADICs platform developed in Mechanics Laboratory of Lille [7]. In this study, the DVC
65 calculation element is an $8 \times 8 \times 8$ pixel matrix. More details including uncertainty can be found in the
66 reference [6].

67 **3. Results and discussions**

68 *3.1. Analysis of grayscale images*

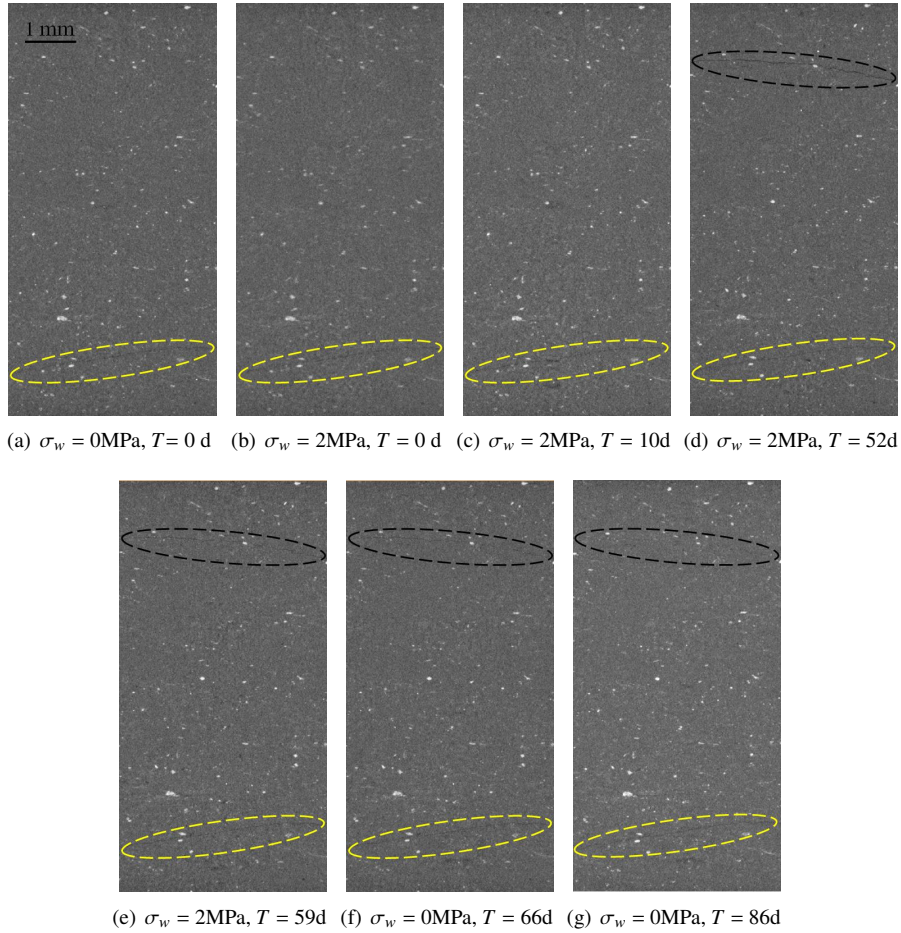


Fig. 2. Tomographic reconstructions of the longitudinal slices on the tested sample during the resaturation test (σ_w is water pressure, T is the accumulated days, the sample is under 14 MPa axial stress and 12 MPa radial stress constantly)

69 Fig.2 presents a set of tomographic reconstruction images of longitudinal slices of the sample during the
70 resaturation test. An initial quasi-horizontal crack, marked by the yellow dashed line, can be clearly seen
71 in the sample near the bottom. This crack did not vary obviously during the whole resaturation process.
72 It indicates that this crack is most likely generated by the processing of the sample and not by shrinkage
73 of water loss. In Fig.2(d), a nascent quasi-horizontal crack (marked by the black dashed line) was found
74 in the grayscale image at the top part of the sample. This is most probably contributed to the non-uniform
75 swelling of the clay minerals. Afterwards, it can be seen that the nascent crack gradually closed until it was
76 almost invisible. This phenomenon can also be found in some recent scientific papers [8].

77 3.2. Analysis of strain fields

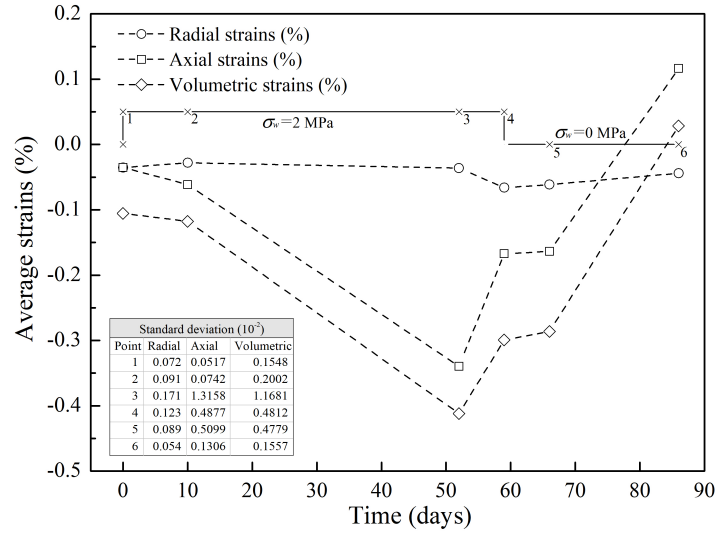


Fig. 3. Evolutions of radial, axial and average volumetric strains in the sample with time during the resaturation test

78 Taking the initial state in which the sample was produced as a reference, six DVC calculations were per-
 79 formed. The obtained global-average strains from the DVC are presented in Fig.3, including the standard
 80 deviation values of each computed point. Positive values for compressive and negative values for tensile
 81 are defined here. The variation of radial strains is relatively smooth, exhibiting a slight dilatation. The evo-
 82 lution of axial strains is consistent with the opening and closure of the nascent crack. The volumetric strain
 83 values are mainly dominated by the axial strains from a global perspective. This should be attributed to the
 84 orientation of the nascent crack opening in the vertical orientation. According to previous investigations
 85 [6], deformation is more susceptible to be achieved in bedding planes. Values of standard deviation also
 86 well reflect the occurrence of large local strains (e.g. tensile cracks) in the sample.

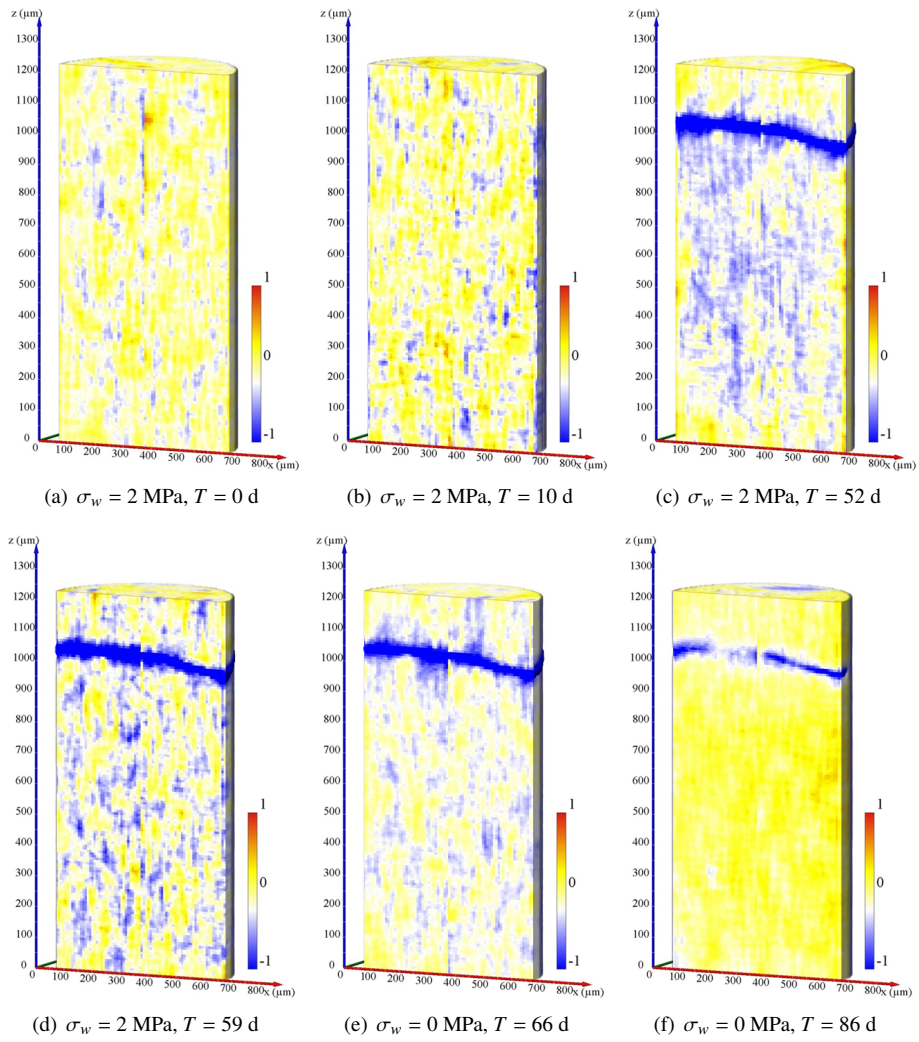


Fig. 4. Distributions of accumulated volumetric strain fields (%) in the sample at different time instances and under an axial stress of 14 MPa and a radial stress of 12 MPa

87 To better visualize the variations in the sample during the resaturation processes, the vertical cross-sectional
88 profiles of the accumulated volumetric strain fields are displayed in Fig.4. Red is defined as compression
89 and blue is defined as dilation. In the early phase, one can find numerous localized concentration strain
90 zones distributed inside the sample. This may be the results of air in the pores of the sample being squeezed
91 by the applied pressures before water arrives. After water entering the sample and interacting with the clay
92 minerals, the sample shows wide localized swelling zones. A quasi-horizontal local tensile zone is observed,
93 which is overlapped with the position of the nascent crack in the gray-scale images. The attenuation of local
94 concentration strains with time can be found. Finally, the strain field of the sample tends to be uniform. At
95 this point, the influence of the produced crack (yellow marked in Fig.2) on volumetric strain fields can be

96 considered negligible. However, the influence of the nascent crack on volumetric strain fields is remarkable.

97 **4. Conclusions**

98 In this study, X-ray microtomography and DVC were applied to the water resaturation test of CO_x claystone
99 for the first time. This approach surpasses the traditional measurement methods, and resaturation processes
100 of the claystone were monitored at a mesoscale. Swelling of unsaturated CO_x claystone is non-uniform
101 during resaturation processes. It is noted that immersion of the sample is done in one direction that could
102 amplify or induce the cracking appearance due to the high suction gradient. The tensile crack closes gradu-
103 ally with time under the actions of applied stress and self-sealing process, and the strain field of the sample
104 tends to be homogeneous. In addition, large local strains can be reflected by the values of standard deviation
105 from the computed points.

106 **CRedit authorship contribution statement**

107 **Hai-Ling Shi:** Data curation, Investigation, Methodology, Visualization, Writing - original draft. **Thomas**
108 **Rougélot:** Supervision, Writing - review & editing. **Shou-Yi Xie:** Investigation, Methodology. **Jian-Fu**
109 **Shao, Minh-Ngoc Vu and Jean Talandier:** Resources, Writing - review & editing.

110 **Declaration of Competing Interest**

111 The authors declare that they have no known competing financial interests or personal relationships that
112 could have appeared to influence the work reported in this paper.

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