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Full Length Article

Determination of elastic modulus of claystone: Nano-/micro-indentation and meso-compression tests used to investigate impact of alkaline fluid propagation over 18 years

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ABSTRACT

Micro-mechanical properties of a claystone were tested after undergoing alkaline perturbation on site (Tournemire, CD borehole) for 18 years. In a saturated context and outside the excavation disturbed zone (EDZ), the claystone exhibits a 11.6-mm black rim at the cement/paste interface, which shows a different mechanical behaviour from the rest of the claystone. Three sets of measurements of elastic modulus were performed using: (i) nano-indentation tests with a constant indentation depth of 2 μm, (ii) micro-indentation tests with a constant indentation depth of 20 μm, and (iii) meso-compression tests with a constant displacement of 200 μm. The increase of the modulus of deformability in the black rim is between 15 GPa and 20 GPa according to the scale. Moreover, an overall decrease of the modulus of deformability from the smallest to the largest scale is observed in each zone. In view of the mineralogy and petrographic observations, higher values of modulus of deformability in the black rim are related to carbonate content and its distribution. Precipitation of cementitious carbonates as inclusions and very thin partings leads to hardening of the claystone.

1. Introduction

Clayey formations are considered as potential host rocks for deep geological storage of high-level radioactive wastes. For a better understanding of the long-term behaviour of clayey host rock, it is important to understand the interactions between mechanical behaviour, micro-fabric, and mineralogy (Bock et al., 2010). The time-evolution of clayey rocks in contact with cementitious materials is also an important aspect for assessing the safety performance of geological disposal. In France, the Institute for Radioprotection and Nuclear Safety (IRSN) has developed a research programme at the Tournemire Experimental Platform (TEP, France). This TEP is based on a tunnel built between 1882 and 1886, in which different galleries have been excavated since 2003. In 2009, claystone/CEM II cement paste contacts were sampled after being in place for 18 years (CD borehole, Fig. 1) in a saturated context outside the excavation disturbed zone (EDZ).

Studies of the mineralogy were carried out on both the claystone and the cement paste in contact using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM), while analyses were performed to determine the specific surface area and geochemistry (total organic content, isotopic compositions of carbon (δ13C), oxygen (δ18O), Sr content and isotope compositions (87Sr/86Sr)) (Bartier et al., 2013). Macroscopic observations of the CD overcore samples reveal the presence of a black rim at the claystone/cement paste interface (Fig. 2a), similar to previous observations for the DM-engineered analogue located in the excavation damaged zone (Tinseau et al., 2006; Gaboreau et al., 2011; Techer et al., 2012).

The thickness of this black rim varies slightly according to the sampling depth, from 10 mm to 14 mm. The black rim corresponds to a domain with higher macroscopic surface resistance (Fig. 2b), but this difference disappears after cutting of the sample, leaving only a zone that is darker than the grey claystone. Post-mortem characterisation of CD claystone with modelling of reactive transport by the HYTEC code (Van der Lee et al., 2003) has been used to outline the nature and scale of disturbances during claystone/cement paste interactions (Bartier et al., 2013). The present study, integrated in the "NEEDS-MIPOR" Research Group project

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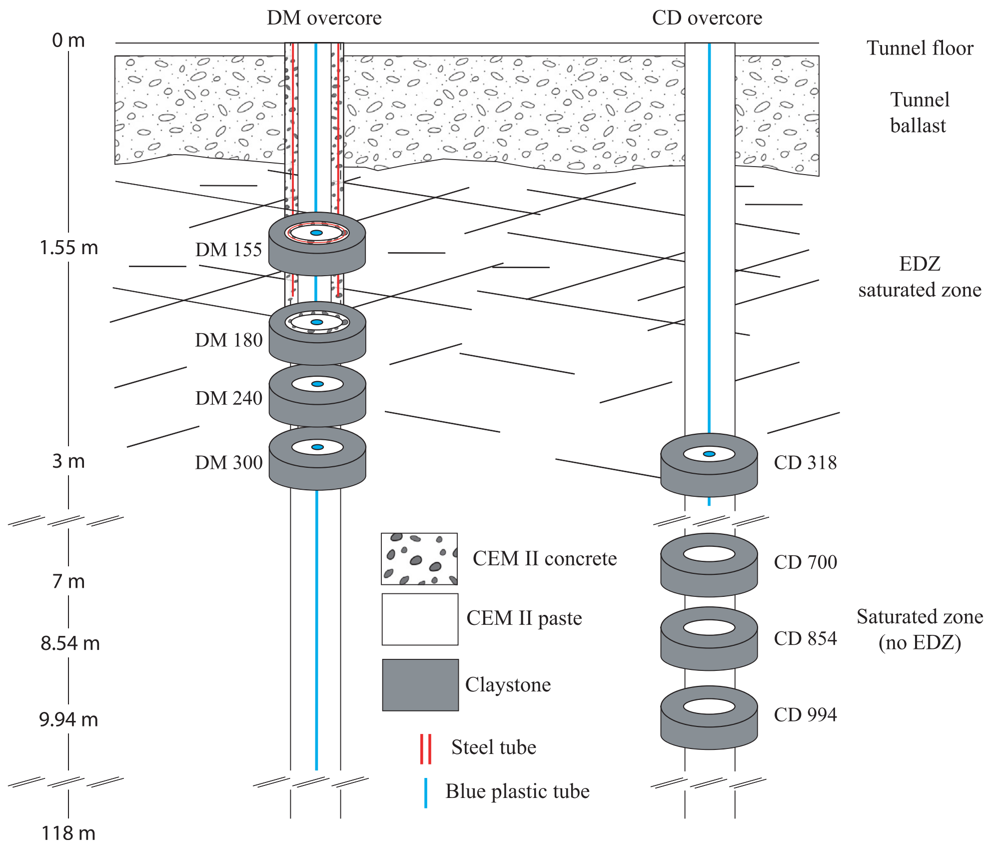

Fig. 1. Schematic diagram of the CD borehole configuration in the Tournemire Experimental Platform and comparison with the DM borehole context (according to Techer et al., 2012). Location of the studied sample: CD 854 outside the EDZ (modified after Bartier et al., 2013).

(Nucléaire, Energie, Environnement, Déchets et Société, projet fédérateur MIlieux POReux), aims to describe the instantaneous mechanical behaviour of a claystone/cement contact at different sample scales, after interaction for 18 years. Due to the strong heterogeneity related to the multiphase microstructure of claystone, an understanding of its mechanical behaviour is of major significance for the storage of nuclear waste. The present study follows on Auvray et al. (2017)'s original measurements of the modulus of deformability determined by: (i) nano-indentation tests, (ii) micro-indentation tests, and (iii) meso-compression tests on Tournemire claystone. The same types of test are applied here on different sample scales to illustrate the impact of a hyperalkaline plume on the mechanical behaviour of claystone.

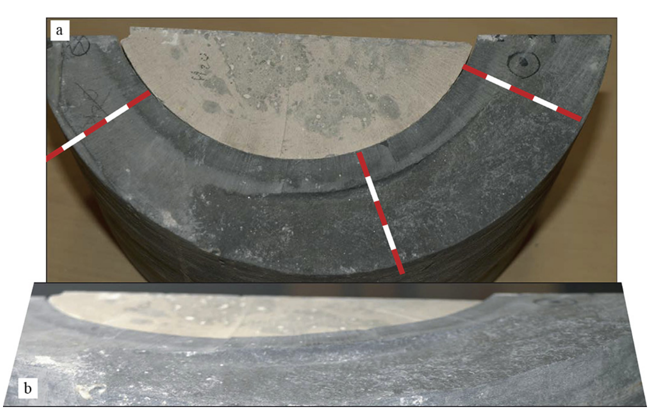

Fig. 2. (a) Core CD 854 sample section showing contact between cement paste inside and the Toarcian claystone outside. A rim of claystone (black rim, in the thickness of 10–14 mm) is observed systematically at the interface between the two materials. (b) Perpendicular view showing that the black rim has a different resistance from the rest of the claystone.

2. Mechanical tests

The technical specifications of the nano-/micro-indentation tester and meso-compression cell are detailed in Auvray et al. (2017).

2.1. Nano-indentation press

The experimental system was developed by the GeoRessources Laboratory (Nancy, France) and has been described in Auvray et al. (2013, 2015). The procedure consists of pressing a Berkovich diamond indenter into a perfectly flat and smooth surface of the sample by applying an increasing normal load. The load is applied by an electromagnet attached to a vertical rod. The rod displacement is measured by a capacitive detector and is supported by two guide springs (Randall et al., 1997). Measurements are performed several times at some points on the sample surface with a constant interval along both the *x*- and *y*-axes.

2.2. Micro-indentation press

The experimental procedure used in this study was presented in detail in Magnenet et al. (2011a, b) and Grgic et al. (2013). The