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Experimental study of elastic properties of different constituents of partially saturated argillite using nano-indentation tests

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ABSTRACT

Callovo-Oxfordian argillite, obtained from the ANDRA underground research laboratory in Meuse/Haute-Marne (France), is characterized as a multiphase material. The argillite is composed of carbonate inclusions (10–50 μm) embedded in an argillaceous matrix (representing expanding clay minerals, such as smectites). The matrix itself is also multiphase, composed of clay aggregates of 1 μm-size carbonate and quartz inclusions. The high sensitivity of the mechanical behaviour of argillite to saturation is an important characteristic of this material as it has been previously demonstrated in macro-scale mechanical experiments performed under varying degrees of humidity. The study presented here consists of grids of nano-indentation tests performed under controlled saturation conditions. The influence of the load hold time before unloading has been studied. Series of indentations were performed without or with load hold, which led to consider instantaneous or deferred unloading moduli respectively. The experimental procedure employed allows the micro-mechanical properties of the different phases (matrix and inclusions) to be determined under controlled hydration and therefore under partially saturated conditions. Several series of measurements were performed at relative humidity levels of 50%, 85%, 90% and 95%, and at a constant temperature of 20 °C. A statistical analysis enabled to discriminate the deferred unloading modulus of the different phases. At 50% humidity, we measured a mean deferred unloading modulus of 16 GPa for the clay matrix (a mean instantaneous unloading modulus of 13 GPa was observed for the clay matrix and moduli higher than 70 GPa were measured for the carbonate macro-inclusions). The mean deferred unloading modulus of the matrix appears to decrease with increasing saturation; at 95% humidity (near-saturation) it is less than 5 GPa. However, it was impossible to verify the instantaneous unloading modulus of the carbonate macro-inclusions at this high-level of saturation.

1. Introduction

Callovo-Oxfordian argillites obtained from the ANDRA underground research laboratory in Meuse/Haute-Marne (France) are a multiphase material. The rock is composed of an argillaceous matrix of expanding clays such as smectites, which contains inclusions of carbonate of several tens of microns in size. The matrix itself is also multiphase, composed of sheeted clays containing 1-μm size quartz and carbonate inclusions (Robinet, 2008; Robinet et al., 2012). Preliminary results of micro-mechanical tests obtained during millimetre-scale indentation experiments on argillite (Magnenet et al., 2011a) highlighted the importance of being able to discriminate between these phases using a grid indentation technique (Constantinides et al., 2006). The high saturation sensitivity of argillite, an important characteristic that mainly results from the presence of expanding clays, has been demonstrated through mechanical tests on macroscopic samples under varying degrees of humidity (Escoffier, 2002; Hoxha et al., 2007; Hu et al., 2014).

In order to better apprehend the mechanical behaviour of the different constituents, we have designed a study in which several series of nano-indentation tests were conducted on partially saturated samples. The area of the contact surface in our experiments is on the order of a few square microns, allowing the different phases to be discriminated at a much finer scale than it has previously been possible. The experiments were performed using a nano-indentation system that is placed within a climatic chamber. This procedure therefore allows nano-indentation tests to be performed under variable saturation levels (Auvray et al., 2013). In the present study, series of measurements were performed at a constant temperature of 20 °C at 50%, 85%, 90% and 95% humidity.

The data obtained in this nanometric study can be compared with the elastic properties of different argillite constituents estimated at millimetre-scale in an earlier study (Magnenet et al., 2011a) to establish reference values for the elastic properties of the different constituents. In the future, they could be integrated into multiscale behaviour models

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and lead to important improvements in our understanding of the mechanical and micro-mechanical behaviour of rocks.

2. Experimental procedures

The experimental procedure that was used in this study was developed by the GeoRessources Laboratory (Nancy, France) and is presented in Auvray et al., 2013. The nano-indentation test-bench is installed in a climatic chamber (Vötsch Industrietechnik GmbH) and consists of two modules, one containing the nano-indenter (CSM-Instruments) and the other containing an optical microscope that allows the surface of the sample to be viewed (Fig. 1). The sample is moved between the two modules by means of a motorised stage, which allows indentation tests to be undertaken in grids. The nano-indentation test grid is defined by programming the offset between two measurements, as well as the number of columns and rows. All tests are conducted within a millimetre-scale area. The technical specifications of the nano-indentation tester are provided in Table 1.

The surfaces of the sample must be perfectly flat and smooth as well as parallel to the support stage axes the distance between the support stage plane and the indented surface must not vary more than 5 μm. Though these requirements are systematic when preparing samples for indentation tests, they are especially important for performing nano-indentation experiments (Vandamme, 2008; Miller et al., 2008). To achieve this high degree of surface smoothness, the samples underwent five stages of lapping/abrasion during which progressively finer abrasives were applied: F80, F220, F360, F800 and F1200 (corresponding to mean grit-sizes of 185 μm, 58 μm, 23 μm, 6.5 μm and 3.0 μm, respectively).

The indentation procedure consisted of pressing an indenter into the surface of a sample by applying an increasing normal load. Once the indenter reached a defined depth h_{L1}, a partial unloading was applied (down to h_{U1}) and followed by a second loading up to the maximum depth h_{L2}. Then the indenter was removed with a controlled rate until total unloading. This procedure was performed in a repetitive manner at different points on the sample surface at a constant interval along both the x- and y-axes. The load was directly applied by an electromagnet assembly to a vertical rod, the end of which houses a standard Berkovich diamond indenter (Fig. 2). Displacement of the rod was measured by a capacitive detector and the rod was supported by two guide springs (Randall et al., 1997). The system has load and displacement resolutions of 0.04 μN and <0.04 nm respectively.

Table 1
Indentation tester specifications.

Load range	0.1–500 mN
Load resolution	0.04 μN
Maximum depth	200 μm
Depth resolution	0.04 nm
Maximum load rate	10 N/min
XY stage	120 × 20 mm
XY resolution	0.25 μm

In order to determine the elastic modulus of the different constituents we used the model of Oliver and Pharr (1992) calculated by CSM-Instruments Indentation software. This model allows Young's modulus (E_{it}) of the indented zone to be derived from load–displacement curves (Fig. 3) using the expression of the reduced modulus E_r:

$$E_r = \frac{S\sqrt{\pi}}{2\beta\sqrt{A_p(h_c)}} \quad (1)$$

where S is the elastic unloading stiffness, defined as the tangent of the unloading curve, and β is a correction factor related to the geometry of the indenter. $A_p(h_c)$ is the projected contact area of the indentation as a function of contact depth, h_c, which is obtained from:

$$h_c = h_{max} - \varepsilon\frac{F_{max}}{S} \quad (2)$$

where F_{max} is the load value before unloading and ε is a coefficient that depends on the indenter geometry.

The value of Young's modulus of the indented material is obtained from expression (3) where E_i and ν_i are the elastic modulus and Poisson's ratio of the indenter, and ν_{it} is the Poisson's ratio of the indentation zone (set at 0.30 for the whole sample).

$$\frac{1}{E_r} = \frac{1-\nu_{it}^2}{E_{it}} + \frac{1-\nu_i^2}{E_i} \quad (3)$$

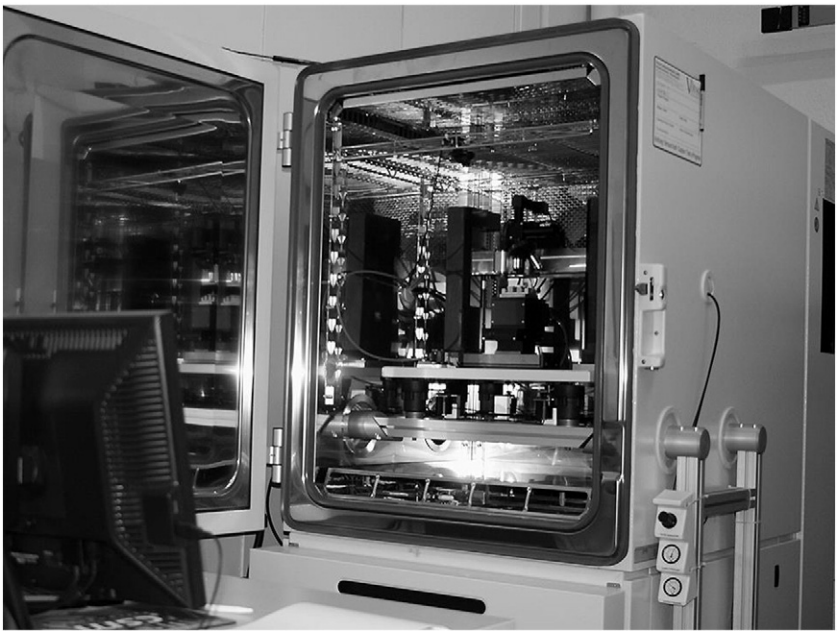

Fig. 1. Nano-indentation tester installed in climatic chamber.