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Full Length Article

Elastic modulus of claystone evaluated by nano-/micro-indentation tests and meso-compression tests

Christophe Auvray*, Noémie Lafrance, Danièle Bartier

Université de Lorraine, CNRS, CREGU, Lab. GeoRessources UMR 7359, Vandœuvre-lès-Nancy, F-54518, France

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ABSTRACT

Toarcian claystone such as that of the Callovo-Oxfordian is a qualified multiphase material. The claystone samples tested in this study are composed of four main mineral phases: silicates (clay minerals, quartz, feldspars, micas) (≈86%), sulphides (pyrite) (≈3%), carbonates (calcite, dolomite) (≈10%) and organic kerogen (≈1%). Three sets of measurements of the modulus of deformability were compared as determined in (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. These three experimental methods have already been validated in earlier studies. The main objective of this study is to demonstrate the influence of the scaling effect on the modulus of deformability of the material. Different frequency distributions of the modulus of deformability were obtained at the different sample scales: (i) in nano-indentation tests, the distribution was spread between 15 GPa and 90 GPa and contained one peak at 34 GPa and another at 51 GPa; (ii) in the micro-indentation tests, the distribution was spread between 25 GPa and 60 GPa and displayed peaks at 26 GPa and 37 GPa; and (iii) in the meso-compression tests, a narrow frequency distribution was obtained, ranging from 25 GPa to 50 GPa and with a maximum at around 35 GPa.

1. Introduction

Understanding the mechanical behaviour of claystone is of critical importance for nuclear waste storage. Previous studies of this material have mostly focused on its mechanical properties at the macroscopic scale. The tests were generally performed on 20 mm or 38 mm diameter cylindrical samples with a length to width ratio of 2, given that the sample lengths are 40 mm and 76 mm, respectively (Chiarelli et al., 2003; Shao et al., 2006; Hoxha et al., 2007; Jia et al., 2010). Micro-macromechanical approaches linked to macroscopic tests have also been attempted on this material, most notably in the study of Shen et al. (2012).

In order to better understand the instantaneous mechanical behaviour of the claystone at different sample scales, we conducted three series of measurements for this study: (i) nano-indentation tests, in which the volume of material tested each time was around 0.001 mm^3; (ii) micro-indentation tests, in which around 1 mm^3 of material was tested; and (iii) meso-compression tests, performed on around 250 mm^3 of material. The representative elementary volume (REV) of the material is in the order of 0.001 mm^3 (Robinet, 2008; Robinet et al., 2012). Consequently, a single nano-indentation measurement takes into account only one REV, and in contrast, a meso-compression test requires a sample with a volume of 1×10^6 REV.

The experimental techniques adopted for the three types of tests have already been validated on other materials: (i) Callovo-Oxfordian claystone from the ANDRA Underground Research Laboratory (URL) at Meuse/Haute-Marnes (France), on which both nano- (Magnenet et al., 2011a,b; Auvray et al., 2013, 2015; Arnold et al., 2015) and micro-indentation (Magnenet et al., 2009) tests were performed; and (ii) iron minerals from underground mines in Moselle (France), and (iii) limestone quarries in Lavoux (France), on which micro- and meso-compression tests were performed (Grgic et al., 2013).

In the present study, the values of the moduli of deformability measured in the tests performed at the three different scales were compared in order to quantify the modulus-volum relationship.

2. Multi-scale mechanical tests

2.1. Typical characteristics of the rock materials

The claystone samples studied are a qualified multiphase material composed of four principal mineral phases: silicates of about

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* Corresponding author. Tel.: +33 0383596301.
E-mail address: christophe.auvray@univ-lorraine.fr (C. Auvray).
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86% (clays, 55%; quartz, 19%; feldspars/micas, 12%), sulphides of about 3% (pyrite), carbonates of about 10% (calcite, dolomite), and a form of organic kerogen of about 1% (Niandou, 1994; Schmitt et al., 1994; Niandou et al., 1997; Charpentier et al., 2001, 2004; Tinseau et al., 2006; Savoye et al., 2008).

The typical physical properties of the claystone are given in Table 1 (Niandou, 1994; Schmitt et al., 1994; Niandou et al., 1997; Chiarelli et al., 2003; Zhang and Rothfuchs, 2004).

2.2. Experimental equipment

The technical specifications of the nano- and micro-indentation testers and mini-compression (triaxial) cell are provided in Table 2.

2.2.1. Nano-indentation press

The nano-indentation apparatus consists of two cells, one of which contains the nano-indenter (CSM-Instruments) and the other contains an optical microscope for viewing the surface of the sample (Fig. 1).

For the nano-indentation tests, the surfaces of the sample must be as flat and as smooth as possible and must lie parallel to the support-stage axes. The distance between the support-stage plane and the indented surface must not vary by more than 5 μm. Though these requirements are systematic when preparing samples for indentation tests, it is particularly important that they are adhered to each other for the nano-indentation experiments (Vandamme, 2008; Miller et al., 2008; Auvray et al., 2015).

The experimental procedure used in this study was developed by the GeoRessources Laboratory (Nancy, France) and was presented in Auvray et al. (2013, 2015). The indentation procedure consists of pressing an indenter into the surface of a sample by applying an increasing normal load. The procedure is performed in a repetitive manner at different points on the sample surface at a constant interval along both the x- and y-axis. The load is directly applied by an electromagnet assembly attached to a vertical rod, the end of which houses a standard Berkovich diamond indenter. Displacement of the rod is measured by a capacitive detector and the rod is supported by two guide springs (Randall et al., 1997).

2.2.2. Micro-indentation press

In the nano-indentation tests, the surfaces of the sample must be flat and smooth. The experimental procedure used in this study was presented in detail in Magnenet et al. (2011b) and Grgic et al. (2013). In brief, the indentation procedure involves pressing an indenter into the surface of a sample by applying an increasing normal load.

Table 1
Typical physical properties of the material tested.

ρ_h (g/cm^3)	ρ_d (g/cm^3)	ρ_s (g/cm^3)	n (%)	w (%)
2.38–2.41[a]	2.18–2.27[a]	2.68–2.73[c]	4–9[a–d]	11–15[a,b]

Note: ρ_h: bulk density; ρ_d: dry density; ρ_s: skeletal density; n: total porosity; w: natural water content.

[a] Zhang and Rothfuchs, 2004.
[b] Chiarelli et al., 2003.
[c] Niandou, 1994; Niandou et al., 1997.
[d] Schmitt et al., 1994.

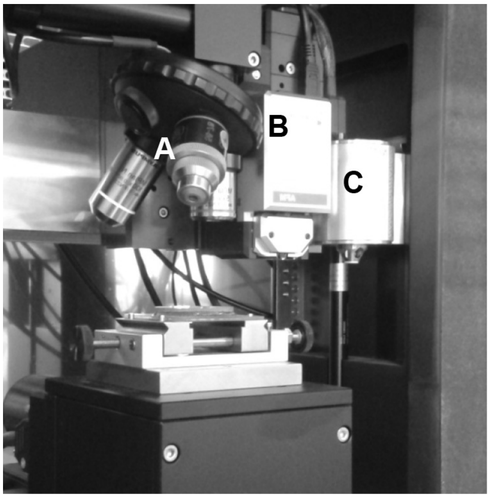

Fig. 1. Nano-indentation tester (A: Optical microscope, B: Atomic force microscope, C: Nano-indenter).

The micro-indentation press (Agotech) is equipped with a 5000-N force sensor. The piston holds a flat indenter tip made of tungsten carbide. The chosen size of the indenter tip ($\phi = 0.5$ mm) is representative of the micro-structure of the claystone. The size of the REV is 0.1 mm (Robinet, 2008; Robinet et al., 2012). The penetration depth corresponds to the mean value of the displacements, as measured using two LVDT sensors, and the force is measured with a force sensor positioned on the axis of the indenter tip (Fig. 2).

2.2.3. The compression cell

Meso-scale uniaxial compression tests were performed on centimetre-scale cylindrical samples ($h = 10$ mm, $\phi = 5$ mm) with a loading rate of 0.25 MPa/min. A mini-triaxial cell was developed in the laboratory for the meso-compression tests (Fig. 3). The experimental procedure used in this study was presented in detail in Grgic et al. (2013). In this assembly, the confining fluid is prevented from penetrating the rock specimen by means of a flexible sleeve placed around the cylindrical sample. The confining pressure is zero as the tests are essentially uniaxial compression tests. A self-compensated axial piston is used, and the cell is autonomous and does not require an external load press. Axial and transverse deformations were measured using four extensometers. Two of the gauges were diametrically opposed and used to measure axial deformation, and the other two gauges were used for measuring transverse deformation.

2.3. Equations for the modulus of deformability

The model of Oliver and Pharr (1992, 2004) was used for the nano- and micro-indentation tests. This model allows the Young's modulus (E_{it}) of the indented zone to be derived from load–

Table 2
Technical specifications.

Testing device	Load range (N)	Load resolution (N)	Maximum depth (mm)	Depth resolution (mm)	Maximum load rate (N/s)	Indenter
Indentation tester	0.001–0.500	4.0×10^{-8}	0.2	4.0×10^{-8}	4.0×10^{-3}	Berkovich
Micrope tester	0.1–5000	1.0×10^{-5}	2.5	0.01	0.5	Flat ($\phi = 0.5$ mm)
Mini-triaxial cell	1–20,000	0.1	2.5	0.01	5	–