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## Effect of transverse isotropy on the thermal volume changes of shales: insight from the Callovo-Oxfordian claystone

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### Abstract

An accurate monitoring of axial and radial thermal strains of a specimen of the Callovo-Oxfordian claystone, heated under constant confining stress condition close to that prevailing in the Bure Underground Research Laboratory at 490 m depth, evidenced a particular behaviour feature. The effect of the transverse isotropy of the claystone on thermal strains was confirmed, but it was also shown that the thermo-elasto-plastic overall volumetric response was mainly due to the axial response, in which some influence of the adsorbed water is suspected. Conversely, the reversible radial response observed along a temperature cycle could be modelled through a simple homogenisation method that confirmed that it was governed by the thermo-elastic expansion/contraction response of the constitutive minerals. Hence, the simultaneous occurrence of elastic thermal radial strains and elasto-plastic thermal axial strains was evidenced.

### Keywords

Anisotropy, clays, fabric/structure of soils, Radioactive waste disposal, temperature effects

### List of notations

$w$	Water content
$\phi$	Porosity
$\sigma$	Stress
$p$	Mean stress
$u$	Pore water pressure
$\alpha_i$	Linear thermal expansion coefficient of mineral $i$
$\alpha_{d, ax}$	Drained linear thermal expansion coefficient in axial direction, perpendicular to bedding
$\alpha_{d, rad}$	Drained linear thermal expansion coefficient in radial direction, parallel to bedding
$P_i$	Proportion of mineral $i$
$V$	Specimen volume
$t$	Time
$T$	Temperature

## 1 Introduction

2 The assessment of the thermal impact of heat-generating radioactive wastes in clays,  
3 claystones and shales has been investigated in Europe, with particular attention paid to  
4 the Boom clay, a stiff clay from Belgium (e.g. Hueckel and Baldi 1990, Sultan et al. 2002),  
5 and two claystones, the Opalinus Clay in Switzerland (e.g. Monfared et al. 2011, Favero  
6 et al. 2016) and the Callovo-Oxfordian claystone in France (e.g. Mohajerani et al. 2014,  
7 Menaceur et al. 2015a and b, Zhang et al. 2017 (their characteristics are given in Gens  
8 2013). In all concepts, the maximum temperature allowed in the host rock is 100°C. Few  
9 data are available on the thermal volume changes of claystones.  
10 On both the Opalinus Clay (Monfared et al. 2011) and the Callovo-Oxfordian claystone  
11 (Belmokhtar et al. 2017a), thermo-elastic volume changes have been observed at  
12 temperatures smaller than the highest temperature experienced during the geological  
13 history of the claystone (estimated around 50°C in the COx, Blaise et al. 2014), whereas  
14 plastic thermal contraction occurred at higher temperature. The physical mechanisms  
15 governing this contraction is poorly understood, most authors suspecting it to be due to  
16 the release of adsorbed water within the clay fraction.  
17 Thermal volume changes have often been monitored in a global way, with no  
18 consideration on the effects of the transverse isotropy that results, in clays and  
19 claystones, from the preferential orientation of clay platelets along the bedding plane. In  
20 this paper, these effects have been further investigated on the Callovo-Oxfordian  
21 claystone thanks to an accurate monitoring of the axial and radial strains by using strain  
22 gauges.

## 23 2. Material and methods

### 24 *The Callovo-Oxfordian claystone*

25 The Callovo-Oxfordian (COx) claystone is a low permeability sedimentary rock ( $k$  smaller  
26 than  $10^{-20}$  m<sup>2</sup>, Escoffier et al. 2005, Mohajerani et al. 2011, Menaceur et al. 2015b)  
27 deposited 155 millions years ago on the eastern fringe of the Parisian basin. At 490 m,  
28 in the middle of the COx layer where the Bure Underground Research Laboratory (URL)  
29 is located, the claystone is composed of a clay matrix (average 42% +/-11%) in which  
30 are embedded 30% carbonates, 25% quartz and a small fraction of feldspar (Conil et al.  
31 2018). The clay matrix is composed of platelets preferably oriented along the bedding  
32 plane, providing some degree of anisotropy to the texture. It contains 10-24% mixed-  
33 layer illite/smectite (with 50 – 70% smectite, Yven et al. 2007), 17-21% illite, 3-5%  
34 kaolinite, 2-3% chlorite (Gaucher et al., 2004). At the URL, the stress state is (Wileveau  
35 et al. 2007, Conil et al. 2018): total vertical stress  $\sigma_v = 12$  MPa, major horizontal stress  
36  $\sigma_H = 16$  MPa, minor horizontal stress  $\sigma_h = 12$  MPa, (hydrostatic) pore pressure  $u = 4.9$   
37 MPa, resulting in a mean Terzaghi effective stress  $p' = (\sigma_v + \sigma_H + \sigma_h)/3 - u = 8.4$  MPa  
38 and a Biot mean average effective stress of 8.9 MPa (with a Biot coefficient of 0.9,  
39 Belmokhtar et al. 2017b).

40 The specimens come from horizontal cores EST53650 and EST57185 (80 mm diameter)  
41 from the Bure URL. Specimens were diamond cored perpendicular to the bedding plane  
42 with a diameter of 38 mm and cut at a length of 10 mm with a diamond saw. The average  
43 water content of both cores ( $w = 7.7$  %) was determined on cuttings after oven-drying  
44 during 48 h at 105°C. The average porosity of both cores ( $\phi = 18.1$  %) was determined  
45 by hydrostatic weighing on some cuttings in unflavoured hydrocarbon, providing a  
46 degree of saturation of 93.9 %. An average suction of 20.1 MPa was measured with a  
47 dew point potentiometer (WP4C, Decagon Devices), indicating good specimen  
48 preservation with minimised evaporation after coring.

49  
50

51 *Thermal isotropic compression cell*

52 The used thermal isotropic compression cell (Figure 1, Belmokhtar et al. 2017a, b and  
53 Braun et al. 2019) accommodates the 38mm diameter porous disc. It is connected to two  
54 pressure-volume controllers (PVC, GDS Brand) for confining pressure (using oil) and  
55 back-pressure (using a synthetic water with same pore water salt composition as that of  
56 the COx, Table 1). Axial and radial strains were monitored by strain gauges glued parallel  
57 and perpendicular to bedding. Thermal effects were compensated by means of a  
58 reference gauge glued to a metal dummy piece placed close to the specimen, supporting  
59 the same stress/temperature path. A heating belt wrapped around the cell allowed for  
60 temperature control ( $\pm 0.1^\circ\text{C}$ ) and temperature was measured by a thermocouple inside  
61 the cell. Drainage was ensured through a thin porous metal disk at the specimen bottom.  
62 Given that specimens were slightly desaturated due to coring, storage and trimming  
63 (Monfared et al. 2011, Ewy 2015), they were placed on the dry porous disk and loaded  
64 to in-situ stress conditions (under constant water content, with only air escaping) at a  
65 rate of 0.1 MPa/mn. The ducts and porous disk were put under vacuum prior to be filled  
66 with synthetic pore water, injected under a pressure of 100 kPa. Water saturation was  
67 hence ensured under stress conditions equal or larger than in-situ, so as to minimize  
68 swelling and damage and to fit as much as possible with in-situ conditions.  
69 Instead of applying a drained temperature increase at low constant rate (Belmokhtar et  
70 al. 2017a), it was preferred to increase temperature by step and to wait for the dissipation  
71 of induced thermal pore pressure (see Delage et al. 2000 and Braun et al. 2019 for more  
72 details). The strain response, once stabilised, is representative of the drained thermal  
73 response of the claystone.

74 **3. Experimental data**

75 Four tests were performed. Samples ISO1 and 4 were loaded under 10 MPa and  
76 samples ISO2 to 4 under 8 MPa. Once the specimen in contact with water, both the back  
77 and the confining pressures were simultaneously and equally increased to reach a final  
78 pore pressure of 4 MPa, resulting in an effective isotropic confining stress of 10 MPa ( $\sigma$   
79  $- u = 14 - 4$  MPa) for ISO1 and 4, and of 8 MPa ( $\sigma - u = 12 - 4$  MPa) for ISO2 and 3,  
80 not far from the in-situ stress conditions.

81 Tests ISO2 and ISO3 were carried out according to a two stages protocol (like in Sultan  
82 et al. 2002), by keeping Valve V1 (Figure 1) opened, with a 4 MPa back-pressure applied  
83 by the PVC at the specimen bottom. The fast temperature increase resulted in an  
84 undrained volume and pore pressure increase, followed by a progressive volume  
85 decrease due to pore pressure dissipation. In tests ISO3 and 4, valve V1 was initially  
86 closed during 5 hours, prior to be opened and to allow the dissipation of thermal pore  
87 pressures during 20h.

88 The changes in volume with respect to temperature of tests ISO 3 and 4, run under  
89 constant Terzaghi effective mean stresses of 8 and 10 MPa and maximum temperatures  
90 of 90 and 80°C, respectively, are presented in Figure 2. Unfortunately, whereas the  
91 horizontal strain gauges work properly in all 4 cases, some problems were met with  
92 vertical strain gauges in tests ISO 1 and 2, and the corresponding bulk volume changes  
93 could not be monitored.

94 Both curves exhibit comparable trends, with however some differences due to the fact  
95 that small temperature cycles have been performed at 50°C and 70°C in test ISO4. Once  
96 the maximum temperature attained, both cooling curves are parallel. As already  
97 observed by Belmokhtar et al. (2017a), the thermal volume change curves start by a  
98 thermal expansion, defined by a slope close to that observed during the cooling phase  
99 afterwards, indicated by the two parallel straight dotted lines in Figure 2. The initial  
100 thermal expansion is followed by contraction, with an expansion/ contraction transition  
101 observed in both tests between 50 and 60°C, close to the maximum temperature  
102 experienced during the geological history of the COx, i.e. 50°C  $\pm$  5°C (Blaise et al.

103 2014). A similar trend was also observed on the Opalinus Clay by Monfared et al. (2011),  
 104 with a maximum temperature of 80°C.  
 105 Data from all tests are presented in terms of axial and radial strains in Figure 3, that  
 106 evidences a significant difference between strains perpendicular (axial) and parallel  
 107 (radial) to bedding. The radial strain response to the temperature cycle is fairly  
 108 reversible, at least for tests ISO1, 2 and 4, providing an average radial thermal expansion  
 109 coefficient  $\alpha_{d, rad} = 0.51 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$ . Conversely, contraction and irreversibility is quite  
 110 apparent in the axial (and comparable) responses of tests ISO 3 and 4. The slope  
 111 obtained along the cooling phase of tests ISO3 and 4 are quite close, giving a smaller  
 112 axial thermal expansion coefficient  $\alpha_{d, ax} = 0.21 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$ .

#### 113 4. Discussion

114 The data of tests ISO 3 and 4 in Figure 2 confirm the similarity of the thermal volume  
 115 response between the COx and Opalinus claystones, with an initial thermo-elastic  
 116 expansion followed by thermo-plastic contraction, once the highest temperature  
 117 experienced by the claystone has been reached. However, the accurate measurements  
 118 of strains parallel and perpendicular to bedding presented in Figure 3 provide further  
 119 information on the effects of the anisotropy due to the preferable orientation of clay  
 120 platelets along the bedding direction. A remarkably reversible strain response parallel to  
 121 bedding is observed along the temperature cycles in tests ISO 1, 2 and 4 (whereas some  
 122 degree of irreversibility is observed in test ISO3), indicating a thermo-elastic response  
 123 characterised by a slope providing the linear thermal expansion coefficient parallel to  
 124 bedding  $\alpha_{d,rad} = 0.51 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$ . This value can be compared to the literature data  
 125 reported in Table 2, that presents the thermal dilation coefficient of quartz, calcite and  
 126 feldspar, together with those, parallel and perpendicular to bedding, of (dry) muscovite,  
 127 a sheet silicate comparable to illite and smectite.

128 The value of  $0.35 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$  parallel to bedding provided by McKinstry (1965) is close to  
 129 our value ( $\alpha_{d,rad} = 0.51 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$ ). A more precise estimation can be made by using a  
 130 quite simplified homogenisation technique based on the schematic representation of the  
 131 grains embedded in a clay matrix, presented in Figure 4. Along the radial direction, one  
 132 can consider that the global thermal radial dilation  $\alpha_{d,rad}$  is the sum of those of the various  
 133 minerals, provided their effect is weighted by their proportion ( $P_i$ ), resulting in the  
 134 following expression:

$$135 \alpha_{d,rad} = \sum_1^3 \alpha_i P_i \quad (1)$$

136 Adopting the  $P_i$  and  $\alpha_i$  data of Table 2 (parallel for muscovite), one obtains a value  $\alpha_{d,rad} =$   
 137  $0,58 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$ , fairly close to the measured value of  $0,51 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$ . This good  
 138 correspondence confirms that the driving force of thermal strain parallel to bedding is the  
 139 reversible thermo-elastic dilation of minerals.

140 Things are less evident when considering the axial thermal response, for which the same  
 141 simplified approach provides a larger value of  $1.18 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$  (because of the larger  
 142 thermal dilation of muscovite perpendicular to bedding), whereas the slope of the cooling  
 143 curves of tests ISO3 and 4 provide an average smaller value  $\alpha_{d, ax} = 0.21 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$ . Due  
 144 to the preferred sub-horizontal orientation of the clay platelets, it is suspected that the  
 145 thermo-elastic volume changes (observed along the cooling path) are no longer only  
 146 dependent of the mineral thermal expansion/contraction, but also influenced by the  
 147 thermal response of the adsorbed water molecules, either within the platelets or in the  
 148 inter-platelets porosity (see Menaceur et al. 2016). The reason why this results in a  
 149 smaller coefficient is not yet fully understood. The thermo-plastic contraction observed  
 150 at higher temperature response is poorly documented in the literature.

151  
 152

153 **Conclusion**

154 Thermal tests conducted on specimens of the Callovo-Oxfordian claystone under  
155 constant effective stress confirmed the trend already observed on both the Opalinus Clay  
156 and the Callovo-Oxfordian claystone, i.e. a drained thermo-elastic expansion followed by  
157 a thermo-plastic contraction occurring once the maximum temperature experienced  
158 during the geological history has been overpassed.

159 The accurate monitoring of axial and radial strains by means of strain gauges confirmed  
160 the effects of the structural anisotropy of the Callovo-Oxfordian claystone. It was shown  
161 that the overall thermo-elasto-plastic volumetric behaviour was mainly due to the axial  
162 response (in which adsorbed water molecules appear to be involved), whereas the radial  
163 response remained thermo-elastic, only governed by the expansion coefficients of the  
164 constitutive minerals. This new observation illustrates quite a singular volume change  
165 response that results from the transversely isotropic nature of the Callovo-Oxfordian  
166 claystone.

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 219

220 **Tables**

221

222 Table 1. Composition of synthetic pore fluid close to the in-situ one, provided by Andra  
 223

	NaCl	NaHCO <sub>3</sub>	KCl	CaSO <sub>4</sub> , 2H <sub>2</sub> O	MgSO <sub>4</sub> , 7H <sub>2</sub> O	CaCl <sub>2</sub> , 2H <sub>2</sub> O	Na <sub>2</sub> SO <sub>4</sub>
Concentration [g/L <sub>water</sub> ]	1.95	0.13	0.04	0.63	1.02	0.08	0.70

224

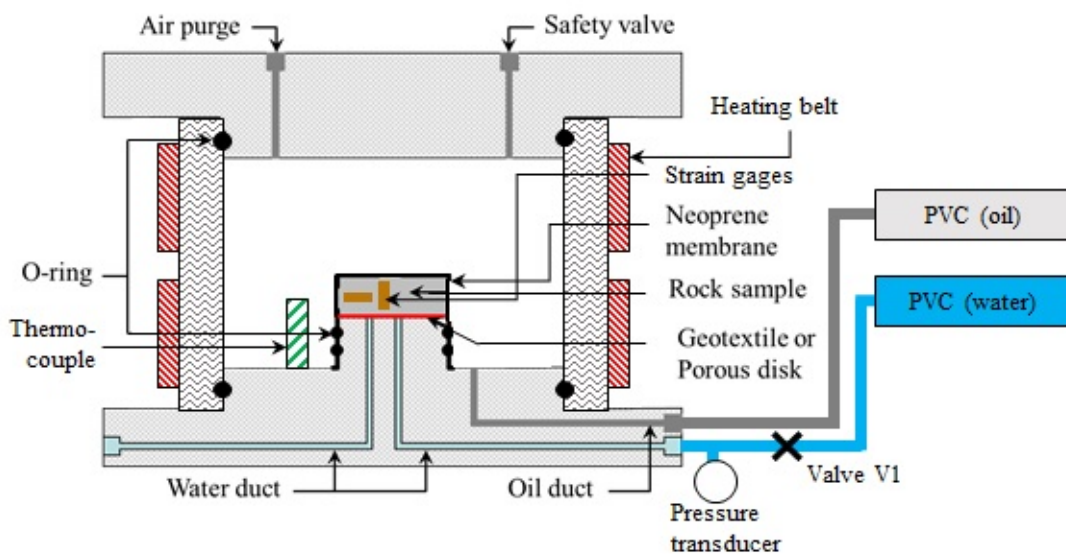
225 Table 2. Volumetric and linear thermal expansion coefficients of different minerals  
 226

Mineral	Mass-fraction $P_i$ (%)	Thermal expansion coefficient $\alpha_i$ ( $10^{-5} / ^\circ\text{C}$ )		
		volumetric	perpendicular	parallel
Muscovite	42	2.48	1.78 <sup>1</sup>	0.35 <sup>1</sup>
Calcite	30	1.4 <sup>2</sup>	0.5	
Quartz	25	3.3 <sup>3</sup>	1.1	
Feldspar	3	1.1 <sup>2</sup>	0.4	

<sup>1</sup>McKinstry (1965), <sup>2</sup>Fei (1995), <sup>3</sup>Palciauskas and Domenico (1982)

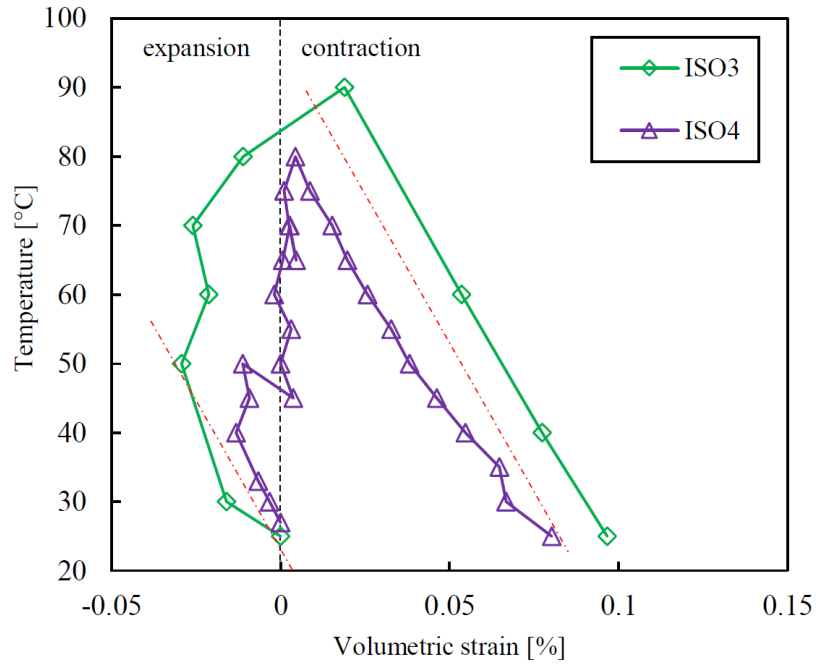
227

228 **Figures**

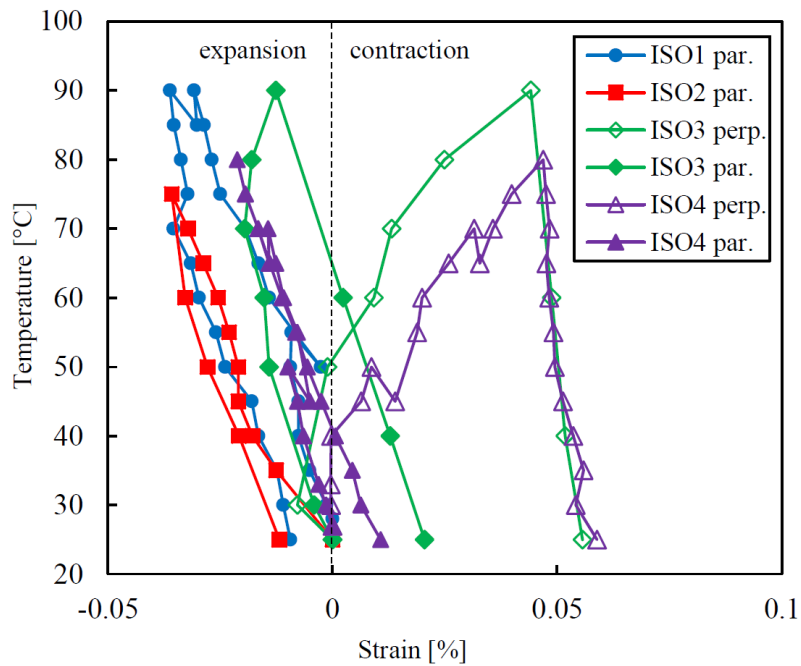


229

230 Figure 1. Temperature controlled isotropic compression cell for claystone specimens  
 231 equipped with strain gauges.

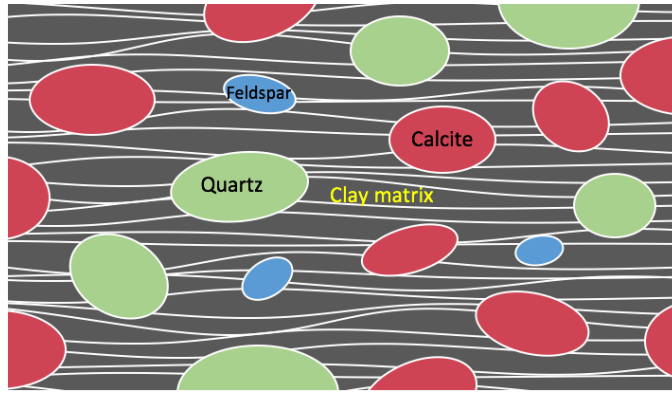


232  
 233 Figure 2. Drained volume changes with respect to temperature changes, measured  
 234 under constant stress conditions (8 MPa for ISO3 and 10 MPa for ISO4)  
 235  
 236



237  
 238 Figure 3. Thermal strain changes parallel to bedding (radial) and perpendicular to  
 239 bedding (axial) observed under constant confining stress (8 MPa for ISO3 and 10 MPa  
 240 for ISO4).  
 241





242

243 Figure 4. Schematic simplified model of the COx microstructure with granular minerals  
244 embedded within the clay matrix  
245