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Analysis of the Mechanical Behavior of Callovo-Oxfordian Claystone under Temperature: Contribution of 3D X-Ray Tomography

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Abstract

The Callovo-Oxfordian claystone (COx), as a host rock for radioactive waste disposal in France (Cigéo Project), will be subjected to coupled THM (Thermo-Hydro-Mechanical) processes, such as stress variations during excavation, changes in saturation, and thermal fluctuations induced by exothermic wastes. To ensure the short- and long-term safety of the disposal and optimize its design, it is essential to analyze the impact of these THM solicitations on the mechanical behavior of the clay host rock.

This study investigates the effect of temperature on the mechanical behavior of the COx claystone through triaxial compression tests conducted under various confining pressures, on samples oriented both parallel and perpendicular to the bedding planes. The tests, performed between 20 and 90 °C with a heating rate of 10 °C/h, revealed a decrease in peak strength with increasing temperature for the perpendicular orientation (from -10 % to -20 %), and no significant effect for the parallel orientation. This is due to the competitive effects of damage induced by pore-fluid overpressure and suction induced by the sample desaturation during heating with the latter being more significant in the parallel orientation.

3D micro-computed tomography was used to analyze the evolution of fractures between the initial state (after core sampling) and the final state (after the mechanical test). This approach highlights the combined influence of anisotropy, confinement and temperature on the fracture mechanisms. For the perpendicular orientation, axial stress generally tends to close pre-existing microcracks; however, at 90 °C, an interconnected fracture network develops, mainly concentrated in the lower part of the specimens. For the parallel orientation, temperature does not induce the development of an additional crack network and the rupture results from the progressive opening of pre-existing cracks, already aligned according to the direction of the bedding plane, without temperature exerting a significant influence on these mechanisms.

Keywords

COx claystone, Triaxial tests, Temperature, Microcracking damage, X-ray tomography.

1 Introduction

The Callovo–Oxfordian (COx) claystone, the host rock selected for the French deep geological repository (Cigéo Project), will be subjected to coupled Thermo-Hydro-Mechanical (THM) processes induced by excavation and heat emitted by radioactive wastes. In recent years, various studies on the thermo-hydro-mechanical (THM) behavior of COx claystone have been conducted, including in situ investigations carried out by the ANDRA (French National Agency for Radioactive Waste Management) and, in parallel, laboratory experiments performed by different research institutions under a range of conditions and experimental methods.

Although numerous laboratory studies have investigated the thermo-mechanical behavior of COx claystone, experimental results remain highly scattered due to differences in testing protocols, limited sample sizes, geological variability, and intrinsic heterogeneity (mineralogy, porosity, microcracking), as well as sample disturbance and saturation conditions(Zhang et al. 2014; Liu et al. 2019; Zhang 2021; Gbewade et al.2023;Wang et al. 2026). Thermal damage is further influenced by stress path, anisotropy, saturation, loading rates, and drainage conditions, highlighting the need for better quantification of these coupled effects.However, few studies combine controlled THM conditions with quantitative 3D fracture analysis.

This study aims to characterize the effect of temperature on the short-term mechanical behavior of COx claystone while reducing experimental dispersion through consistent and reproducible protocols. Triaxial compression tests are performed at different temperatures, confining pressures, and orientations, combined with 3D X-ray tomography to quantify fracture volumetric changes and link macroscopic responses to fracture network evolution.

2 Experimental procedures

The analyzed core samples were extracted from two horizontal boreholes (OHZ8573 and OHZ3011) drilled outside the Excavation Damaged Zone (EDZ) at the main level of the Meuse/Haute-Marne Underground Research Laboratory (~490 m depth). They were stored in T1 cells, concept developed by Andra, in order to preserve their initial degree of saturation and mechanical integrity (Conil et al. 2018). Cylindrical specimens (diameter 20 mm and height 40 mm) were tested parallel and perpendicular to the bedding plane. The mechanical properties of COx claystone being highly sensitive to water saturation (Zhang 2017; Conil etal. 2018; Grgic etal.2023; Gbewade etal. 2023);samples were therefore wrapped and stored under controlled humidity ($Sr > 90$ %).Physical characterization of the cores showed: wet density (ρ_w) ≈ 2.4 g/cm^3, dry density (ρ_d) ≈ 2.23 g/cm^3, grain density (ρ_s) ≈ 2.67 g/cm^3, total porosity (n_t) ≈ 17 %, natural water content (W_{nat}) ≈ 7 %, degree of saturation (S_r) ≈ 95 %, and calcite content ($CaCO_3$) ≈ 18 % (OHZ8573) and ≈ 26 % (OHZ3011).

Before instrumentation, samples were scanned using 2D X-rays to select the least damaged specimens and avoid biases from preexisting microcracks. 3D X-ray scans were performed on some selected samples with the following acquisition parameters: 1440 projections, 3 images per projection, 1000 ms exposure per image, 120 kV voltage, 200 μA current, and a voxel resolution of 20 μm. Data were analyzed using VGStudio MAX software. Strain gauges measured axial and lateral deformations accounting for transverse isotropy (perpendicular orientation: Fig.1a; parallel orientation: Fig.1b. Volumetric strains were calculated from the measured axial and lateral components, with compression taken as positive.After instrumentation, specimens were sealed in a triaxial cell and immersed in a synthetic-oil bath for temperature control (Fig.1 e-f).

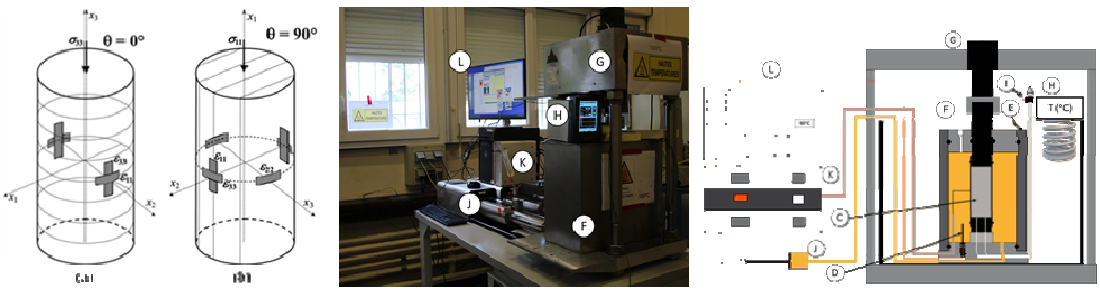


Fig. 1 Strain gauge arrangement and coordinate system for COx claystone specimens in both orientations—(a) perpendicular and (b) parallel to the bedding plane—along with a photo and schematic of the experimental setup: (c) sample, (d). thermocouple, (e) triaxial cell, (f) stainless-steel bath, (g) mechanical press, (h) immersion heater, (i) drainage valve, (j) oil pump, (k) data acquisition system, and (l) press and pump controller.

Four temperatures (20, 45, 70, 90 °C) and four confining pressures (0.5, 4, 8, 12 MPa) were tested on COx claystone in parallel and perpendicular orientations to bedding. Hydrostatic confining pressure was applied at 1×10^{-3} MPa/s and maintained constant (Fig.2). Heating (for T > 20 °C) used a 10 °C/h ramp, generating transient undrained conditions due to the low permeability of the material, followed by 14 - 16 h equilibration to dissipate pore overpressures (Souley et al. 2026). Deviatoric loading was applied at 0.04 μm/s (10^{-6} s^{-1} considering the specimen height) with unloading-reloading cycles each 4 MPa. Before each unloading, a 15 min relaxation phase was applied to dissipate the viscous effects, thus allowing a better determination of elastic parameters.

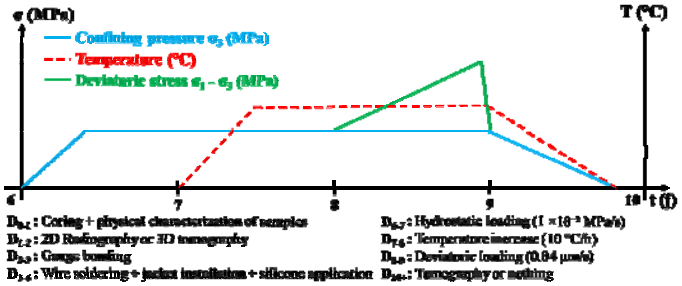


Fig. 2 Stresses and temperature paths during the tests.

After testing, specimens were removed from the Viton membrane, strain gauges were taken off, and samples were protected with plastic and cellophane before a final X-ray tomography scan. Due to the low permeability and short drainage length (38-42 mm), the chosen deformation rate (10^{-6} s^{-1}) ensured quasi-drained conditions (Liu et al. 2019).

3 Influence of temperature on mechanical behavior

A total of 99 triaxial compression tests were conducted under parallel and perpendicular orientations across various confining pressures (0.5, 4, 8, and 12 MPa) and temperatures, with Fig.3 illustrating the peak deviatoric stress versus temperature for both orientations, along with cubic polynomial fits to describe the trends. At least two tests were conducted for each temperature at 4, 8, and 12 MPa, while only one test was performed at 0.5 MPa above 20 °C. First, it is worth noting that peak stress increases with confining pressure, for both parallel and perpendicular orientations. Second, for specimens perpendicular to the bedding plane, peak strength generally decreases with increasing temperature (16-21 % reduction at 90 °C) for confining pressures of 4 - 12 MPa, whereas for parallel orientation the temperature effect remains negligible.

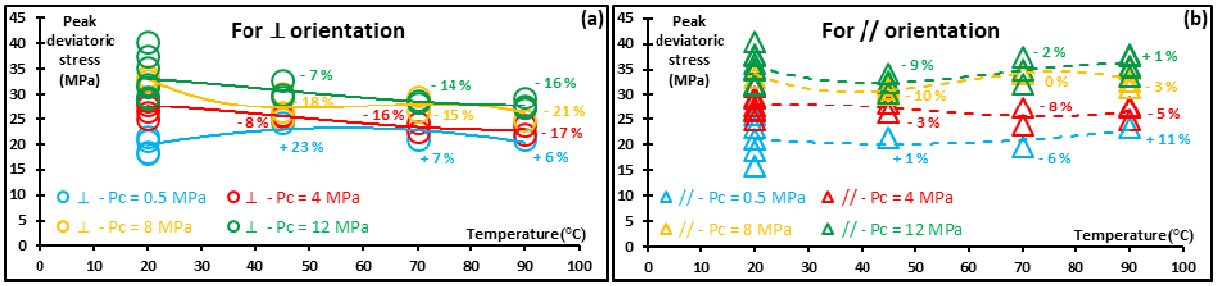


Fig.3 Evolution of peak deviatoric stress as a function of temperature (20, 45, 70, and 90 °C) for different confining pressures (0.5, 4, 8, and 12 MPa) and orientations – (a) Perpendicular; (b) Parallel.

The decrease in strength with increasing temperature for the perpendicular orientation does not follow a linear trend. This behavior has already been reported in the literature (Gbewade et al. 2023 ; Wang et al. 2026). This trend may be related to the limited temperature range studied and the intrinsic heterogeneity of the material, making the interpretation of this behavior challenging. For specimens parallel to the bedding plane, temperature has little effect on peak strength. The suction (capillary attraction forces), which plays a major role due to the small pore size of COx claystone, can explain this different impact of temperature as a function of orientation. Indeed, heating induces desaturation of the samples and therefore the development of suction forces, thus generating an overall confining effect, which partially hides the negative impact of temperature on the peak strength for both orientations. In addition, at mesoscopic scale, suction generates also compaction of bedding planes, as already proposed by Zhang et al. (2012). For this orientation, the failure is mainly due to the opening of bedding planes aligned in parallel to the axial stress. Therefore, the mechanical strength significantly increases due to the action of suction, thus explaining why temperature has almost no negative impact for this orientation.

Heating a low-permeability porous medium such as COx induces pore fluid overpressure, primarily due to the large difference between thermal expansion coefficient of pore water (2.6×10^{-4} °C^{-1} at 25 °C and 5.8×10^{-4} °C^{-1} at 80 °C) and that of the argillite solid skeleton (2.6×10^{-5} °C^{-1} at 25 °C and 11×10^{-5} °C^{-1} at 80 °C) (Mohajerani 2011; Zhang et al. 2017). An initially high heating rate (10 °C/h) was deliberately chosen to simulate in situ undrained conditions, generating significant interstitial water overpressure due to the very low permeability of the argillite. This overpressure was used to analyze microfracture-induced damage, and it likely dissipated during the 14 - 16 h waiting period imposed on each sample before deviatoric loading. Moreover, COx argillite can be regarded as a composite material composed of a clay matrix containing carbonate and quartz inclusions. These constituents exhibit different thermo-mechanical properties, particularly in terms of thermal expansion (Belmokhtar 2017). Mismatches in expansion coefficients - similar for clay and quartz ($\alpha_s = 3.4 \times 10^{-5}$ °C^{-1}) but two to three times lower for calcite and feldspar ($\alpha_s = 1.38 \times 10^{-5}$ °C^{-1}) - may induce localized debonding or shear at mineral interfaces, ultimately degrading the mechanical properties of the rock.

4 Tomographic observations

Fig.4a shows two samples (20 °C and 90 °C) cored perpendicular to the bedding plane and tested under a confining pressure of 12 MPa. Initially, the 20 °C sample exhibits coring-induced cracks mostly parallel to the bedding planes, reflecting the transverse isotropy of COx argillite and the role of bedding as weakness planes. After mechanical loading, a macroscopic shear fracture inclined at about 45° develops in the upper part of the sample, consistent with the expected failure angle under confining pressure. While many initial cracks and bedding planes close under axial stress and confining pressure, some preexisting inclined cracks promote stress concentration and coalescence, leading to final macroscopic failure. Quantitative analysis (Fig.4b, green squares) shows a negligible change in total fracture volume (2.1% to 1.9%), as the reduction of initial crack volume is offset by the formation of the macroscopic fracture.

Initially, the 90 °C sample exhibits very few coring-induced microcracks, representing a negligible volume (0.1%), likely due to a different coring device that generated less initial damage. These cracks do not influence the subsequent test. After heating and loading, a vertical macroscopic fracture (≈90°) develops, accompanied by a marked increase in fracture volume (0.1% to 3.9%)(Fig.4b, red squares), reflecting macroscopic failure. X-ray tomography reveals a dense, interconnected fracture network concentrated in the lower part of the sample, identified as the main deformation and fracture propagation zone, likely enhanced by edge effects and higher thermal gradients in the metallic base plate. The main fracture propagates preferentially through the matrix near inclusion-rich areas, where stress concentrations promote crack initiation and coalescence, consistent with the findings ofShi et al. (2021).

These results indicate that, for the perpendicular orientation, temperature promotes the development of an additional fracture network with specific orientations, which partly explains the moderate but noticeable effect of this parameter on the peak strength observed for this configuration (Fig.3a).

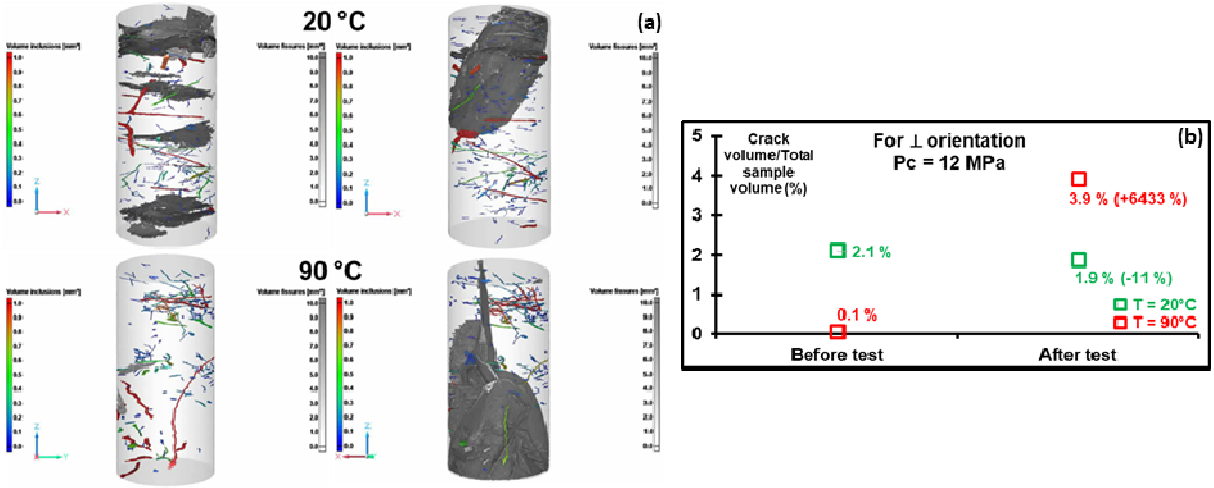


Fig.4 (a) 3D tomography scans of two samples oriented perpendicular to the bedding plane, before testing (left) and after testing (right), under Pc=12 MPa, at T=20 °C (top) and T=90°C (bottom); (b) Comparison of fracture volume before and after testing for samples perpendicular to the bedding plane under Pc=12 MPa at two different temperatures: T=20°C → green; T=90°C → red.

Fig. 5a highlights two samples (20 °C and 90 °C) cored parallel to the bedding plane and tested under a confining pressure of 12 MPa. Initially, the 20 °C sample exhibits coring-induced cracks mainly parallel to the bedding planes. After mechanical loading, a failure fracture inclined at about 60° to the horizontal develops, consistent with expected shear mechanisms under these conditions. Unlike the perpendicular sample tested at room temperature, a significant increase in fracture volume is observed after testing (1.6% to 2.7%) (Fig. 5b, green triangles). This increase results from the progressive opening of preexisting cracks aligned with the bedding plane (parallel here to the major principal stress), combined with the formation of the main macroscopic failure fracture. Preexisting cracks slightly inclined to the bedding likely acted as initiation sites, promoting strain localization and fracture coalescence.

Initially, the 90 °C sample exhibits a higher density of coring-induced microcracks, representing 2.7% of the total volume. After heating and loading, tomography reveals the development of a diagonal macroscopic failure fracture inclined at about 60° to the horizontal, also consistent with the expected shear angle in clayey materials. This failure is accompanied by a significant increase in fracture volume (2.7% to 4.4%)(Fig. 5b, red triangles). Most preexisting cracks keep their original morphology and appear to have had limited influence on the main fracture path, which adopts an inverted "Y" geometry, with part of the initial network remaining isolated while another portion connects to the newly formed shear fracture.

These results indicate that, for the orientation parallel to the bedding plane, and given the 20 μm resolution of the tomographic imaging, increasing the temperature does not generate additional fracture networks within the samples, partly explaining the lack of temperature effect on peak strength for this orientation (Fig.3b).

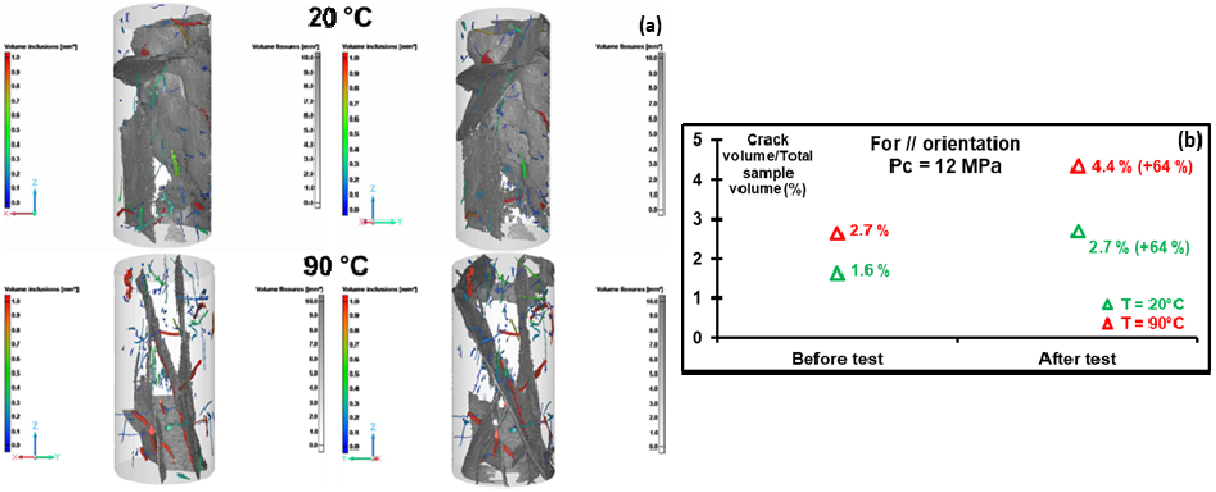


Fig. 5(a) 3D tomography scans of two samples oriented parallel to the bedding plane, before testing (left) and after testing (right), under Pc=12 MPa, at T=20°C (top) and T=90°C (bottom); (b) Comparison of fracture volume before and after testing for samples parallel to the bedding plane under Pc=12 MPa at two temperatures: T=20°C → green and T=90°C → red.

5 Conclusions

The experiments showed that peak strength of the COx argillite decreases with increasing temperature for the perpendicular orientation, while temperature has no effect on the parallel orientation. The initial heating phase induced interstitial water overpressure due to thermal expansion, likely reducing peak strength in the perpendicular orientation due to microcracking damage, with the effect increasing at higher temperatures. For the parallel orientation, suction induced by sample desaturation during heating hides the negative temperature impact on strength due to the compaction of bedding planes. Confining pressure also plays a significant role: higher confinement increases peak strength, confirming that confinement enhances the material's resistance to deformation.

Tomographic results highlight the combined influence of anisotropy, confinement, and temperature on fracturing mechanisms. For the perpendicular orientation, axial stress generally tends to close preexisting cracks aligned along the bedding plane. However, at 90 °C, an interconnected fracture network develops, mainly in the lower part of the samples, linked to edge effects and inclusions. In contrast, for the parallel orientation, temperature does not promote additional fracturing, and failure

occurs through progressive opening of preexisting cracks aligned with the bedding planes. Temperature appears to have little effect on the deformation mechanisms for this orientation.

These results enable the analysis of temperature effects on the mechanical behavior of COx by linking macroscopic responses to fracture network geometry. They also highlight the key role of structural anisotropy (orientation relative to bedding) in controlling thermo-mechanical fracturing and emphasize the importance of carefully controlling experimental conditions to predict the long-term behavior of this host rock. These findings also contribute to improving the design and safety assessment of the Cigéo repository, particularly for optimizing repository layout.

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