MULTI-SCALE ANALYSIS OF A POROUS CARBONATE ROCK UNDER TRIAXIAL CONDITIONS

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Introduction

Sequestration of $\text{CO}_2$, energy storage ($\text{H}_2$), or geothermal energy in deep aquifers or depleted reservoirs: porous and permeable rocks

- Sedimentary rocks: sandstones or limestones

**Deformation bands in porous rocks**

- Heterogeneous and/or localised deformation in triaxial conditions for carbonate rocks with porosities >20-25%;
- Deformation bands can reduce permeability up to several orders of magnitude;
- Presence of fluids can reduce the resistance

Post-localisation behavior? Geometry, number + spacing of deformation bands, relation to the microstructure at different scales?

**Experimental investigation**
Saint-Maximin Limestone (SML) was extracted from Rocamat quarry, in Saint-Maximin-sur-Oise

- Grainstone;
- 80% calcite and 20% quartz;
- 38% of connected porosity
- Isotropic
Previous results


Strain Localisation in SML [1,2]

- Deformation bands visible at all confining pressures
- Micromechanisms only visible post mortem on thin blades (SEM imaging)

How to access bands 3D geometry?
Experimental Method - *Ex situ/post mortem Testing*

Y. Abdallah, 2019

XRCT image of reference state
Voxel size = 24 μm

Triaxial test conducted at a confinement on 40x80 mm samples

Unloading after beginning of yielding

XRCT image at Stage 1

Loading at confinement + “continuation” of deviatoric loading

XRCT of sample at Stage 2

Unloading

Strain Maps

Porosity maps

1 step = 1.5 day
1 scan = 24 hours + frequent leaking

CFM 2/09/2022

Y. Abdallah, 2019

Multi-scale analysis of a porous carbonate rock
Triaxial press for 40 mm in diameter samples
Experimental Setup

Multi-scale analysis of a porous carbonate rock
Local Porosity Map Calculation

→ Explore relationship between bands and microstructure by calculating local porosity from X-ray images

On the whole sample: Mean Grey Level (\( \bar{\text{GL}} \)) is known with corresponding porosity, \( \phi \). \( \text{GL} \) of a pore is manually evaluated from a large pore in XRCT image. \( \text{GL} \) of solid phase is calculated (calcite and quartz attenuation coefficients are similar).

\[ \bar{\text{GL}} > \bar{\phi} \cdot g_{\text{pore}} + i \]

Locally: The unknown is the local porosity \( \phi_{\text{local}} \), deduced from local mean GL.
Results for Sample Tested Under High Confinement (11 MPa)

After Stage 1

SEM images of the sample under high confinement.

XRCT images with a 24 µm resolution.
How Would Strain Be Accommodated In Single Dense/Porous Zones?

Standard size sample (40 mm in diameter and 80 mm in height) and porosity map with a 40 voxels window

8mm in diameter samples cored in dense and porous zones

Before tests, cored samples are imaged to visualize their microstructure and evaluate the porosity field

Porous microstructure

Dense microstructure
Experimental Method
A new testing device for micro tests on samples with diameter smaller than 15 mm, developed at LMS for in situ (4D-imaging) tests.
Experimental Setup

Beamline Psiché @ Synchrotron Soleil, A. King, proposal 20220588, September 2022
Synchrotron Imaging Advantages

- 40 mm sample with 24 µm voxel size
- 15 mm sample with 8.5 µm voxel size
- 8 mm sample with 3.25 µm voxel size

+ duration of a scan = 10 min (vs 16h at Navier)
Typical results
Overview – porous samples

Multi-scale analysis of a porous carbonate rock
Overview – dense samples

Multi-scale analysis of a porous carbonate rock
Overview – all triaxial tests on SML

- 8 mm denses
- 8 mm poreux
- 15 mm

Catherine 40mm (37%)
Abdallah, 2020 40mm (38%)
Baud et al., 2009; 2017 40 mm
8 mm denses (35-37%)
8 mm poreux (42-45%)
Triaxial Test on a Dry, Dense (= 35%) 8 mm Sample at a Confining Pressure of 6 MPa

Horizontal cross section of an 8 mm dense sample obtained by XRCT 3D imaging and zoom on microstructure

Multi-scale analysis of a porous carbonate rock
Triaxial Test on a Dry, Dense (= 35%) 8 mm Sample at a Confining Pressure of 6 MPa

Multi-scale analysis of a porous carbonate rock
Triaxial Test on a Dry, Dense (= 35%) 8 mm Sample at a Confining Pressure of 6 MPa
Triaxial Test on a Dry, Dense (= 35%) 8 mm Sample at a Confining Pressure of 6 MPa
Triaxial Test on a Dry, Porous (= 41%) 8 mm Sample at a Confining Pressure of 6 MPa

Horizontal cross section of an 8 mm porous sample obtained by XRCT 3D imaging and zoom on microstructure

Local density map

Multi-scale analysis of a porous carbonate rock
Triaxial Test on a Dry, Dense (≈ 35%) 8 mm Sample at a Confining Pressure of 6 MPa
Multi-scale analysis of a porous carbonate rock
Triaxial Test on a Dry, Dense (= 35%) 8 mm Sample at a Confining Pressure of 6 MPa
Triaxial Test on a Dry 40 mm Sample (= 38%) at a Confining Pressure of 6 MPa (Abdallah, 2019)

- Porosity heterogeneity at centimeter scale;
- Transition from brittle to ductile behaviour at 6 MPa, perfectly plastic behaviour;
- Strain localisation is already visible on shear strain volumetric map right after plasticity onset at stage 1;
- New bands are formed at stage 2;
- The bands pass through high porosity zones, but cut sometimes through dense zones to connect high-porosity zones.
Perspectives
Conclusions

- In situ triaxial tests on small samples permit to explore micromechanisms of deformation at small scale;

- Porous and dense samples exhibit different behaviors. Dense samples have a larger elastic domain and the brittle/ductile transition occurs at a higher confinement (11 MPa as compared to 3-6 MPa for porous samples);

- Under hydrostatic loading, pore collapse is observed at 13-14 MPa in porous samples while it occurs above 20 MPa for dense ones. At macro-scale (40 mm in diameter samples), pore collapse is observed at 16 MPa;

- Diffuse compaction has been observed in small porous samples, while a single deformation band is observed in dense ones and spreads progressively throughout the whole sample, while several deformation bands initiate and develop through mostly porous zones at macro-scale.
Future Tasks

• Confirm first observations by systematic DVC analyses to build 3D deformation maps and porosity maps;

• Link local porosity evolutions with hardening of sample;

• Identify the micro-mechanisms (pore collapse, grain rearrangement, inter or intra granular fracturing, etc.) involved deformation band formation + determine their sequence;

• Define the key features of the microstructure (beyond porosity) that differentiate dense and porous sample;

• Complete data with ex situ testing in order to fully define the yield loci for porous and dense samples;

• Link permeability at several scales to deformation history;

• Upscaling from the small samples to standard-sized samples.