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Experimental Rock Deformation under µCT: two new apparatuses

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Summary

Physical and mechanical properties of rocks are essential information for geophysical methods. However, these properties are intimately related to microscopic features such as pore shape and distribution, grain orientation, saturation and fluid distribution. Therefore, to fully and deeply comprehend macroscopic rock properties there is a quest to understand such microscopic aspects. The present contribution reports the development and the preliminary results obtained with a couple of new high pressure vessels which are paired with a micro-CT system to investigate the influence of fluid distribution and microstructural features on the physical and mechanical properties of rocks.

Introduction

Geophysical methods rely on the macroscopic rheological behaviour of materials. However, the macroscopic (i.e. apparent) rheology of rocks often depends on microscopic aspects. For instance, in granular porous media elasticity is mainly affected by the grains and pores distribution and orientation, but also by the cracks closure, and the pore-fluid content and distribution. All these aspects cannot be easily observed during laboratory tests. And often, such observations are performed upon to destructive tests. For instance, Tisato and Quintal (2013) demonstrated that the non-uniform distribution of liquid and gas in sandstones causes viscoelasticity, which, in turn, subtracts a substantial amount of energy from a propagating seismic wave (i.e. causes attenuation). Nevertheless, the authors were unable to compare the calculated and the real fluid distribution as their apparatus was lacking of an “in-situ” analytical tool to investigate this aspect.

Similarly, studies about dynamic friction in rocks, which are important for natural and induced seismicity, are rather limited to the measurement of apparent physical and mechanical properties. And the role of powders, nano-powders, and gouge formation, observed by many authors in their experiments, is still rather unclear (Tisato et al., 2012). The investigation of the structures and the gouge produced by these experiments was only possible perturbing the gauge layer (Tatone and Grasselli, 2014), thus introducing uncertainties on the final state of the deformed sample which could lead to errors in our understanding of the wearing processes.

Many natural and anthropogenic activities cause thermodynamic disequilibria in subsurface domains (i.e. physical property variations), which in turn causes a variation in the signals recorded by a contingent geophysical monitoring. For instance, carbon sequestration is believed to cause the dissolution and/or precipitation of mineral phases in crustal rocks. Nevertheless, how such interactions affects physical properties is still rather unclear and unstudied. Again, such a lack is related to the inability to perform laboratory experiments and, concurrently, “see” the evolution/response of the sample interior.

Lately, X-Ray tomography became a versatile and effective technique to image materials and their saturating phases. Here we report the design of two new high-pressure X-Ray transparent vessels which can fit and perform measurements inside the X-Ray computed tomography apparatus (µCT) installed at the University of Toronto. Hence, using these machines the scientist can measure changes in physical properties in the sample (e.g. seismic wave attenuation) and, simultaneously, link them to saturation variations, or precipitation-dissolution of minerals. We discuss how the use of the μCT will allow shedding light on rock physics, and present the preliminary results obtained with the new vessels in the μCT. This technological development and the results obtained will better understanding of rock physics to aid geophysical methods.

Methods

Viscoelastic properties of rocks – Seismic Wave attenuation (1/Q)

The X-ray transparent vessel used to measure seismic wave attenuation is provided of a vertical actuator which applies the vertical stress on the specimen. A piezoelectric motor (PZT, Fig. 1 green part) controlled by a high voltage amplifier generates a sinusoidal variation of the vertical stress necessary to estimate the complex Young's modulus (E_Z). E_Z is calculated according to the amplitudes of the sinusoidal stress and strain. In fact, a load cell (Fig.1 red part) placed between the PZT and the sample (Fig. 1 brown part) measures the sinusoidal force, and a cantilever (Fig. 1 orange part) measures the sinusoidal bulk vertical shortening of the sample. Vertical stress and strain are calculated according to the end-faces area of the sample. Moreover the attenuation related to the Young's modulus can be calculated as:

$$1/Q_E = \tan(\varphi)$$

where φ is the phase shift between the sinusoidal stress and strain (Lakes, 2009).

As the apparatus is equipped with an hydraulic circuit it is possible to vary the confining pressure between 0 and 30 MPa, the pore pressure between 0 and 20 MPa and saturate the sample with different fluids. Ideally, the main plan is to measure attenuation before and after injection of brine and CO_2, which will cause dissolution and/or precipitation of mineral phases, to better understand the impact of microstructural and saturation variations on the rock viscoelasticity.

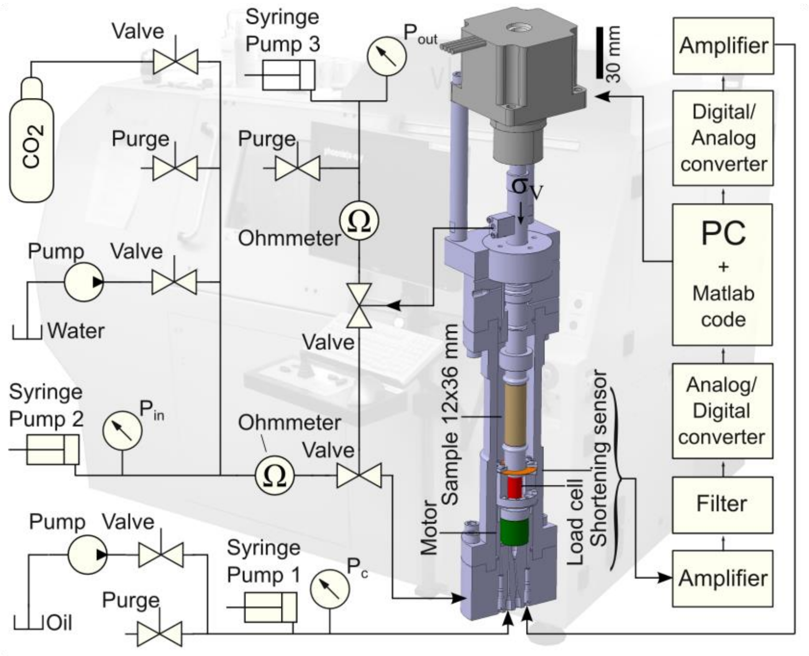

Figure 1. Schematic of the newly designed vessel called ERDμ, which is X-ray transparent. A micro-CT (in the background) can be used to image the confined sample simultaneously to the measurement of seismic attenuation. The ERDμ can confine a sample 12 × 36 mm as high as 30 MPa (after Tisato et al., 2014).

Rock wearing and friction

Another version of the X-ray transparent vessel, equipped with a torsion motor, can perform rotary shear tests inside the X-ray tomography apparatus (μCT) installed at the University of Toronto. Such a setup allows performing rotary shear tests on 12 mm diameter samples and image the slipping surfaces and the forming gauge without disturbing the specimens. Rotary shear tests are conducted at sub-seismic slip rates (e.g. from 0.8 – to 48 mm/s) and for several incremental short slip distances (e.g. 180 steps of 2 deg) in order to reconstruct the 4D evolution of the slipping surfaces and the gauge layer. While rotating the specimen normal stress, displacement and torque are acquired by an analog to digital converter with a sampling rate of 2 kHz. The samples can be confined up to 10 MPa and saturated with liquids by means of a hydraulic circuit. Such experiments will help understanding the impact of the gauge layer on the rock friction.

Examples

Here we present some preliminary results, as the present contribution mainly focuses on the discussion of the design and the potential studies which might be conducted with these new X-ray transparent vessels. The preliminary X-ray tomographies show that the newly designed vessels whose irradiated part is made of aluminum alloy (7075-T6), allow effectively to image the enclosed sample (Fig. 2). In particular, there is no evidence of noise or artefacts introduced by the high pressure vessel (Fig. 2DEF).

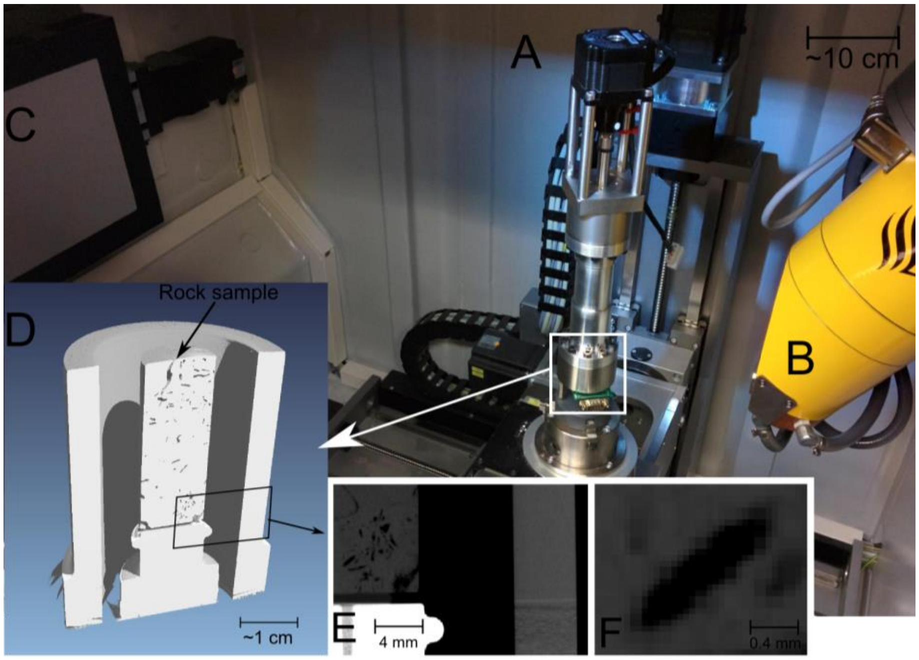

Figure 2. A) The X-ray transparent vessel ERDμ is placed inside the micro-CT apparatus of University of Toronto between the X-ray tube (B) and the CCD sensor (C). D) Longitudinal section of 3D model obtained from X-ray tomography imagery. The rock sample is Muschelkalk, a carbonate from Switzerland which is a potential target rock for carbon sequestration. E) Detail of panel D shown as tomography image. It is possible to observe the sample (material ~$CaCO_3$), the sample older (material AISI304) and the wall of the vessel (material 7075-T6). F) Detail of panel E showing a pore in the sample.

The preliminary measurements of vertical force and shortening, necessary to estimate seismic wave attenuation, have measurable amplitudes (Fig. 3). The quality of the shortening signal will be improved by an updated arrangement of the strain gauges on the cantilevers.

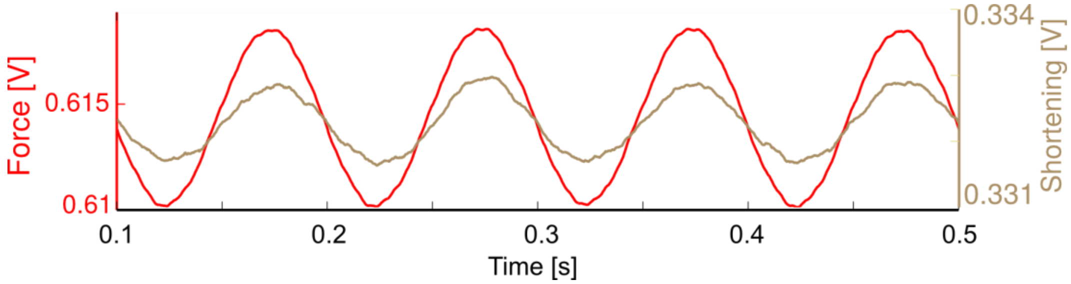

Figure 3. Example of raw signals obtained with the X-ray transparent vessel ERDµ. The 10 Hz sinusoidal force applied vertically on the aluminum sample (red curve), causes the sinusoidal shortening of the specimen (brown curve). The magnitude of the shortening is ~150 nm.

Conclusions

Albeit experimental rock deformation has been greatly improved in the last century, one of the last challenges which must be overcome is to image, in time and space, complex deformation experiments. The presented apparatus will serve to this scope. In addition, the experimental apparatus, equipped with the torsion motor, will help to better understand the formation and the geometrical evolution of a brittle shear zone.

In conclusion the newly designed and built equipment will help to uncover the relationships between chemical/physical fluid-solid interactions and variations of effective elastic properties. Such an achievement will aid the monitoring of subsurface domains. Moreover, they will be employed to link the evolution of the elastic parameters to the observed changes in the internal structure of the investigated sample, this research will allow, for instance, to calibrate the experimental methodology and to use the variation in dynamic Young’s modulus to infer the damage/change in the subsurface.

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