ICTMS 2022

5th International Conference on Tomography of Materials & Structures
27th June- 1st July 2022, Grenoble, France

# MULTI-SCALE MULTI-DIMENSIONAL MICROSTRUCTURAL IMAGING OF PRECAMBRIAN GRANITE USING X-RAY MICRO-TOMOGRAPHY (µCT) COMBINED WITH NANOSIMS, µRAMAN SPECTROSCOPY AND SEM-EDS

Kanchana Kularatne[1], Valentine Combaudon[1,2], Olivier Sissmann[2], Pascale Senechal[1], Maria Angels Subirana[1], Dirk Schaumlöffel[1], Caroline Delhaye[3], Arnaud Proietti[4], Eric Deville[2], Hannelore Derluyn[1]
[1]Université de Pau et des Pays de l'Adour, France ; [2]IFP Energies Nouvelles, France ; [3]Université Bordeaux 1, France ; [4]Centre de Microcaractérisation Raimond Castaing, France.

*Speaker: kanchana.kularatne@univ-pau.fr

## 1. Introduction

Phase identification, quantification, and 3D visualization in granite using µCT is challenging because (i) granite is a polymineralic rock and could contain minerals with approximately similar densities and molar masses, (ii) the lack of chemical information in µCT [1]. Here, we present a workflow developed to image the multi-scale multi-dimensional microstructure of a highly heterogeneous granite from the Precambrian basement of Kansas, USA, to better understand the fluid-rock interactions leading to an atypical production of native $H_2$ reported in [2].

## 2. Materials and Methods

We obtained a drill core from a $H_2$-emitting well (DR-1, at 452 m depth) in Kansas [2]. A small cylinder (d=8.75 mm, l= 17 mm) was drilled and imaged at low resolution (6 µm voxel size) with X-ray µCT. The cylinder was cut and polished at 4.1 mm (from its top), to expose a cluster of mineral grains with different grey values, observed in the low-resolution scan. A distinct fracture network filled with (re)precipitated minerals was observed throughout the scanned volume as well. On the polished surface, we conducted µRaman point analysis, scanning electron microscopy with energy dispersive spectroscopy mapping (SEM-EDS) and nano scale secondary ion mass spectroscopy (nanoSIMS). SEM analysis is performed on the whole surface, whereas NanoSIMS is performed on a selected area (150 µm x 400 µm) at 50 nm resolution, to obtain chemical information on minerals filling the fine fractures. Finally, a sub-core of 2 mm diameter was imaged at high resolution (2 µm voxel size), to visualize and extract the fine fractures. The data were analyzed and merged using the ORS Dragonfly software package [3].

## 3. Results and Conclusion

Precambrian granite from Kansas is composed of coarse grain orthoclase (>1mm), diopside (~800µm), pargasite (500µm-1mm), fayalite (400µm-1mm) and oxides (rutile, ilmenite, magnetite). We correlated the grey values from the low-resolution tomography with the µRaman data. Grey values of orthoclase are distinct, however, overlapping grey values were observed among fayalite, pargasite, diopside and also among oxides such as rutile, ilmenite, and magnetite. Fayalite is highly fractured and those fractures are filled with phyllosilicate with grey values close to pargasite. To propagate the chemical information through the 3D µCT volume of the 8.75 mm cylinder, the SEM-EDS data is 2D-2D and 2D-3D registered. Phases can be segmented based on this chemical information. NanoSIMS provides further chemical information at a higher resolution than SEM-EDS. The results of this combined approach could have a profound impact on understanding the native $H_2$ production in Kansas, USA.

## 4. Acknowledgements

This research was carried under the framework of the E2S UPPA hub Newpores supported by ANR (ANR-16-IDEX-0002). H. Derluyn acknowledges the support of the European Research Council (ERC grant agreement No 850853).

## 5. References

[1] Boone et al, DOI: 10.1130/GES00562.1
[2] Combaudon et al, 2021, AGU fall meeting
[3] Dragonfly 2020.2 [Computer software]. Object Research Systems (ORS) Inc, Montreal, Canada, 2020