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What time-resolved microtomography can do to improve our understanding of fluids in rocks

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Summary: We show how time-resolved in-situ experiments at x-ray imaging facilities can yield new quantitative data for an improved understanding of rock metamorphism, in particular fluid-rock interaction in geological reservoirs.

INTRODUCTION

Experimental studies have contributed tremendously to our understanding of metamorphic processes over the past 50 years or so, and in particular rates and mechanisms of fluid-rock interaction have been investigated in experiments. However, until recently, a direct observation of metamorphism was impossible and experimental outcomes were quantified through indirect measurements or post-mortem analyses. The utilization of x-rays and/or neutrons to image rocks that are engaged in metamorphic reactions in real time enhances our tool box dramatically. In this presentation, I will synthesize the findings from several collaborative experimental studies that documented and quantified fluid-rock interaction in 4-dimensional x-ray microtomographic datasets. The experiments include 1) carbonation of olivine [1], 2) dehydration of gypsum, both poly-crystalline (2a, [2]) and single-crystal (2b) and 3) dissolution and precipitation of salt [3]. I will report on the bespoke x-ray transparent environments we developed and built for these experiments. I will furthermore outline the most important findings to draw a detailed picture of what in-situ microtomography experiments can contribute to the understanding of metamorphic processes.

DATA ACQUISITION AND PROCESSING

To explore fluid-rock interaction, our experiments utilize x-ray transparent experimental vessels, furnaces and presses that are engineered at our School (see also presentation on the triaxial deformation rig *Mjölnir* by Ian Butler). All of them are designed to be portable and they come in modules that can be adapted to specific experiments. For the presented time-resolved in-situ experiments we employed them at the Advanced Photon Source (APS), Argonne National Laboratory (U.S.) (experiments 1,2) and at our in-house laboratory CT scanner (3). At APS, beam line 2BM offers pink and white beam imaging and a flexible imaging setup that can easily be adapted to experimental requirements. A distinct advantage of this beam line is that it enables particularly long experiments of up to about 7 days, which proved critical for [1], where the progress of carbonation was comparatively slow. At the other extreme, the possibility to acquire 3-dimensional data in 1 second or less facilitates, e.g., the imaging of migrating fluid fronts in porous rocks. Combined with our x-ray transparent Hassler-type core holders [4], one of which will be available to general users at beam line 2BM from mid 2017, this enables studies that span a wide range of crustal conditions (currently up to 270° C and 35 MPa confining pressure, with the pore fluid pressure being controlled independently).

Depending on the experiments, we have acquired between 10 and 360 microtomographic datasets during a single experimental run, a raw data volume that often amounts to several TB (35 TB in the case of [1]). We have processed these data on a computational infrastructure that centers around a storage server, which receives incoming data via grid-FTP and distributes them between a large-memory cluster, several workstations and a tape library. Our data are reconstructed with *Tomopy* [5] or *Octopus*© and processed, analyzed and visualized with *Fiji*, *Sci-kit Image* [6] and/or *Avizo*©.

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RESULTS

Our experiments outline the transformative potential that in-situ microtomography has for experimental geosciences:

- The olivine carbonation experiment explored the consequences of a substantial volume increase that results from the transformation of forsterite to magnesite and quartz in the presence of a CO_2 saturated fluid, which was previously speculated to clog existing pore space and shut down the reaction. We could show that the volume increase causes fractures to form in the host rock and thereby exposes unreacted host rock to the fluid. The sequestration of CO_2 via carbonation of olivine can thereby become a self-sustaining process.
- Our experiments that investigated the dehydration of polycrystalline gypsum to hemihydrate targeted a reaction that is associated with a solid volume reduction. Our data revealed that, at reservoir conditions, this reduction was accommodated by the formation of porous moats that wrap around hemihydrate needles. The moats were fluid-filled, and diffusion of dissolved ions across them controlled reaction progress. As the moats got wider, the reaction rate slowed down. Since the moats interconnected early during the experiment, excess pore fluid pressure dissipated rapidly, which has important implications for dehydration-induced seismicity.
- Single gypsum crystal dehydration at atmospheric conditions explored the control of the gypsum lattice on the dewatering process. Our data pointed to a strong control of the crystal surface geometry on the process, giving rise to desiccation cracks that collected and channelized the escaping water. The crystal lattice seems to have had a lesser influence on the migration pathways of water leaving the crystal.
- The compaction of granular *NaCl-biotite* aggregates was found to be accommodated by grain boundary sliding as well as pressure solution/precipitation creep albeit in proportions that changed in favor of solution/precipitation creep during the experiment. Porosity evolved heterogeneously in layered aggregates of *NaCl* and *NaCl+biotite*, with shortening localizing in the latter, which trapped fluid in these layers. It seemed that active dissolution concentrated at the interface between the layers over time. This has important implications for the formation of stylolites, which control fluid flow in many carbonate hydrocarbon reservoirs.

Our experiments gave new insights into reaction mechanism, the spatiotemporal advance of reactions, porosity evolution during fluid-rock interaction and the distribution of strain. The possibility to quantify all of these parameters from time-resolved microtomography data critically advances studies of rock metamorphism and fluid-rock interaction. However, processing and analyzing these data is non-trivial and any quantification should come with the appropriate documentation and error estimates.

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