



## **Formation of secondary porosity in 4D Synchrotron X-ray tomography experiments**

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Synchrotron X-ray tomography at the Advanced Photon Source (APS) allows to investigate secondary porosity in three dimensions on the nano- to microscale. We utilised the key advantage of the technique, the rapid data acquisition time (seconds to about half an hour/data set), to study the formation of porosity in natural rock samples in real time (4D). The spacious instrument setup in the experimental hutch allowed us to install an X-ray transparent furnace to heat millimetre-sized samples up to 230 °C (>400 °C in the next generation) in the X-ray beam. We focused on two porosity-generating mechanisms: thermal expansion cracking in Westerly granite and dehydration (volume loss/hydraulic fracturing) of Volterra gypsum. The spatial resolution was 1.3 micron in both experiments.

We heated a 2mm diameter cylinder of Westerly Granite stepwise from 50 °C to 230 °C and then quenched it to investigate the effects of thermal expansion cracking on the three-dimensional porosity architecture. The sample was scanned after increasing the temperature in 10 °C steps to record the cracks formed during each heating interval. Preliminary analysis of the heterogeneous 4D displacement fields proved that the approach works well. We documented the opening and closing as well as interconnection of grain boundary- and intragranular cracks. A full quantification is currently under way. This experiment also serves to benchmark numerical simulations of thermal cracking that will be used to upscale the permeability evolution during heating (see abstract of Schrank et al.).

A second heating experiment aimed at documenting the fluid escape pathways during the dehydration of gypsum to bassanite. We heated a gypsum sample to 115 °C for increasing periods of time. The reaction progress was directly observed in two-dimensional tomographic projections, 3D tomographic datasets were collected during cooling at 50 °C in between the heating intervals. The experiment demonstrated how a permeable pore network formed and stabilised during fluid escape and volume decrease. Our analytical routines allowed a full quantification of the process.

These are the first steps towards being able to evaluate the distribution and behaviour of all secondary porosity and fluids in rocks at conditions from near-surface to the middle crust in 4D nano- to microscale tomographic experiments. From there it is a small step to manipulate secondary porosity in the lab and provide valuable information for the assessment of longevity of low to high-temperature geothermal reservoirs and the sealing capacity of nuclear waste deposits.