Imaging the textural evolution of gypsum through a metamorphic cycle

Eilidh Vass Payne [1], Ian Butler [1], James Gilgannon [2], Damien Freitas [3], Andrew King [4], Roberto Emanuele Rizzo [5], Lisa Eberhard [5], Florian Fusseis [6] | [1] University of Edinburgh, [2] University of Glasgow, [3] University of Manchester, [4] PSICHÉ beamline (synchrotron SOLEIL) [5] Utrecht University , [6] RWTH Aachen | The following work is supported by: NERC grant NE/T001615/1 to FF & IB; NERC DTP grant NE/S007407/1 to EVP; D.F is supported by an AFOSR/EOARD grant (FA8655-21-1-7005) and beamline access was provided by synchrotron SOLEIL.

Introduction

Water facilitates deformation processes throughout the Earth's crust (Rubie, 1986). Knowledge of how fluid-rock interactions proceed at the grain-scale informs our understanding of larger scale processes.

The calcium sulfate system is used as an accessible analogue to study mineral transformations that occur at elevated temperature and pressure (e.g. mineral transformations in serpentinite, (Heard and Rubey, 1966)). Here we use the calcium sulfate system to perform pressure and temperature controlled cyclical dehydration-rehydration (D-R) experiments using whole rock gypsum.

Our experiments were conducted on the PSICHÉ beamline of the SOLEIL synchrotron using 4D synchrotron computed microtomography (4D µCT). The technique enables direct observation of mineral reactions and textural evolution while the sample is at reaction pressure and temperature.

$$CaSO_4 \cdot 2H_2O \leftrightharpoons CaSO_4 \cdot \tfrac{1}{2}H_2O + 1\tfrac{1}{2}H_2O$$
$$CaSO_4 \cdot \tfrac{1}{2}H_2O \rightleftharpoons CaSO_4 + \tfrac{1}{2}H_2O$$

Equation 1: The calcium sulfate system. Reversible dehydration and rehydration reactions from gypsum to anhydrite via the hemi-hydrate bassanite.

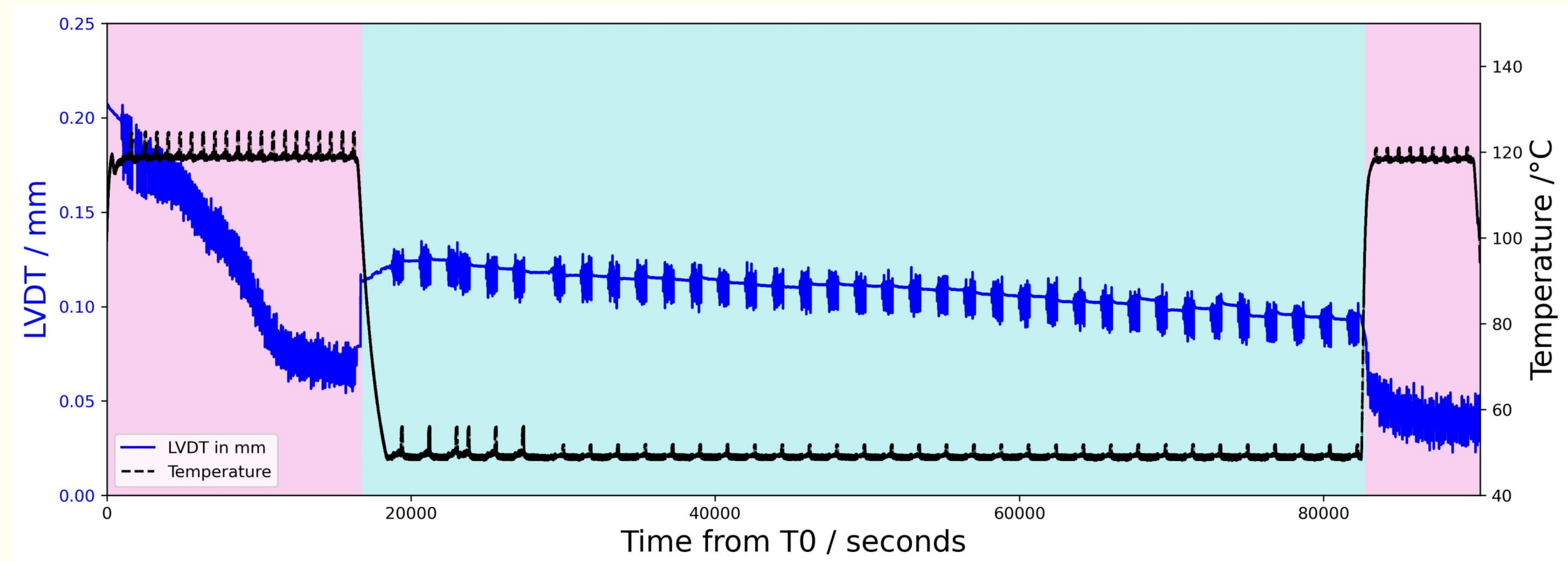

Figure 1: The experimental temperature profile (black line) for dehydration (pink) and rehydration cycles (blue), and the sample height change (blue line) measured by an LVDT. There is sample shortening clearly evident during dehydration and a subtle height decrease during rehydration. Dehydration was carried out at 125°C, while rehydration was allowed to proceed at 50°C.

Research Questions

✧ How do mineral textures evolve during a D-R metamorphic cycle?

✧ Are neoformed mineral textures inherited from the pre-existing phase?

✧ What does gypsum look like as a product phase of re-hydration?

✧ The D-R reactions involve significant volumetric changes. By quantifying the total and solid volume change can we understand the consequences of connected porosity and see any evidence of shrinkage or swelling in the sample?

Dehydration at 125° C

Rehydration at 50° C

REACTION

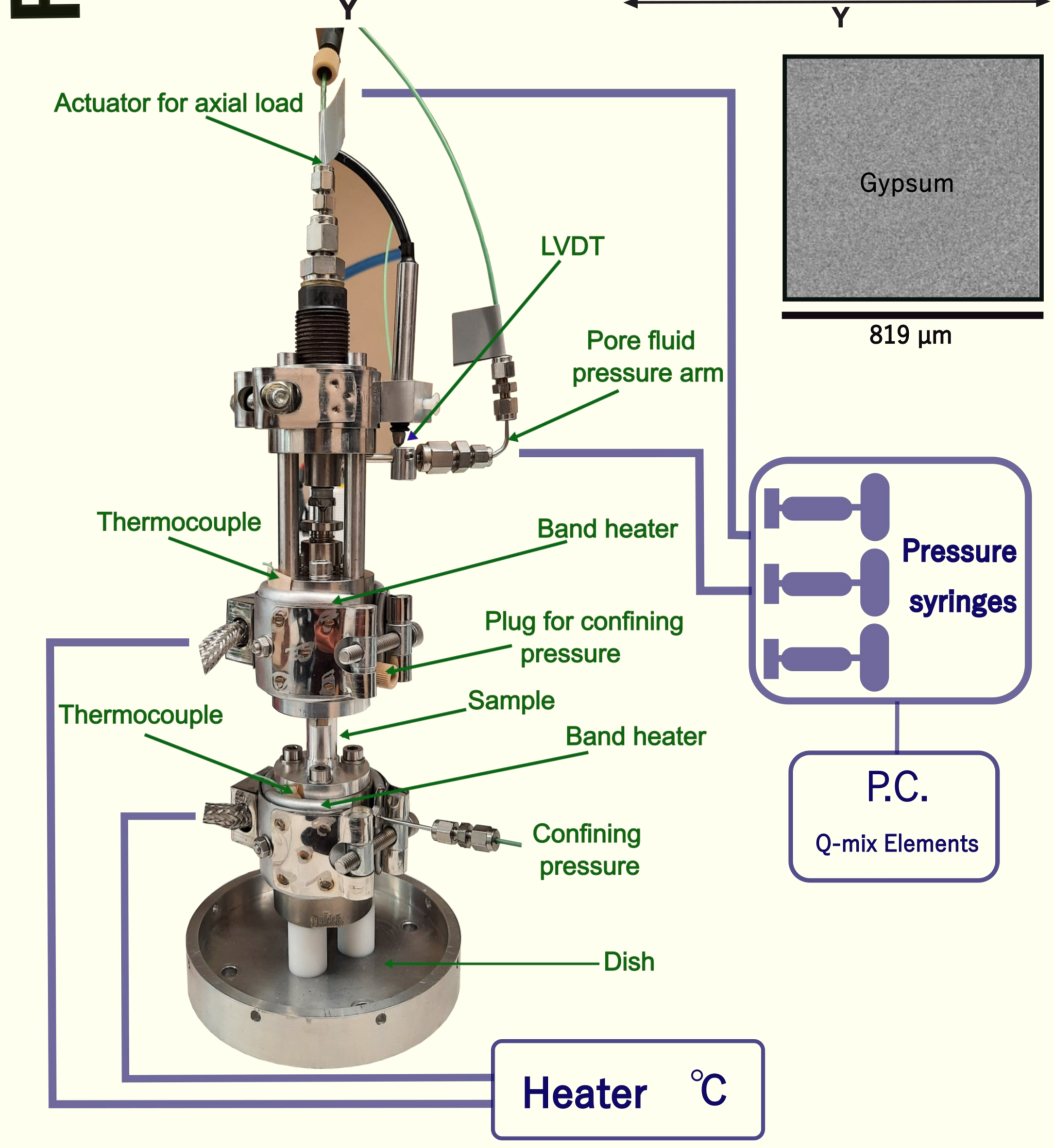

Figure 2: Mjölnir rig, miniature x-ray translucent triaxial rig used for these experiments. It uses 3.2 mm * 10 mm cylindrical samples and controls the confining pressure, axial load and pore fluid pressure independently using hydraulic fluids. This rig features a heating system via band heaters and can measure the sample temperature via thermocouples.

Experimental procedure

✧ Whole rock samples of gypsum (Volterra Alabaster, VA) were cored to cylinders of 3.2 mm diameter and 10 mm length.

✧ The VA cores were loaded in the heated triaxial deformation cell Mjölnir (Butler et al., 2020) which was mounted on the beamline (filtered white beam with peak energy at 65kV) and connected to high-pressure syringes, heater controller and PC control (figure 2). The experimental temperature profile is shown in figure 1.

✧ Hydrostatic load was maintained throughout (20 MPa) with an applied pore fluid pressure of 5 MPa.

POROSITY

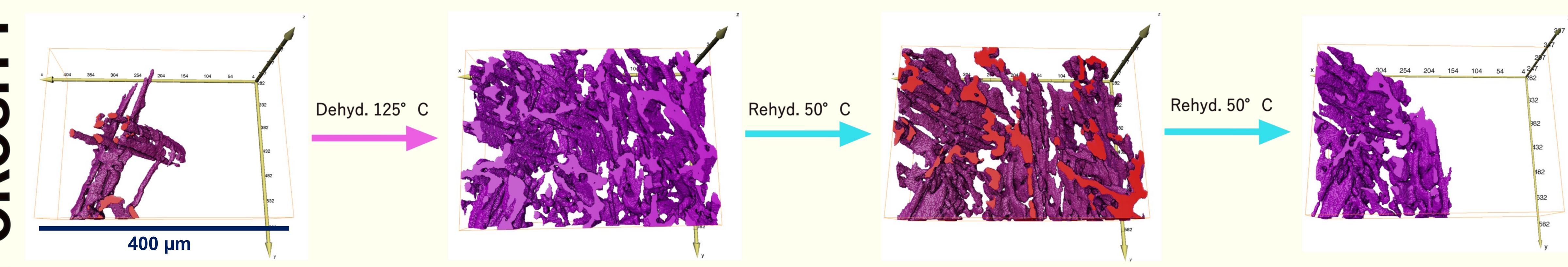

Figure 3: Visualization of the evolving connected pore space in an equivalent sub-volume ($14x10^6 \mu m^3$) during dehydration (scans 170 and 200) and rehydration (scans 320 and 350). The volume corresponds to the blue box shown in the tomographic slices above. Pore space is generated around growing acicular bassanite. Initially patchy porosity becomes a pervasive pore network during dehydration. Rehydration shows similarly patchy and heterogeneous infilling of the pore network.

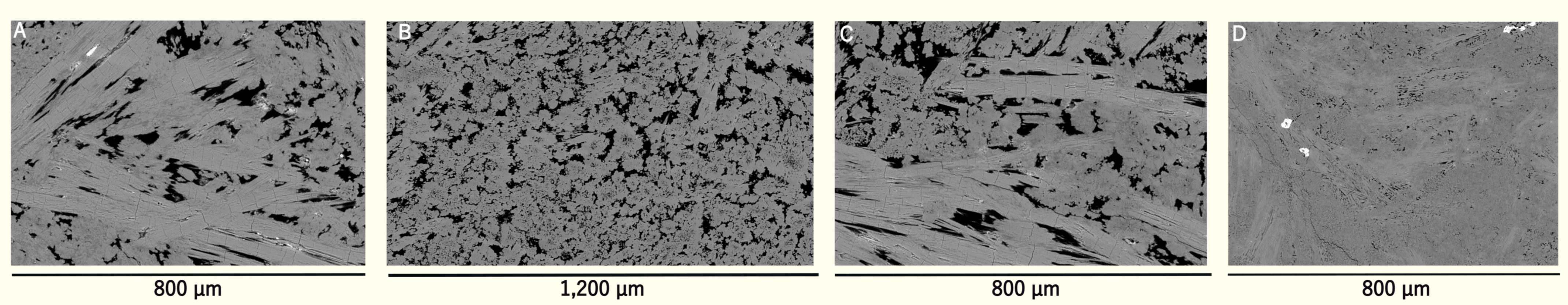

Figure 4: Scanning Electron Microscope images of textures and textural relationships from a sample that has undergone a dehydration and rehydration cycle. (A) shows cross-cutting acicular bassanite with porosity between grains. (B) shows bassanite florets which are sections across the acicular crystals. Grains are skeletal with porous cores and are surrounded by moats of porosity. (C) elongate and floret shaped sections of acicular bassanite with gypsum from rehydration filling both grain cores and moat-like porosity. (D) Gypsum infilling leaving only microporosity.

Key Findings

✧ These data are the first time resolved tomographic investigation of textural evolution during a mineral D-R cycle.

✧ The nucleation, growth and spatial location of both the bassanite phase during dehydration and gypsum phase during rehydration appears random with no evidence for preferred grain orientation.

✧ Mineral transformation and porosity infilling during rehydration is patchy and localised within the experimental sample.

✧ Gypsum formed during rehydration is distributed across bassanite grain boundaries, fills skeletal pore space and inter-grain pore space.

✧ Gypsum grains produced by rehydration appear small, <1.3 micron with nucleation and early growth not discernible in our data.

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