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Pressure Solution Studied In Situ via X-ray Reflectivity

Roy A. Wogelius (roy.wogelius@man.ac.uk)[1], **Dag K. Dysthe** (d.k.dysthe@fys.uio.no)[2], **Chiu C. Tang** (c.c.tang@dl.ac.uk)[3] & **A. A. Nield** (a.a.nield@dl.ac.uk)[3]

[1] Department of Earth Sciences, University of Manchester, Manchester M13 9PL, UK
[2] Departments of Physics and Geology, University of Oslo, P.O. Box 1048, Blindern, Norway
[3] CLRC Daresbury Laboratory, Daresbury, Warrington, Cheshire, WA4 4AD, UK

The detailed physics that describe the process of pressure solution are still not well understood. Pressure solution occurs when two solid surfaces are in contact with each other under pressure with a fluid phase filling in the void space between the surfaces. Redistribution of material along the solid interface may occur even if the fluid is saturated with the solid in question because the local solubility is enhanced by placing the surface under pressure. Bulk experiments have concentrated on studying grain size changes and similar properties of packed columns of fine-grained material. Direct observation of surfaces during reaction has been difficult, although some studies have attempted to quantify surface evolution using visible light imaging or other methods. *Ex situ* observation and linear stability analysis suggest a suboptical dynamic structure of the interface.

We have developed a new approach which takes advantage of the fact that X-ray scattering is sensitive to changes in surface topography at the angstrom-scale and can be used *in situ* to probe buried interfaces. Application of X-ray scattering to the study of pressure solution can thus give critical new insights into how surfaces evolve under pressure. Experiments designed to study pressure solution via X-ray scattering were completed at Daresbury Laboratory with the aid of a purpose built pressure cell. The cell used a chemo-mechanically polished perspex anvil as the surface which applied pressure to a mineral substrate. Perspex is reasonably X-ray transparent, and this thereby allowed us to use the anvil both as pressure medium and as the medium of transmission for both the incident and scattered beam.

Experiments were completed with polished single crystals of halite (NaCl) and calcite ($CaCO_3$). Surfaces were characterised both dry as free surfaces and dry under confining pressure. After characterisation a fluid which was saturated with the phase of interest was placed on the surface of the mineral and then the pressure was brought up to the desired value by the controlled compression of a calibrated spring in contact with the reverse side of the anvil. Measurements of the reflected beam intensity from the interfacial region were then completed. Clear, well-developed oscillations in the reflected beam intensity were observed in the confined systems and the shapes of these oscillations were modified by the introduction of fluid. These oscillations, often termed Kiessig fringes, result from interference in the reflected signal due to the multi-layer nature of the interface. Overall decrease in the intensity of the reflected beam as a function of incident angle corresponds to the combined roughness of the two surfaces. Interference fringe evolution can be seen distinctly in both the halite and calcite experiments. Fresnel theory and a roughness model (Cowley and Ryan, 1987) were used to construct a mathematical model of the scattered data and thus constrain a physical model of the surface. Preliminary calculations suggest that the surface roughness of both halite and calcite decreases under pressure with fluid present.

Cowley R.A & Ryan TW, *J. Phys. D*, **20**, 61, (1987).