In situ experiments in the SEM

Dan Tatham & Dave Prior
School of Earth and Ocean Sciences
University of Liverpool, U.K.

UNIVERSITY OF LIVERPOOL

Introduction: The EuroMinScI Initiative

The development of sub-structure is an important process in rocks deforming at elevated temperature by crystal-plastic processes. Sub-structure is a useful indicator of deformation mechanisms and palaeostress in the lithosphere. Moreover, it provides an important control on the mechanical properties of materials.

A comprehensive understanding of the nature and mechanical significance of sub-structures is key, therefore, in increasing the accuracy and reliability of palaeostress estimates and to properly describe the rheological properties of rock aggregates deforming by dislocation processes. Such an understanding will prove central in the advancement and refinement of manifold models of multi-scale dynamic processes through the material and Earth sciences, from the atomic-scale to orogenesis and mantle convection.

This study is part of a larger collaborative research project into sub-grain structure development in rocks and metals, funded by national agencies as part of the EuroMinScI initiative.

Aims of the collaboration include gaining an increased understanding in:

1. the nucleation of new (sub-) grains during deformation;
2. the behaviour of sub-grain boundaries during deformation and phase transitions;
3. the interaction of processes across all scales, form dislocations to whole grains and aggregates; and
4. the numerical prediction of sub-structure,

with an emphasis on integrating *in situ* experimental techniques with multi-scale numerical simulations.

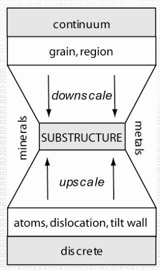


Figure 1: Research model for the EuroMinScI project. Substructure processes are an essential link between processes active at the atom-, dislocation- and grain-scale. Moreover, numerical sub-structure dynamic modelling is at the crossover between continuum and discrete modelling.

In Situ Experiments in the SEM

In situ high-temperature electron backscatter diffraction (EBSD) and SEM imaging experiments on metals and selected rock-forming minerals have proved successful in the observation and quantification of recrystallisation and phase transformations up to ~1000°C (Figures 2 & 3; e.g. Seward et al., 2002; Seward et al., 2004; Bestmann et al., 2005), and the incorporation of the resulting microstructural data into numerical models (e.g. Piazolo et al., 2004).

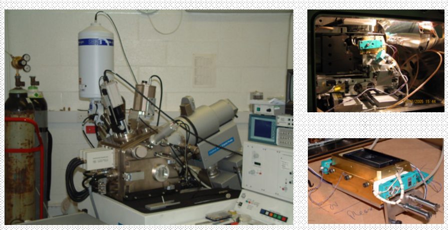


Figure 2: The CamScan X500 SEM. The tilted-column design permits *in situ* experimentation on a horizontal sample stage whilst satisfying the geometrical requirement of EBSD for a high angle between the incident electron beam and the specimen surface (Seward et al., 2002). Inserts: static heating stage in (top) and out (bottom) of the SEM.

The range of samples that can be analysed is in part limited by their material properties and the operating conditions of the SEM. That is, an interplay exist between (1) the attainment of temperatures suitable for studying crystal-plastic processes on experimental timescales, (2) preserving the integrity of lattice structure at said temperatures such that EBSD analysis is productive and pattern quality is maintained, and (3) avoiding sample deterioration (e.g. calcite, upon heating) or significantly reduced grain boundary mobility (e.g. quartz) at the reduced pressure conditions in an SEM.

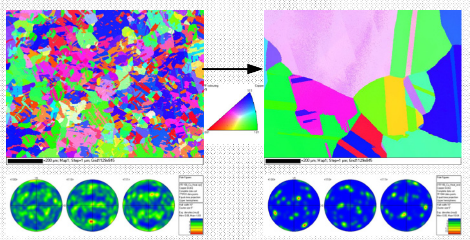
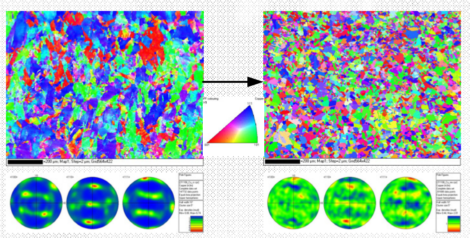


Figure 3: Examples of *in situ* static recrystallisation experiments in copper.

(a) EBSD orientation maps, in inverse pole figure colours, and pole figures for grain growth of an initially annealed copper, at ~850°C. The finial grain growth pole figures show the influence of a few large grains.



(b) EBSD orientation maps, in inverse pole figure colours, and pole figures for the annealing of cold-rolled copper at ~350°C. Rolling direction in starting material is N-S. Randomisation of the initial cold-rolled fabric can clearly be seen.

Heating & Deformation Experiments

At Liverpool, we document the kinematics of intracrystalline sub-structure development and the way such sub-structures interact with intergranular boundaries during high-temperature deformation experiments of MgO and forsterite.

Deformation of single crystal, layer-cake and polycrystalline samples at a range of temperatures between 1100°C and 1400°C will provide information on sub-structure development and the interaction of sub-structure with boundaries that are relatively easy to relate to 2D models, and those representative of real rock microstructures, respectively.

A recently commissioned, custom-designed sample stage for the CamScan X500 FEG-SEM at Liverpool incorporates a high-temperature heating system with a deformation rig, permitting simultaneous heating and tensile deformation of samples with real-time EBSD analysis and conventional SEM imaging.

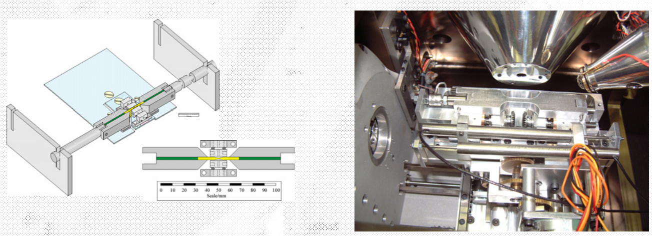


Figure 4: (Left) Schematic illustration of the heating/deformation stage. Necked, tape-like samples are heated from bordering assemblies, and the extensional load is applied in series along the sample length. Deformation in the necked region remains in the field of view and focus of the electron beam. (Right) The heating/deformation stage *in situ* in the SEM. The pole piece of the inclined electron column can be seen at the top of the picture.

MgO and forsterite present excellent materials for low-pressure experiments, and the new stage permits the attainment of temperatures (up to 1400°C) needed to conduct experiments on these materials. Being comparatively easy and inexpensive to fabricate, MgO represents a particularly suitable material from which to gain analogue information and being of simple structure and chemistry, will be the easier phase to couple with numerical models. Forsterite is clearly the more relevant Earth material, however, and will be used subsequent to the establishment of sound experimental protocols with the new equipment.

References

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EUROCORES Programme
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EuroMinScI
European Mineral Sciences Initiative

SubStructure Dynamics

This presentation has been made possible thanks to the support from the European Science Foundation (**ESF**, www.esf.org) under the EUROCORES Programme **European Mineral Sciences Initiative** (**EuroMinScI**, www.esf.org/eurominsci), through contract No. ERAS-CT-2003-980409 of the European Commission, DG Research, FP6.