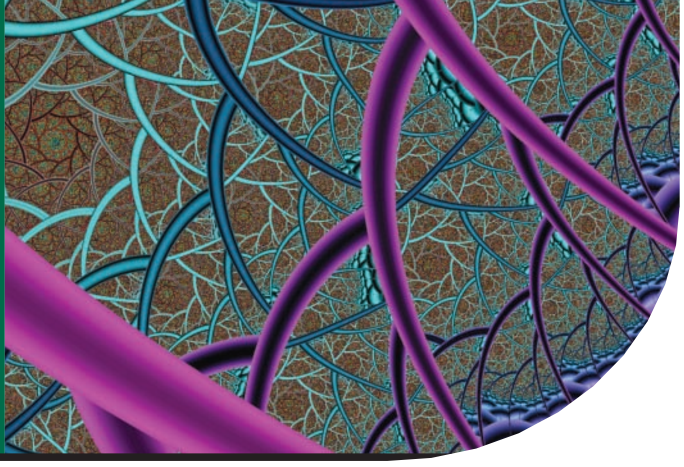

A Level Playing Field for EBSD Analysis of Crystalline Materials

Electron BackScatter Diffraction (EBSD) is a rapidly growing addition to the battery of techniques routinely employed by scanning electron microscope users, producing an electron backscattering pattern (EBSP) that relates exactly to the crystal structure and orientation of the sample under examination.

Although the first EBSP was published by Nishikawa and Kikuchi in 1928, many would correlate its growing popularity with the rise of the scanning electron microscope (SEM) since its commercial introduction in 1965.

By 1967, Coates was describing observation of Pseudo Kikuchi lines in work that led directly to selected area electron channelling patterns (SAECP). In this technique, the electron beam is rocked from side-to-side over a point on a horizontal sample surface and a map of the intensity of backscattered electrons generated. Although the angular resolution of SACP is superior to EBSD, two main drawbacks exist: a fundamentally low resolution of 5 µm and a complex electron optical configuration.

> **“When studying the structure of geological specimens, scientists are confronted with the result of micro-scale chemical and physical processes...”**

Author Details:

Dave Prior[1], John Gilbert[2] and Dick Paden[2]

Email:
davep@liverpool.ac.uk
John.Gilbert@camscan.com
dick.paden@camscan.com

Venables and Harland first described electron backscatter diffraction in the SEM in 1973 using a stationary electron beam focused on the surface of a specimen tilted to make a high incident angle, typically 160°. When, in the early 1980's, Dingley introduced an external camera viewing a scintillator screen through a lead glass window rather than using photographic film within the vacuum chamber, a major constraint on throughput was eliminated. Computer assisted indexing of EBSD patterns followed in the early 1990's and developed rapidly. Parallel advances in camera technology, computing power and high-speed storage capacity have facilitated the evolution of modern automated data collection; in a little over a decade, the EBSD user has gone from being able to capture, perhaps, 200-300 data points per day to 500-600 data points per second.

Because of these remarkable strides to reduce operational complexity and speed up data collection, EBSD mapping at the micron and sub-micron scale is now routinely used to characterise crystalline, metal and ceramic materials; including texture, individual grain size, grain shape, grain orientation, grain boundary character, strain state, crystallography-based phase identification, area percentages of multiple phases in bulk samples, crystallography of facets and failure initiation sites, and other materials characteristics.

However, the essential mechanical layout of the SEM has not changed during this period and is challenged when faced with certain experimental protocols, such as in-situ heating experiments. One answer is to optimise the design of the SEM.

IN-SITU HEATING EXPERIMENTS

We are all familiar with the phase transformation undergone by water when it cools to ice or is heated to vapour. Metals, ceramics and minerals also undergo phase transformations when they are heated and cooled. For instance, heat pure titanium and at 883 °C its crystal structure changes from hexagonal to cubic – a process that reverses if it is cooled. It follows that an appreciation of the physics of these changes is essential if we are to understand the high temperature properties, mechanical behaviour and strength of materials used in engineering.

Previous studies on annealing and recrystallisation, such as Humphreys and Ferry (1996) and Springer and Radomski (1998) achieved results at temperatures up to approx. 400 °C. But, to go beyond this, to 1,000 °C and above, a radical design departure is required, which has been achieved in the CamScan X500 Crystal Probe field-emission gun (FEG) SEM. Putting manipulation of the sample and ideal positioning of the heating and straining stage attachments at the core of the design, the X500 maintains the essential geometry of a high angle of incidence between the electron beam and the specimen by inclining the column and electron-optic axis at 70° to the vertical, allowing the stage to be mounted horizontally (*Figure 1*).

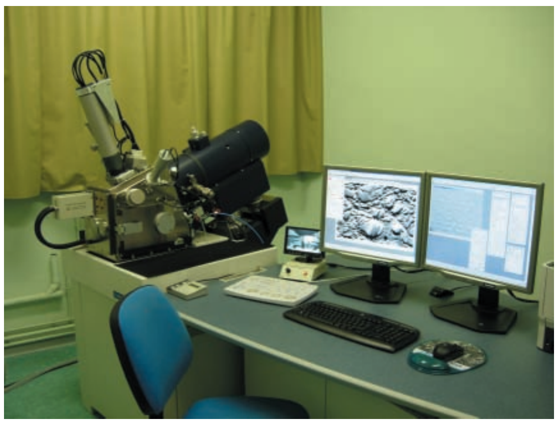

Figure 1. The CamScan X500 Crystal Probe field-emission gun (FEG) SEM.

Three key advantages arise from this revolutionary SEM configuration:

1. Accurate and rapid specimen movement - the horizontal stage eliminates the need for vertical movement of the sample during analysis and simplifies the construction of heating and straining stage attachments. This is significant when the weight of these stage attachments is considered; reducing stress on the driving mechanism of the sub stage, which only has to move in the X and Y planes, and benefiting accurate specimen movement at high speed. Accurate, rapid stage positioning during automated EBSD contributes to decreased data acquisition time.
2. Analysis up to melting point and beyond – because the sample is not tilted, there is no danger of the specimen moving or flowing away.
3. Separation and protection of the detector domain – the X500 Crystal Probe naturally divides into a lower domain for the sample and stage and an upper domain for the detectors. This allows heat shielding to be introduced to protect and extend the life of the detectors, especially the forescatter array, during in-situ heating experiments. A circulating cooled water system also protects the detector mountings, heat shields and body of the heating stage from the effects of the furnace and allows experiments lasting tens of hours to be conducted.

Our team at Liverpool University's Department of Earth Sciences has been working with the CamScan X500 to probe the fundamentals of phase transformations, grain growth and recrystallisation in metals and rocks (*Figure 2*).

WARM FOR ROCKS, HOT FOR METAL

When studying the structure of geological specimens, scientists are confronted with the result of micro-scale chemical and physical processes that only occur under high

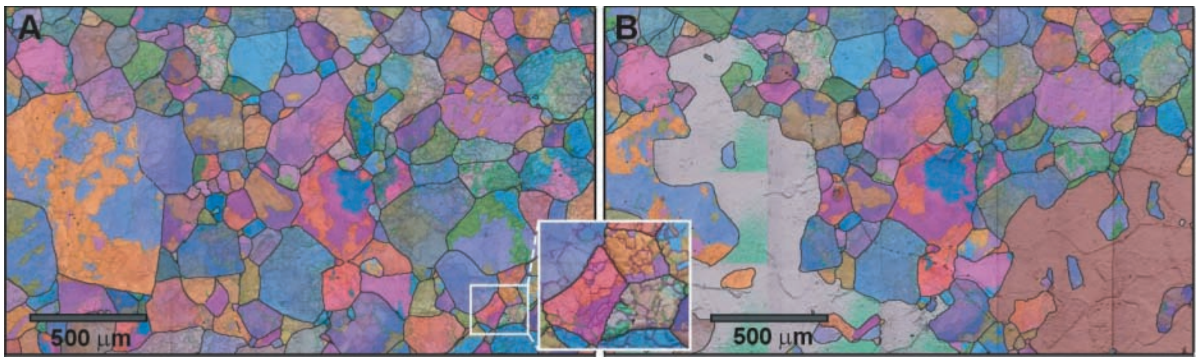

Figure 2. Annealing of deformed NaCl. EBSD map before heating (A) and after 2.5 hours at 350-410 °C.

pressure deep within the earth's crust and may have occurred over a timeframe measured in millions of years. Obviously, reproducing these conditions in the laboratory is problematical. What can be done, though, is to observe microstructural changes as they occur at laboratory scale and use this information to decode the process by which different rocks and rock formations evolved.

One of the driving forces behind our experiments is to identify the processes that naturally deformed a rock sample simply by matching its characteristics against those obtained in the laboratory and so shed light on the forces driving both plate tectonics and earthquakes. Another is to put our quantitative data about parameters such as grain size, grain shape and grain boundary geometry into numerical simulations so that deductions about the underlying principles of the active processes can be tested. If we can accurately predict microstructural behaviour, then it can be applied to materials that are not suitable for physical experiments.

But, simulating nature means having to capture information about the rapid changes that take place in the microstructure at temperatures that are high enough to make them occur. As most natural rock samples need temperatures in excess of 1,000 °C, we have focused much of our attention on synthetic rock salt. This is not without its direct application, as a number of companies throughout the world are looking at the possibility of burying nuclear waste in rock salt-based repositories and only by understanding its behaviour will it become apparent whether these areas will make good disposal sites or not. However, synthetic rock salt's greatest virtue is acting as an analogue for other minerals (*Figure 3*).

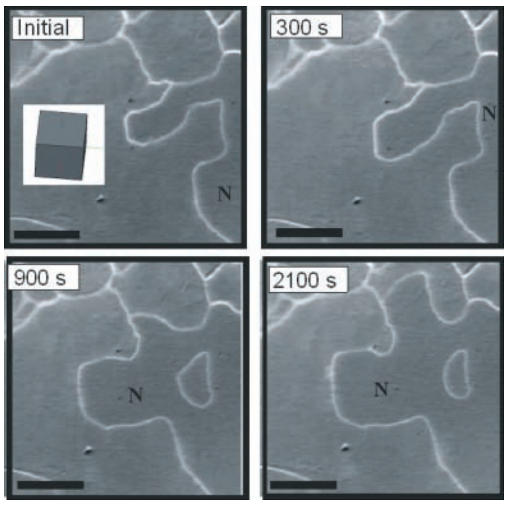

Figure 3. Character of moving boundaries shown as series of SE images; insets indicate time intervals in seconds, new grains are marked with 'N'. Material held at 403 °C for 40 minutes; scale bar=200 µm; inset depicts the 3D representation of the crystallographic orientation of a new grain.

The research group is also experimenting with another rock proxy – metals – with results that benefit materials scientists as well as geologists. Metals exhibit many of the features of rocks, such as phase transformations and recrystallisation, but at a much lower temperature. Therefore, if we can understand these processes in metal it provides insight into geological processes.

One such study has concerned phase transformations in titanium. Pure titanium melts at about 1600 °C. But, at 883 °C, its crystal structure changes from a hexagon (alpha phase) to a cube (beta phase); cool it and it reverts back to hexagons. Theorists thought this change would be predictable and a useful analogue of how similar transformations may occur deep in the earth. However, efforts to demonstrate this using synchrotron radiation and X-ray diffraction failed. Using the X500 and techniques which we developed, we can demonstrate what really happens when pure titanium is heated above 883 °C and then cooled. When the cubes 'morph' back into hexagons, their orientation relative to each other is predictable. However, in the initial transformation when the hexagons 'morph' into cubes, the team has discovered a random element, where the beta phase nucleates and grows within the alpha grains and at the alpha/alpha grain boundaries. The team uses EBSD to establish the presence of the beta phase and *Figures 4* and *5* show surface relief features.

FOCUS ON STEEL

Grain growth is one of the simplest processes of microstructural modification in both rocks and metals. For instance, heat fine grained steel for approximately one hour and the grains become coarser and fewer in numbers. This growth appears to involve existing grains moving their boundaries but, why some and not others? Just as important, when steel is deliberately deformed in a rolling mill, new grains appear to replace deformed ones in a 'recrystallisation' phenomenon.

Given that the kinetics and effect on crystallographic texture of recrystallisation and grain growth are key issues for the successful processing of steel with properties optimised to meet specific applications requirements and the need for predictability is evident. Using our high temperature capabilities we are quantifying migration velocities during recrystallisation and grain growth and exploring possible crystallographic controls in experiments supported by the steelmaker, Corus.

FUTURE DEVELOPMENTS

We are now working with CamScan on a new deformation stage designed to produce in situ heating in excess of 1300 °C. This will be sufficient to enable more advanced studies on natural rocks and geological material to be performed. These temperatures will also be of interest to ceramicists.

Meanwhile, the advanced EBSD capabilities of the CamScan X500 should be of interest in the emerging field of biominerals. Natural biocrystalline structures, such as shells or pearls, are often highly anisotropic with distinct microstructures. Moreover, their production on the nanoscale by organisms surpasses our attempts to synthesise equivalents. Organisms tend to produce tougher materials, at low temperatures, in aqueous environments, with consistency and uniformity, using macromolecule assemblers. Our approach is energy rich, requiring high temperature, high pressure, a radical pH or a mixture of all three, tends to produce toxic by-products, and is irreproducible because of the difficulties in controlling agglomeration. The study of these biominerals is a new area of EBSD application that spans the boundaries of the geosciences and the life sciences and has great potential in engineering.

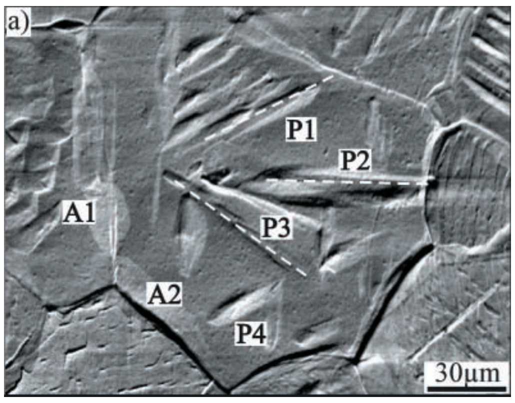

Figure 4. A snapshot of the transient microstructure at approx. 882 °C where beta crystals are forming within an alpha grain of the pure titanium slab. An SEI image with intragranular plates and grain boundary allotriomorphs indicated P and A, respectively.

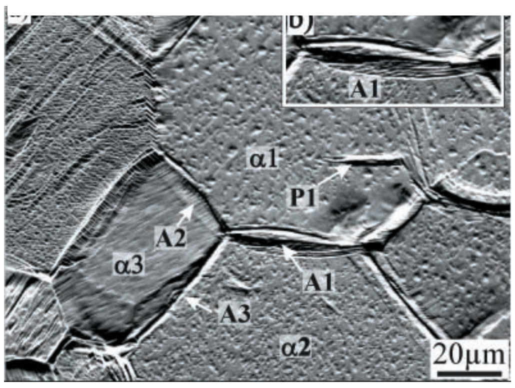

Figure 5. A BEI image with grain boundary allotriomorphs and intragranular beta crystals indicated A and P, respectively; (b) an enlarged section of the BEI to show the difference in thermal etching between the beta and alpha phases.

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Authors

1. Department of Earth Sciences, University of Liverpool, Liverpool L69 3GP, UK
2. Obducat CamScan Ltd, CamScan House, Pembroke Avenue, Waterbeach, Cambridgeshire CB5 9PY UK

Understanding the Causes of Disease

FEI Company announced that the University of California at Los Angeles (UCLA) has installed a multi-million dollar Titan Krios™ transmission electron microscope (TEM) from FEI. In an effort to understand the causes of disease, UCLA's Dr Hong Zhou, Director of the newly established Electron Imaging Centre for NanoMachines (EICN), part of the California NanoSystems Institute, has initiated high-resolution molecular imaging studies using the new Titan Krios TEM.

The Titan Krios is specifically designed for 3D molecular imaging applications where samples are imaged at cryogenic temperatures, which preserves the biological samples in their native hydrated state. The microscope's ability to generate images used in the creation of 3D molecular structures with resolutions as small as a few tenths of a nanometer allows scientists to investigate the structure and function of biological nanomachines at the molecular scale.

Matthew Harris, FEI's Vice President and General Manager of the Life Sciences Division, said: "Dr Zhou and his colleagues recently achieved breakthrough results in 3D molecular reconstruction with resolution better than four Ångströms using an FEI Tecnai Polara™ TEM. We are confident that Dr Zhou will continue to push the boundaries of molecular imaging, and we look forward to supporting him with many groundbreaking discoveries using the Titan Krios."

Dr Zhou added: "The advanced optics, automation and cryo capabilities of the Titan Krios are absolutely essential for our research in nanobiology and nanomedicine. Developments in these areas will expand opportunities to contribute to major advances in rational drug design and targeted delivery, and ultimately advance us towards biology-inspired nanomachines."

Recent advances have made cryo-electron microscopy (cryo-EM) an important imaging tool for major applications in both medicine and nanobiological research. Researchers can use cryo-EM to visualise a broad range of assemblies or nanometer-scale structures at near-atomic resolution and in three dimensions. This imaging method covers a scale range from tens of micrometers to Ångströms and provides valuable structural information for numerous scientific disciplines including structural biology, cell biology, medical and pharmaceutical science.

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