Large Area, High-Resolution EBSD Mapping Of Complex Geological Samples





Seeing beyond

Authors: Dr. David Wallis Dept of Earth Sciences, University of Cambridge, UK Dr. Rich Taylor Carl Zeiss Ltd., Cambourne, UK

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Introduction

Mapping the spatial distributions of crystalline phases and their lattice orientations using electron backscatter diffraction (EBSD) is a central task in both the materials and earth sciences. In both fields, these microstructural parameters and a multitude of derivatives are used to investigate the formation and deformation of man-made and natural materials. One example from the earth sciences involves mapping the microstructures of rocks deformed in Earth's mantle, the layer beneath the crust, to analyze the strength of tectonic plates and the mechanisms by which they deform. This task is challenging because rocks formed at high temperatures are coarse grained, with individual crystals up to a centimeter in size, but also contain lattice distortion over micrometer length scales within grain interiors. As such, the microstructures must be mapped with a combination of both large areal coverage and high spatial resolution that has historically required prohibitively long acquisition times.

EBSD Analysis

However, recent advances in microscope and detector technology have made this task significantly easier and more efficient. Here we take advantage of the state-of-the-art optics and beam technology combined with the latest generation stage technology of ZEISS GeminiSEM for high precision imaging and analysis. Meanwhile, recent developments in EBSD detector technology, including the replacement of CCD cards with CMOS chips and fiber-optic signal pathways in Oxford Instrument's Symmetry detector allow diffraction data to be collected orders of magnitude faster than just a few years ago.

EBSD Mapping Data

Energy	20 kV
Magnification	200×
Area	33 × 19 mm
Hit rate/pattern matching	>95 %
Step size	6 µm
Phases	5



Figure 1 Large-area EBSD maps of a rock from Earth's mantle brought to the surface by a volcano in the Eifel region of Germany. Phase map of the distribution of different minerals.

Speed	~700 pps
Beam current	30 nA

Figure 1 presents an EBSD map consisting of over 17 million pixels, covering several square centimeters at a step size of 6 μ m, collected in only 12 hours. The three plots take the orientation data obtained from the EBSD analysis to increasing levels of detail. Figure 1a shows the distribution of different minerals within the rock determined from lattice parameters, which can be used to investigate the history of metamorphism and melting.

Figures 2 and 3 reveal distortion at both the sample scale and individual crystal lattice scale of the mineral olivine, demonstrating that the rock has been highly sheared as the European tectonic plate has been deformed during past mountain-building events. The details of this lattice distortion



Figure 2 Map of lattice orientation of olivine grains colored by the rotation axis between each pixel and the sample Z-axis orientation. Black lines indicate grain boundaries.



Figure 3 Map of lattice misorientation within olivine grains colored by the rotation axis between each pixel and the average orientation of each individual grain.

can be used to investigate the mechanisms and conditions of deformation and to quantify the stress exerted by continental collision.

Figure 2 is colored by the crystal direction aligned with the Z axis of the specimen (in and out of the page) as indicated on the inverse pole figure. The predominance of red-purple grains indicates that the sample has a crystal preferred orientation resulting from dislocation creep. As the average grain size of olivine is approximately 0.5 mm, large-area mapping is required to measure a representatively large sample of grains.

Figure 3 takes the dataset to greater detail, this time plotting the lattice misorientation compared to the average for each individual olivine grain. This instantly demonstrates the highly deformed nature of the rock as highlighted by the mineral olivine, with different mechanisms of deformation occurring within the olivine crystal lattice itself not seen in the previous figure.

Summary

High-resolution EBSD datasets are unlocking numerous new fields of research across the Earth Sciences, ranging from the deformation of Earth's interior to the flow of ice in polar glaciers, to the growth of biominerals.

With the expansion of these lines of investigation, ongoing developments in microscope and detector technology will play an important role in addressing present and future Earth-Science challenges related to geohazards, climate change, and natural resources present and future earth-science challenges related to geohazards, climate change, and natural resources.

Carl Zeiss Microscopy GmbH 07745 Jena, Germany microscopy@zeiss.com www.zeiss.com/microscopy