Fast Characterisation of Deformed Quartz Rocks

Application Brief

Introduction

Although conventional CCD-based EBSD detectors have been able to analyse metals and alloys with speeds in excess of 1000 indexed patterns per second (pps), the weaker signal and lower symmetries of mineral phases have usually limited data collection from geological samples to significantly slower speeds, often below 100 pps. The release of the groundbreaking Symmetry CMOS-based EBSD detector, together with the powerful AZtec® software, has resulted in a significant improvement in acquisition speeds. Although metal samples can now be routinely measured at speeds in excess of 3000 pps using Symmetry, the fact that extreme pixel-binning of the diffraction patterns is not necessary for high collection speeds means that similarly impressive results are also achievable on rock samples without compromising data quality.

In this application brief we show how Symmetry can be used to characterise the microstructure in a fine-grained, deformed quartz rock at speeds approaching 1000 pps.

Experimental Method and Results

A polished thin section was prepared from a mylonitic quartz vein from Lewisian rocks in NW Scotland. The vein has been severely deformed, with significant dynamic recrystallisation causing grain size refinement down to 10-20 µm. The sample was mechanically polished down to a final stage using colloidal silica, and was then coated with ~5 nm carbon prior to analysis. Two areas of the sample were analysed on a field emission gun SEM, with EBSD data collected using the AZtec software and the Symmetry detector, acquiring patterns with a 312 x 256 pixel resolution.

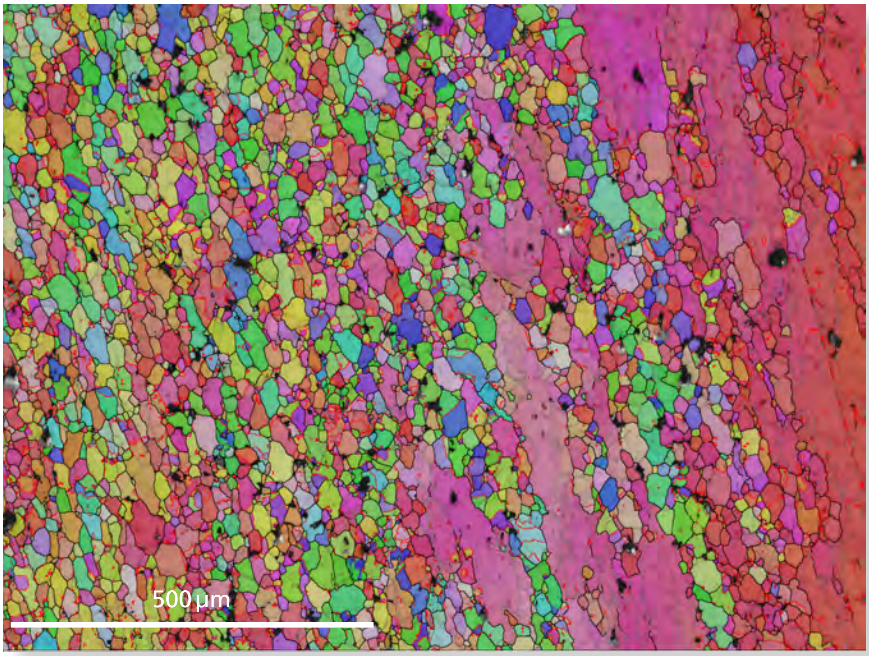


Fig. 1a. Orientation map of the first area (inverse pole figure colouring scheme). Grain boundaries are marked in black, low angle boundaries in grey and Dauphiné twin boundaries in red. Black spots are voids in the sample.

The first single area analysis covered an area of ~1.2 x 0.9 mm, using a measurement step size of 0.75 µm. The total of ~1.9 million analysis points was completed in 32 minutes at almost 1000 pps, with a 96% indexing success rate. Numerous pores in the sample (visible as black regions in the orientation map) contributed to the non-indexed fraction.

The orientation map (Fig. 1a) shows clearly the dynamically recrystallised grains to the left of the area, and a few large relict grains to the right. The relict grains contain significant numbers of low angle boundaries and the whole area shows an abundance of Dauphiné twins (effectively 60° rotations about the c-axis). The grain size distribution is shown in Fig. 1b (overleaf): for the whole area the mean grain size is 18.54 ± 0.32 µm, excluding the twin boundaries. Even in this relatively small

OXFORD INSTRUMENTS

The Business of Science®

area, the crystallographic preferred orientation as visualised in a c-axis pole figure (Fig. 3c) shows the development of a crossed girdle structure, common in this type of deformed quartz-rich rock.

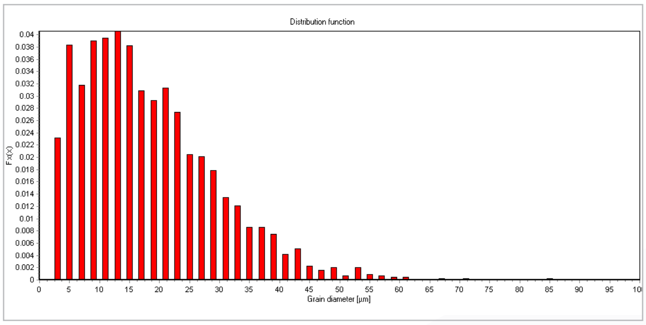


Fig. 1b. Grain size distribution histogram for the first measured area. N=2267, mean=18.54 ± 0.32 µm.

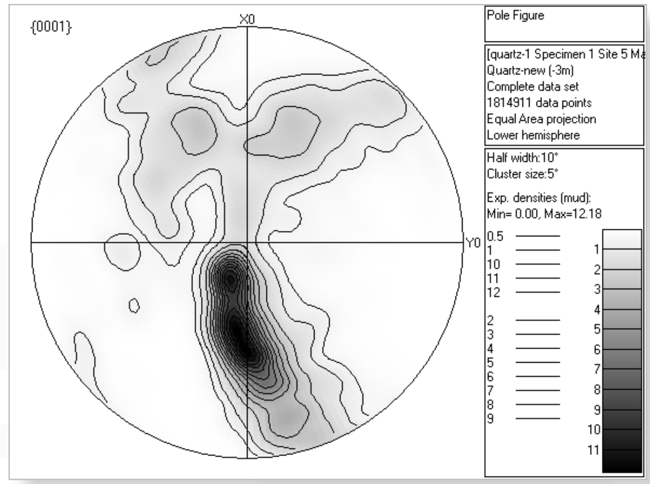


Fig. 1c. Contoured {0001} pole figure showing development of a c-axis crossed girdle.

A second, larger area was analysed using Large Area Mapping. In total an area of 9 x 1.5 mm was covered with a measurement step size of 1.5 µm. As with the first analysis, the hit rate was over 96% and the acquisition speed was just under 1000 pps; 6.2 million points were collected in 105 minutes. This area covered a higher strain area of the sample, and the orientation map (Fig. 2a) shows that few relict grains remain, with almost complete recrystallisation. Different coloured bands represent regions with different crystallographic preferred orientation, but the pole figure for the whole area (Fig. 2b) shows that here the c-axis crossed girdle has been fully developed.

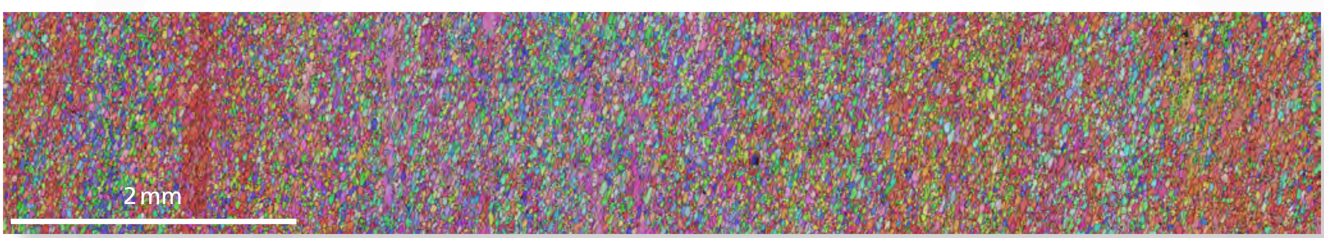


Fig. 2a. Orientation map of the second area (inverse pole figure colouring scheme). Grain boundaries are marked in black, low angle boundaries in grey and Dauphiné twin boundaries in red.

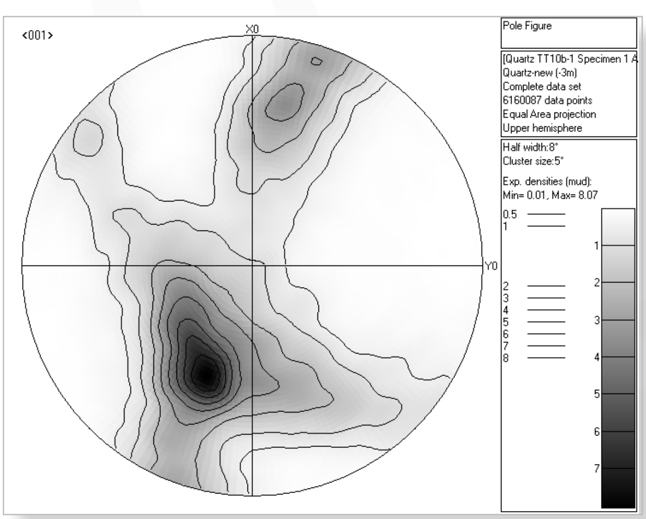
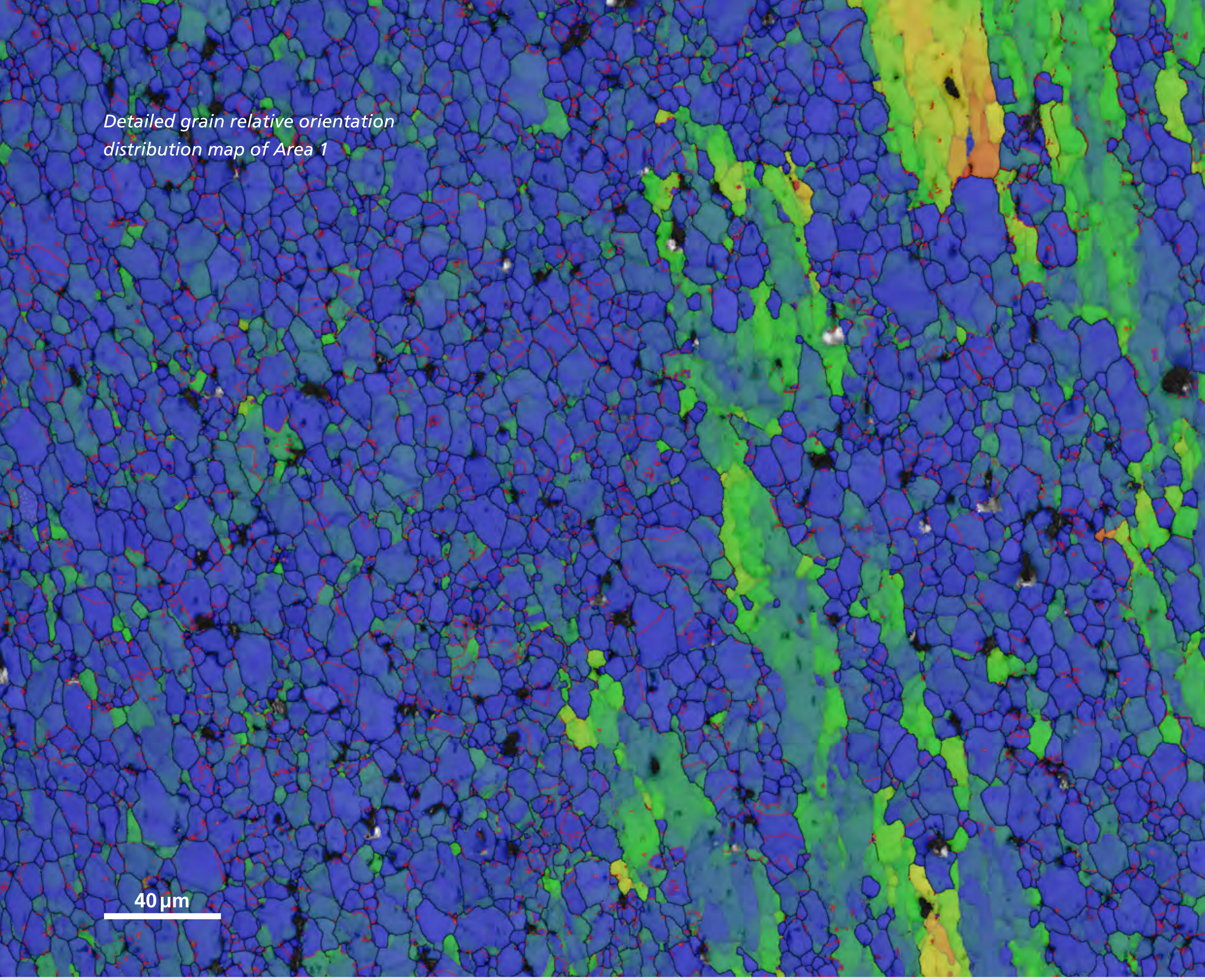


Fig. 2b. Contoured {0001} pole figure showing full development of a c-axis crossed girdle.



Detailed grain relative orientation distribution map of Area 1

Conclusion

This example demonstrates how the AZtec EBSD software in combination with the Symmetry EBSD detector is an ideal combination for the rapid and effective characterisation of simple geological samples. Unlike conventional CCD-based detectors, Symmetry permits high acquisition speeds without the need for excessive pixel binning, ensuring that good, high resolution patterns are collected at every point. The resulting, uncompromised data is of a high quality, making fast characterisation of the microstructures of rock samples a simple and routine process: what took hours or even days with conventional CCD-based detectors can now be completed in a matter of minutes, expanding the appeal of EBSD as a routine characterisation tool.

www.oxford-instruments.com/Symmetry

Document reference: OINA/AB/EBSD Symmetry/Rock samples/0517.