3D Crystal Structure Mapping by Diffractive Scanning Confocal Electron Microscopy (DSCEM)

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Background

Many material properties depend on the size / morphology of a sample's grains, and determining these accurately is essential for modern materials science. FIB milling with EBSD analysis offers large fields of view (10s of µm) and reasonable spatial resolution (≈25nm)[1] but is time consuming (10s of hours) and destructive, precluding any further specimen analysis. Here a non-destructive alternative is presented, including preliminary data; showing an ability to discriminate between grains across a grain boundary.

Optical Geometry

Wide-field STEM can be modified to set up a scanning-confocal geometry (SCEM)[2]; rejecting out of focus rays and improving depth sensitivity, Figure 1 [3].

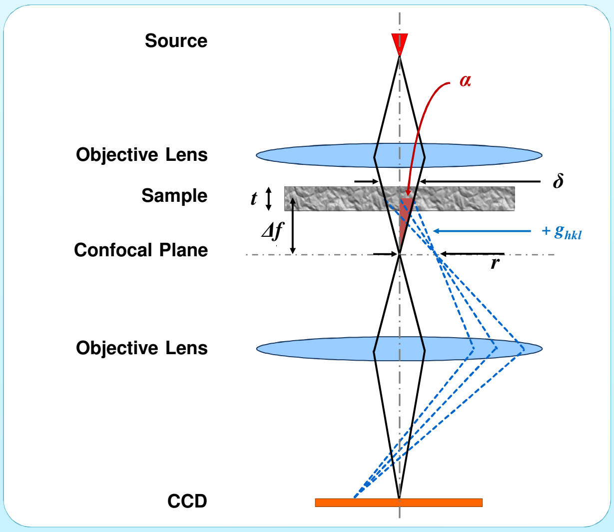

Figure 1. Diffractive SCEM optical geometry showing sample-probe intersection volume.

Wang et al. has demonstrated that raising the specimen well above the confocal plane yields a family of diffracted probe images; yielding information about crystal structure, as well as specimen thickness and height [4].

By positioning the CCD in the real-space plane optically conjugate to the confocal plane this entire plane can be recorded, Figure 2.

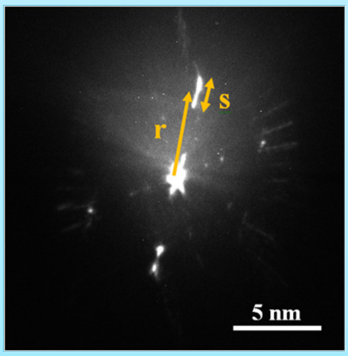

Figure 2. Experimental confocal-plane probe image from an YBCO film (t ≈ 200 nm).

The intensity and angular distribution of scattered spots depends on:

- Size of the objective aperture,
- Specimen thickness and height, and
- Angular proximity to a zone axis.

The radial distance of the diffracted probes, r, is the product of:

- Their reciprocal lattice vector, g_{hkl},
- The sample height, Δf, and
- The electron wavelength, λ.

$$r = \lambda \,.\, \Delta f \,.\, g_{hkl} \qquad (1)$$

As λ and g are known then the specimen offset Δf can be determined.

Each spot in Figure 2 is streaked with a finite radial spread, s, from diffraction through the thickness of the sample, t. Spots then are expected to be streaked between the solutions to Eqn. 1 from the entrance and exit surfaces [4].

$$t = \Delta f_{ent} - \Delta f_{ext}$$

$$t = s \,/\, (\lambda \,.\, g_{hkl}) \qquad (2)$$

Eqns. 1 & 2 allow a thin crystal's to be measured to a resolution of ≈ 5 nm [4]. Repeating this measurement over an XY raster means that the 3D profile of a grain, or boundary, can be mapped.

Specimen

The specimen studied in this work was an YBCO thin film prepared on a MgO substrate similar to those in ref 2. A Zeiss NVision was used to mill a specimen 10µm by 5µm with a thickness ≈200 nm.

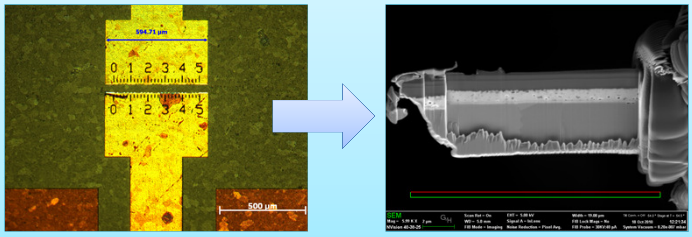

Figure 3. Left) Optical pre-milling image and, right) SEM post-milling image of YBCO sample.

Results & Discussion

A grain boundary was imaged edge-on using the Oxford-JEOL 2200MCO with pre- and post- specimen spherical aberration correction. Confocal plane images were recorded along a 400nm line at 20nm intervals, Figure 4.

Insets show a) no diffracted probes when the beam is over a hole, b) the pattern from grain 1, and c) the pattern from grain 2.

Lateral resolution is determined by the intersection volume width, δ (Figure 1), which is proportional to the sample offset and probe semi-angle.

$$\delta = 2 \,.\, \Delta f_{ent} \,.\, \alpha \qquad (3)$$

For the imaging conditions used in ref. 4 this would correspond to a lateral resolution of 8.1 nm. However for the thicker specimen imaged here ≈18nm is expected.

In Figure 4c) a weak contribution from grain 1 is seen. This arises from the probe-specimen intersection volume containing more than one grain. As the radial distance of the spots remains unchanged from inset b), the two patterns originating crystals must be at the same height. This is consistent with the predicted lateral resolution and an intersection volume being probed near an edge on grain boundary.

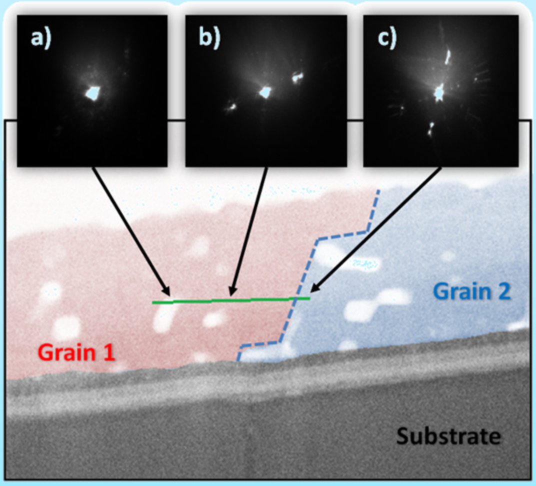

Figure 4. False-colour ADF image of grain boundary. Inset: corresponding confocal plane images.

Conclusions

- A non-destructive scanning-confocal grain-mapping technique is described.
- A line across the sample was investigated at intervals using diffractive SCEM and the associated confocal plane images were used to clearly discriminate between different grains, or holes, in the specimen.
- Extending this analysis to an XY raster and incorporating determination of the specimen height and thickness is expected to yield a non-destructive three-dimensional grain mapping technique.

References

1) Inkson, Mulvihill & Möbus 2001. Scripta Mat. 45.
2) Nellist et al. 2006. Applied Physics Letters 89.
3) Jesson & Pennycook 1993. PRS A 441, 261-281.
4) Wang et al. 2010. Ultramicroscopy 1-10.

Acknowledgments

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Further Information

Copies of this poster, the associated manuscript and author contact details are available online at:

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