Type of presentation: Poster

IT-5-P-2311 Characterising severe plastic deformation: a quantitative assessment of TEM imaging using transmission Kikuchi diffraction in the SEM

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The enhanced physical properties attributed to nanocrystalline materials have resulted in significant recent research focus on grain size refinement using severe plastic deformation (SPD). SPD-processed materials typically have ultrafine or nanocrystalline grain sizes, often with very high dislocation densities. These attributes make them difficult to analyse using conventional scanning electron microscope (SEM) based techniques such as electron backscatter diffraction (EBSD), resulting in most researchers relying on the higher resolution capabilities of the transmission electron microscope (TEM).

The recent emergence of transmission Kikuchi diffraction (TKD) in the SEM enables routine characterisation of materials with mean grain sizes below 50 nm, and with high intragranular dislocation densities [1, 2]. However, TKD analyses of samples previously characterised using TEM have sometimes produced discrepancies in the final grain size estimates.

This is the first in-depth direct correlation between TEM imaging and TKD in the SEM: we used a variety of Al-alloys that have been deformed using SPD, firstly imaging using 200 kV TEM (JEOL 2100) and then analysing the same areas using TKD in the SEM (Carl Zeiss Ultra Plus with Oxford Instruments AZtec EBSD).

The results reveal some startling differences. In an Al-6060 alloy deformed by high pressure torsion (5 revolutions at 180 °C, under 6 GPa), the recovery of dislocations enables relatively clear TEM imaging of the grain structure, as shown in fig. 1. TKD mapping (fig. 2) confirms the location of the high angle boundaries and shows that the intragranular dislocations and precipitates visible in the brightfield image are associated with very low misorientations (<1°). However, a misorientation profile across one grain shows that the cumulative lattice distortion can be significantly higher, in this case nearly 4° (fig. 3).

In Al-Cu-Mg alloys that have been deformed by equal channel angular processing (ECAP) at room temperature, the correlation between TEM and TKD is more challenging. The increased dislocation density makes clear TEM imaging difficult, and TKD results indicate that many grain-like features imaged in the TEM are in fact part of larger grains with significant intragranular subgrain structure. We will present numerous correlative analyses between the two techniques; the results have important implications for the characterisation of SPD materials, and show the benefit of the TKD technique for the rigorous measurement of microstructural properties.

References:

[1] R.R. Keller and R.H. Geiss, J. Microscopy, 245 (2012), p. 245-251.

[2] P. W. Trimby et. al., Acta Materialia, 62 (2014), p. 69-80.

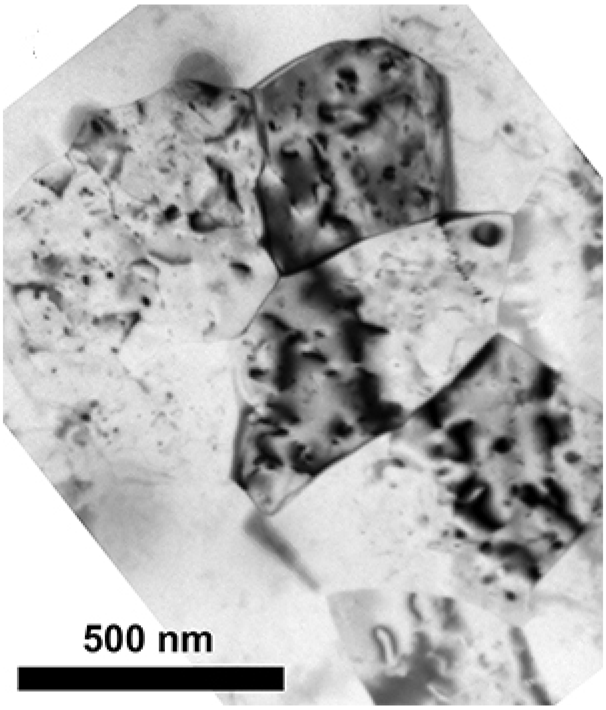

Fig. 1: Bright field TEM image of an HPT deformed Al-6060 alloy.

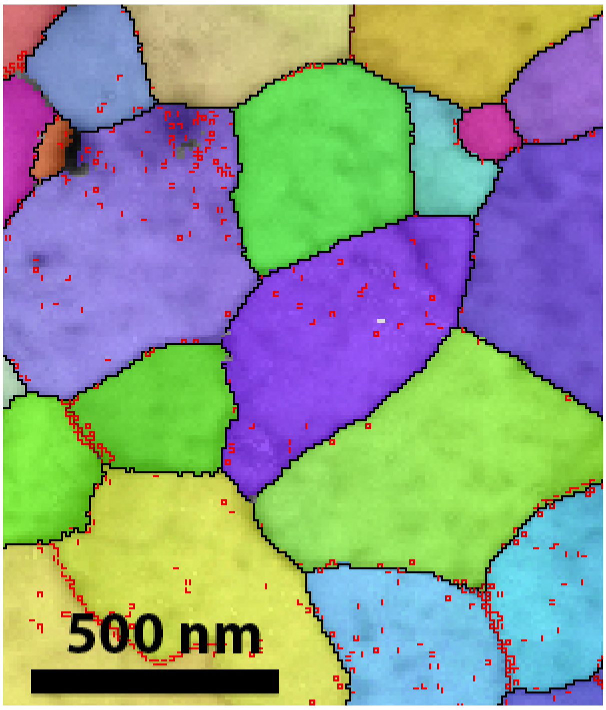

Fig. 2: TKD orientation map (IPF colouring) of the same area (measurement step size 8 nm), with high angle boundaries (>10°) in black, low angle boundaries (1-10°) in red.

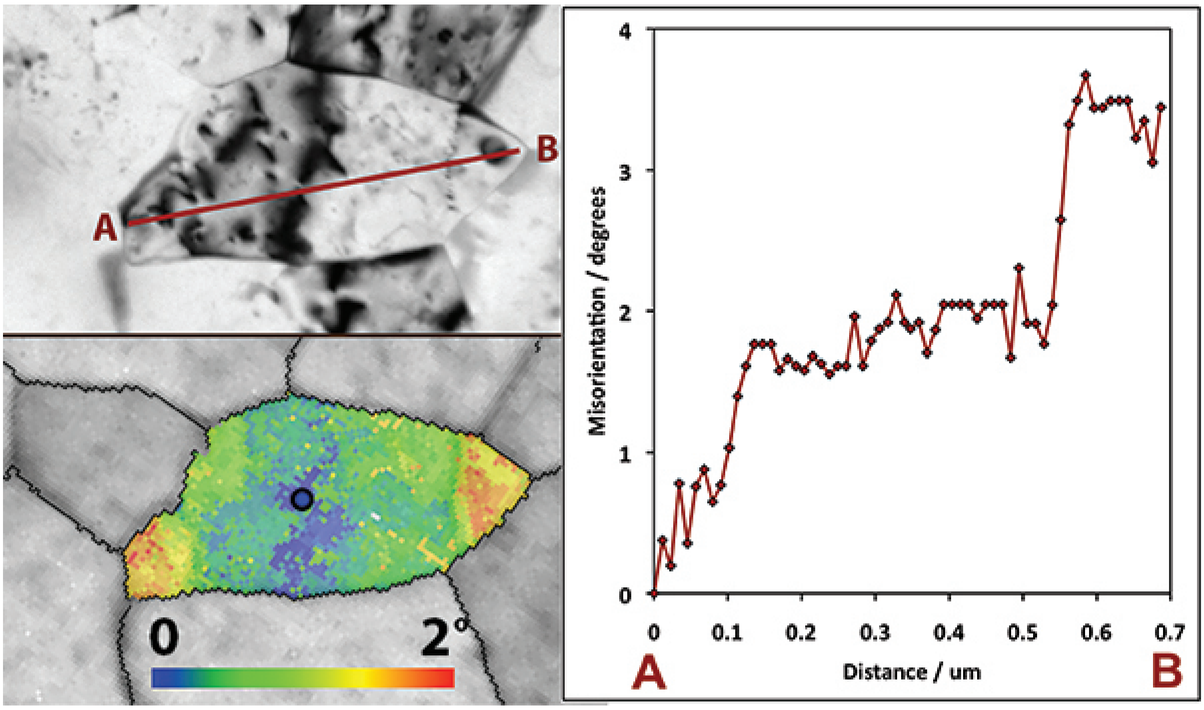

Fig. 3: Detailed quantitative analysis of a single grain in Figs 1 & 2. Top left: bright field TEM image. Bottom left: TKD map showing the change in lattice orientation relative to the central spot. Right: graph showing the change in orientation across the 700 nm transect A-B, relative to point A.