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Characterization of a Sub-Grain Boundary Using Accurate Electron Channeling Contrast Imaging

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Electron Channeling Contrast Imaging (ECCI) is a powerful technique in scanning electron microscopy (SEM) for observing and characterizing crystallographic defect such as dislocations, stacking faults, and grain boundaries. In order to detect defects, ECCI uses the fact that backscattered electrons are very sensitive to the angle between the incident beam and the crystal lattice. To characterize defects, it is necessary to carry out ECCI under controlled two beam channeling conditions. In the past, these imaging conditions were established using electron channeling patterns (ECPs), selected area channeling patterns (SACPs), or electron backscatter diffraction patterns (EBSD), but all of these approaches have either spatial or angular resolution limitations.

We have recently developed a novel approach for collecting high angular (0.1^{o}) and spatial resolution (~500nm) selected area channeling patterns (HR-SACPs) that allows high accuracy control of ECCI channeling conditions [1,2].This approach, termed Accurate ECCI (A-ECCI), has been applied to unambiguously characterize screw dislocations in IF-steel using the **g•b**=0 invisibility criteria [1].

In this contribution, A-ECCI performed in a Zeiss AURIGA 40 FIB SEM is used to analyze a sub-grain boundary in a polycrystalline bcc IF-steel slightly deformed in tension. HR-SACPs and ECC images were collected using a large four-quadrant Si-diode backscattered electron detector. Prior to the HR-SACP collection, the sample was tilted to 70° to determine the orientation of the region of interest by EBSD. Since A-ECCI is carried out at low tilt, the EBSD data was used to determine the approximate orientation of the crystal at 0° tilt. Specific channeling conditions were then established by HR-SACP in conjunction with tilt and rotation of the sample.

Figures 1a and b show ECC images of the sub-grain boundary composed of individual dislocations, which appear as white line segments. HR-SACPs collected from either side of the boundary (indicated on Figure 1a) reveal a disorientation of ≈0.2°, with the rotation occurring along the red dotted line. The spacing between dislocations in the boundary increases from the lower left side to the upper right side of the sub-boundary until the boundary vanishes, indicating that the disorientation decreases along the boundary. Using the dislocations projected length (≈450nm) and a depth visibility of ≈60-70nm, the inclination angle was calculated to be about 6°-9°. The dislocation lines show fading dotted contrast, indicating their sense of inclination, which is plotted in red on the stereographic projection in figure 1f. The trace and inclination correspond to the [10-1] direction. The dislocations along the sub-grain boundary were characterized using the **g•b=0** and **g•(bxu)=0** invisibility criterion, as shown in figure 1.b-e. The dislocations go out of contrast for **g**=(10-1), consistent with dislocations having a Burgers vector b=[1-11] and being edge in character. This is also consistent with the misorientation axis of the boundary, and consistent with the boundary being tilt in character. Finally, the average spacing of the dislocations in the boundary is in close agreement with the calculated $\boldsymbol{D} \sim \frac{\boldsymbol{b}}{\boldsymbol{\theta}}$ for tilt boundaries, and b=½[1-11].

This work demonstrates that A-ECCI assisted by HR-SACPs is a powerful technique and experimentally robust method for studying the misorientations of sub-boundaries and dislocations that make them up. The misorientation of low angle boundaries with misorientations as small as 0.2°, below the capabilities of standard EBSD systems, can be characterized along with the nature of the dislocations that make up the boundaries.

References:

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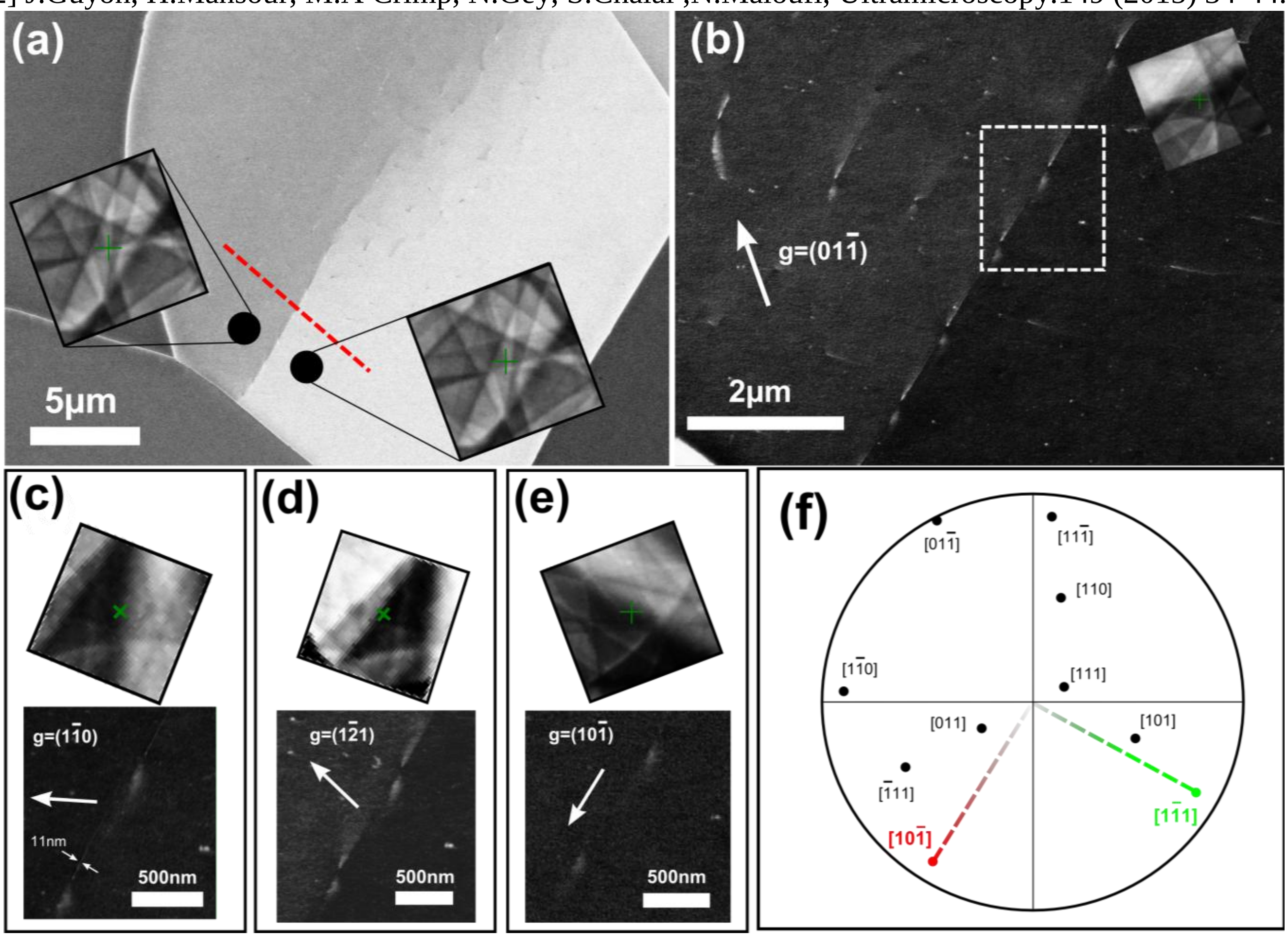

Figure1. Characterization of a sub-boundary using A-ECCI. (a) ECC Image of the sub-boundary. Two HR-SACP were acquired from each side of the boundary.(b-e)Burgers vector analysis on the dislocations applying g•b=0 and g•(bxu)=0 invisibility criterion using different g vectors (b) g=(01-1); (c) g=(1-10); (d): g=(1-21); (e): g=(10-1). (f) Stereographic projection showing {111} and {110} poles.