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Rotation Axes Analysis of Deformed Magnesium Using Electron Backscatter Diffraction and Rotation Contour Contrast Reconstruction

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In a deformed polycrystalline microstructure, each grain has a non-uniform gray level in the backscattered electron (BSE) micrograph in a scanning electron microscope (SEM). A non-uniform gray level represents a local crystallographic contrast i.e., local misorientation inside a deformed grain. If deformation occurs in a progressive manner inside a grain, the variation of crystal orientation is not random. As a result, a regular crystallographic contrast in the form of parallel or concentric contours appears inside a deformed grain [1]. The rotation contour contrast (RCC) terminology was used to relate this contrast to the rotation of crystal about one or multiple rotation axes during deformation [2]. In this study, the RCCs in the form of cross-shaped and intersecting contours were observed in deformed Magnesium (Mg) grains in a BSE micrograph. This contrast was attributed to the rotation of the crystal about two rotation axes. The crystallographic directions of the two rotation axes were identified using electron backscatter diffraction (EBSD) and RCC reconstruction [3].

The Mg-0.2Al-0.3Ca (wt%) alloy was selected for rotation axes analysis in this study. The uniaxial hot-compression test was carried out at a temperature of 400 °C, a strain rate of $0.01s^{-1}$, and a strain of 0.6 using a 100 kN servo-hydraulic materials testing system. The BSE imaging was carried out at 10° specimen tilt, a 20 keV electron beam energy and a 15 mm working distance using a Hitachi SU-8000 cold-field emission SEM. The EBSD crystal orientation mapping was carried out at 80º specimen tilt (sample surface at 10º from the electron beam axis), a 20 keV electron beam energy and a 25 mm working distance. To index the electron backscatter diffraction patterns (EBSPs) recorded at 80º specimen tilt, a calibration routine was carried out with the standard single crystal silicon wafer specimen with a surface normal [001] and a [110] type reference direction. Using this calibration, the recorded EBSPs were indexed with the HKL Channel5 Flamenco software. A Matlab code was written to read the gray level of a selected pixel in the recorded EBSPs for RCC reconstruction. This pixel represents the position of a virtual electron beam in the EBSP [4].

Fig. 1a shows the BSE micrograph with RCC_1 and RCC_2 across a deformed Mg grain. Fig. 1b shows a reference EBSP (P_{ref}) obtained from the center of the grain with the pattern center indicated with a red cross. Fig. 1c shows the magnified $[11\bar{2}0]$ zone axis of the P_{ref} for RCC reconstruction. For the first set of RCC reconstruction, five pixels were selected inside the (0002) Kikuchi band indicated with labels *a*-*e* (set I in Fig. 1c). For the second set of RCC reconstruction, five pixels were selected close to the pattern center along a line parallel to the $(\bar{1}100)$Kikuchi band indicated with labels *f*-*j* (set II in Fig. 1c). Fig. 1d and 1e show the reconstructed images for set I and set II, respectively. The reconstructed RCCs in image *b* and image *g* were very similar to the RCC_1 and RCC_2 in the BSE micrograph since pixel *b* and pixel *g* were selected near the pattern center (i.e., BSE micrograph and reconstructed images *b* and *g* have similar diffraction conditions). In set I RCC reconstruction, while RCC_1 remained stationary inside the grain, RCC_2 moved from the top to the bottom of the grain when the virtual electron beam position moved from pixel *a* to pixel *e*. The one-directional movement of the RCC_2 from the top to the bottom of the grain in the images *a*-*e* indicated a one-axis rotation of the grain about the axis perpendicular to the

(0002) Kikuchi band. In set II RCC reconstruction, despite the variations in position, shape and width of the RCC_1 with the movement of the virtual electron beam from pixel *f* to pixel *j*, the RCC_2 remained stationary to a good approximation. However, the RCC_1 moved in a lateral direction across the grain in images *g* to *j*. The one-directional movement of the RCC_1 in a lateral direction across the grain in the images *g-j* indicated a one-axis rotation of the grain about the axis perpendicular to the $(1\bar{1}00)$ Kikuchi band. As a result, the two rotation axes in this grain were identified as crystallographic directions perpendicular to (0002) and $(1\bar{1}00)$ Kikuchi bands, respectively.

References:

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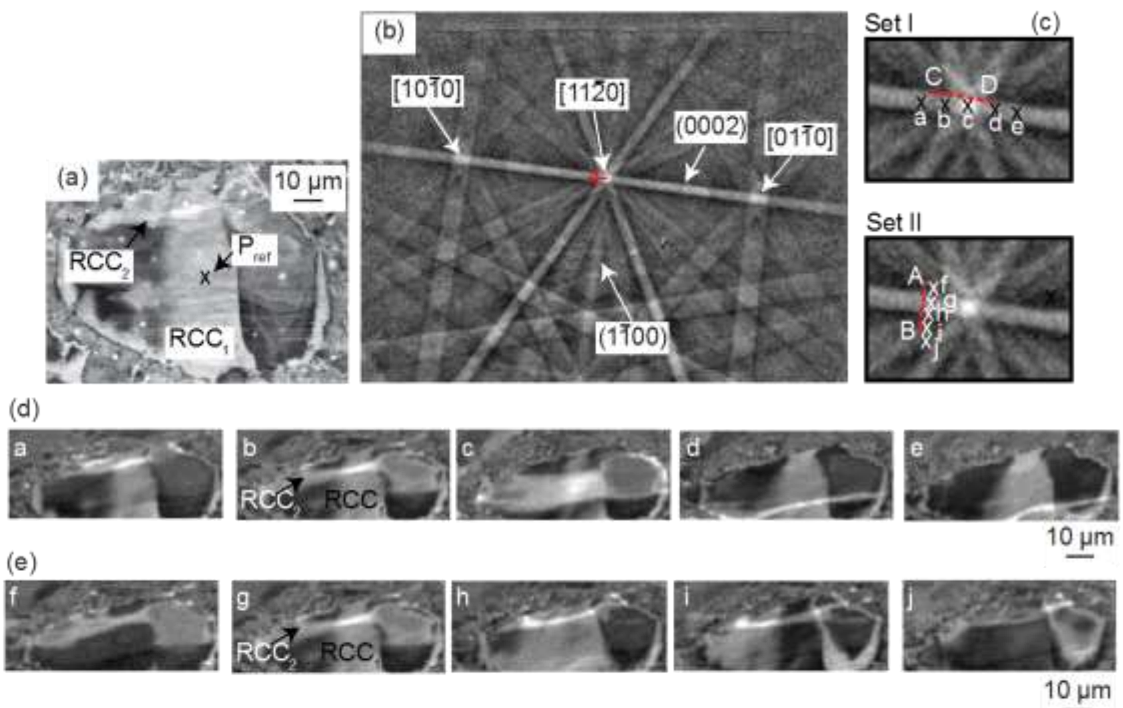

Figure 1. Identification of the crystallographic directions of the two rotation axes in a deformed Magnesium grain. (a) The backscattered electron micrograph of a deformed grain with a rotation contour contrast (RCC) labeled RCC_1 and RCC_2 across the grain, (b) A reference electron backscatter diffraction pattern (P_{ref}) obtained from the center of the grain in (a), with the pattern center indicated with a red cross, (c) The magnified $[11\bar{2}0]$ zone axis for set I and set II RCC reconstructions, (d) The reconstructed images for pixels *a-e* in set I RCC reconstruction, (e) The reconstructed images for pixels *f-j* in set II RCC reconstruction. The two rotation axes in this grain were identified as crystallographic directions perpendicular to (0002) and $(1\bar{1}00)$ Kikuchi bands, respectively.