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Comparison of dislocation content measured with transmission electron microscopy and micro-Laue diffraction based streak analysis

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ABSTRACT

The subsurface dislocation content in a Ti-5Al-2.5Sn (wt%) uniaxial tension sample deformed at ambient temperature was characterized by peak streak analysis of micro-Laue diffraction patterns collected non-destructively by differential aperture X-ray microscopy, and with focused ion beam transmission electron microscopy of material in the same volume. This comparison reveals that micro-Laue diffraction streak analysis based on an edge dislocation assumption can accurately identify the dominant dislocation slip system history (Burgers vector and plane observed by TEM), despite the fact that dislocations have predominantly screw character. Other dislocations identified by TEM were not convincingly discernible from the peak streak analysis.

Dislocation content is commonly characterized using techniques such as electron backscattered diffraction (EBSD) for geometrically necessary dislocation (GND) measurements [1,2], transmission electron microscopy (TEM), and electron channeling contrast imaging (ECCI) in near-surface regions of bulk samples [3]. All these techniques are limited to near-surface regions, rely on destructive preparation, and/or image only small volumes. In contrast, high-energy X-ray Laue micro-diffraction provides an opportunity to non-destructively characterize subsurface dislocation content up to depths of hundreds of microns with an effective diffracting volume of approximately 1 μm^3, but it lacks the direct dislocation imaging of TEM or ECCI. This method was initially used to identify *edge* dislocation content from streaked diffraction peaks [4]. In that study and all subsequent analyses, an assumption of pure edge dislocation character was made to infer the dislocation content giving rise to the streak [5–9]. Nevertheless, Bragg's law states that Laue diffraction peak shapes are determined by the lattice orientation distribution in the diffracting volume, but the relationships between the shape of the peak and the dislocation content of the diffracting volume have rarely been discussed. The objective of this letter is to examine whether the interpretation based on edge dislocation content can be applied to structures with a significant amount of screw dislocations.

To this end, a Ti-5Al-2.5Sn (wt%) sample deformed in uniaxial tension at ambient temperature (see Ref. [10] for details) served as a model material. A diffraction spot streak analysis was carried out on a differential aperture X-ray microscopy (DAXM) data set collected at beamline 34-ID-E (Advanced Photon Source, Argonne National Laboratory) and compared to the dislocation content assessed from a TEM foil extracted with a FEI Helios DualBeam FIB/SEM focused ion beam from the same near-surface region as studied in the prior DAXM experiment. Fig. 1 reveals the spatial relationship between the locations of the DAXM data set penetrating at an angle of 45 ° below the surface and of the TEM foil identified by its top edge A–B. One DAXM voxel lying close to the intersection with the TEM foil and showing clearly streaked peaks was selected for streak analysis (Fig. 1 top right). The orientation of the TEM foil was chosen to allow access to a sufficient number of diffraction vectors to reliably determine the nature of dislocations through $\mathbf{g}\cdot\mathbf{b}$ invisibility assessment of the foil examined with a JEOL 100CX II TEM operating at 120 kV in conjunction with line trace analysis.

The TEM images shown in Fig. 2 reveal dislocations arranged in many bands along traces of the basal plane (lower left to upper right) while only one slip band along the trace of a prismatic plane is apparent, *i.e.* from upper left to lower right, both being consistent with the blue and red traces in Fig. 1 upper left (rotated roughly 60 °). The dislocation contrast variation under different diffraction conditions (shown in Fig. 2) reveals that most of the dislocations in the basal slip bands have $\mathbf{b} = \mathbf{a}_3 = \pm\frac{1}{3}[\bar{1}\bar{1}20]$ Burgers vector (blue arrows in Fig. 2a), while a smaller number of them have $\mathbf{b} = \mathbf{a}_1 = \pm\frac{1}{3}[2\bar{1}\bar{1}0]$

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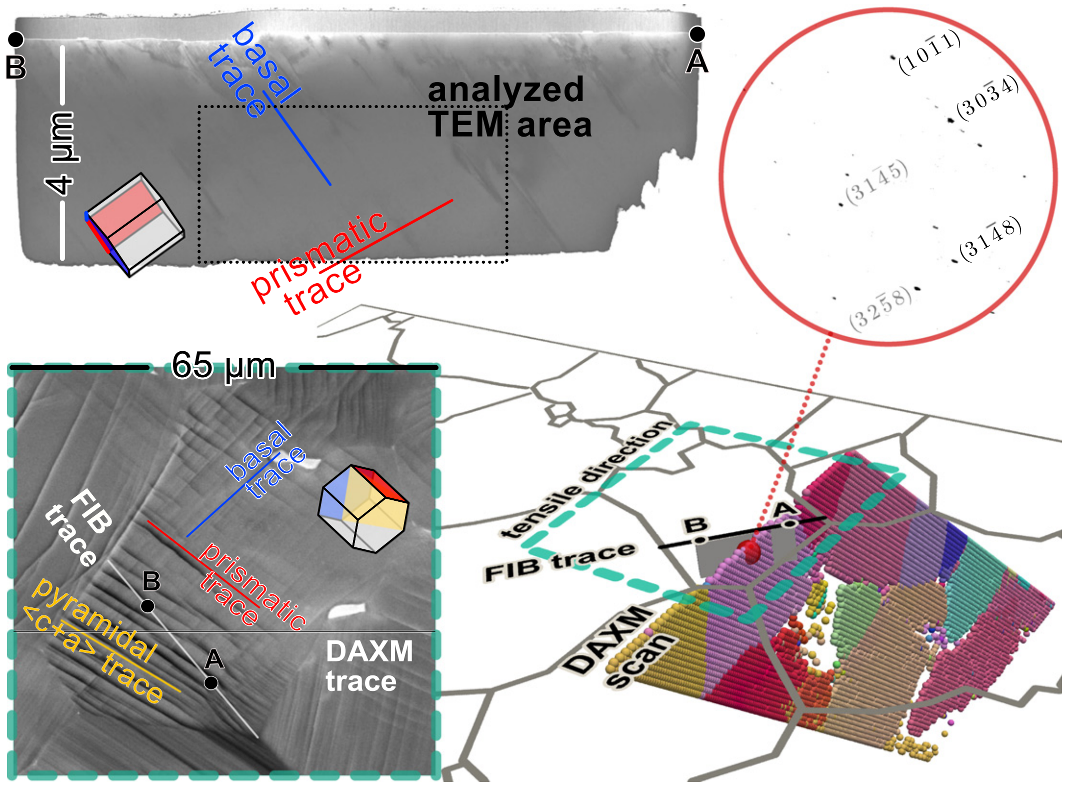

Fig. 1. Three-dimensional view (bottom right) of the analyzed grain patch with gray boundaries on the sample surface and subsurface DAXM scan volume showing the grain structure as colored points on a ~1µm thick plane perpendicular to the surface, with the X-ray microbeam entering the sample 45° from the surface. The FIB-extracted TEM foil (gray rectangle perpendicular to the surface) passes through the DAXM scan; a low magnification image of the foil (top left) identifies the region analyzed by TEM (dotted frame). Backscattered electron image (bottom left, dashed frame) shows that the surface slip steps of the analyzed (central) grain are consistent with traces expected for basal slip (blue) or pyramidal $\langle c + a \rangle$ slip (yellow) but not necessarily with prismatic slip (red). Micro-Laue diffraction pattern (top right) originates from the voxel of interest (located at the center of the larger red sphere close to TEM foil location, roughly 3 µm below the sample surface), where the streak indicates about 0.5° lattice orientation spread. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

(red arrows in Fig. 2a). In all of the five projections, the dislocations marked with blue arrows run almost parallel to their $\mathbf{a}_3$ Burgers vector shown in blue on the hexagonal unit cells; they have screw character. Similarly, the dislocations marked in red can be identified as screw dislocations with $\mathbf{a}_1$ Burgers vector. Most of the dislocations in the prismatic slip band (white arrows) were identified to also have $\mathbf{b} = \mathbf{a}_1$, and hence, they would be mobile on the $(0\bar{1}10)$ prism plane, and a smaller number have an $\mathbf{a}_3$ Burgers vector. Compared to the mostly straight dislocations seen in the basal slip bands, the dislocations in the prismatic band are generally more tangled, which may result from basal slip intersecting the prismatic slip band. The high (low) frequency of slip band observations in the TEM foil of basal (prismatic) slip is consistent with the more narrowly (widely) spaced surface slip traces evident in the SEM image in Fig. 1.

To assess whether the mostly near-screw dislocation content observed in the TEM foil can be identified by a (nominally pure edge dislocation-based) analysis of the streaked diffraction peaks, a DAXM voxel (red sphere in Fig. 1) that falls close to the TEM foil location was analyzed. The upper left part of Fig. 3 shows the streaked $(3\ 0\ \bar{3}\ 4)$ diffraction peak as an example concentrically surrounded by all theoretically expected streak directions from edge dislocation content. The three different $\langle a \rangle$ Burgers vectors are distinguished by line type (solid, dashed, dotted) for the basal (blue), prism (red), and pyramidal (green) systems, but no line type distinction is made for $\langle c + a \rangle$ systems. The $(3\ 0\ \bar{3}\ 4)$ peak streak direction is consistent with the presence of $\mathbf{a}_3$ dislocations on the basal plane (dotted blue line). The observed streak direction is also close to that expected from edge dislocations on two pyramidal (solid and dashed green lines) as well as on one pyramidal $\langle c + a \rangle$ plane (orange line collinear with dashed green line). Analysis of additional streaked peaks (shown in Fig. 3), results in $\mathbf{a}_3$ dislocation content on basal planes (dotted blue) as the only dislocation content consistent with the major streak direction in all three analyzed peaks. This assessment is consistent with the slip activity anticipated using global Schmid factors (unidirectional tensile stress state), as indicated by line opacity of the different theoretical streak directions in Fig. 3, as well as with the predominant dislocation content found in the TEM analysis.

The critical aspect of this study is that although the peak streak analysis model is based on an edge dislocation assumption, the analysis successfully determines the Burgers vectors of the dominant dislocation content despite being predominantly screw in character. This can be rationalized with a thought experiment involving a spherical Eshelby inclusion (Fig. 4 left) that is homogeneously sheared into an ellipsoid by uniformly-spaced dislocation loops. To force this deformed volume back into the originally occupied spherical volume, a homogeneous shear stress as well as a rigid-body rotation are required (Fig. 4 right). Because this rotation is the same everywhere within the sheared volume, any part of the interface has the same misorientation from the surrounding volume. Thus, diffraction from any voxel containing a part of the interface, such as the red and blue voxels indicated in Fig. 4 that comprise both parent and sheared material, would result in the same diffraction spot splitting. During and after plastic deformation, such interfaces are generally not sharp since dislocations are typically not aligned in perfect walls, resulting in diffraction peaks that will be smoothly streaked according to the local orientation gradient.

According to the edge dislocation model for streaked peaks, edge dislocations with the same Burgers vector but different line