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Focused Ion Beam (FIB) based Tomography of Dislocations Using Electron Channeling Contrast Imaging (ECCI)

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Characterizing the distribution of dislocations in 3-dimensional space is vital for understanding the conditions leading to failure in structural and electronic device materials. Traditional transmission electron microscopy (TEM) 3D-tomography is limited to volumes defined by TEM thin foils. SEM based electron channeling contrast imaging (ECCI) is an effective alternative to TEM for imaging and indexing dislocations [1]. Using this technique, dislocations in bulk samples within interaction volumes of < 200nm can be characterized using a bright electron source and solid state BSE detector. ECCI can be coupled with focused ion beam (FIB) serial sectioning to generate 3D dislocation maps [2]. Nevertheless, unlike 3D-EBSD serial sectioning, fully automated ECCI-FIB based tomography has not been developed for a number of reasons: 1) Commercial dual beam FIB-SEMs do not currently offer the software interface for ECCI. 2) It is not trivial to setup proper channeling conditions for ECCI 3) Ion milling conditions have to be optimized to retain the sample surface quality for ECCI. In this study, optimum conditions for 3D-ECCI have been developed and post processing of the images for automated dislocation position identification is demonstrated.

For this study, a polycrystalline high purity nickel sample was deformed to ~3% plastic deformation to generate dislocations. FIB milling conditions were established using a commercial FEI Helios NanoLab FIB-SEM. ECCI was carried out both on the Helios Nanolab and on a TESCAN MIRA SEM, both of which allow the selected area channeling patterns (SACPs) necessary to establish proper imaging conditions to be collected [3,4]. Low-incident surface milling FIB milling was carried out at 54° using a 45° pre tilted stub and an additional 9° stage tilt. Minimum surface damage was balanced with a reasonable milling time using 5KV and 15pA. A rectangular profile of 30μm×7μm was serial sectioned, with each milling slice taking approximately 25 minutes. Atomic force microscopy determined that these conditions led to approximately 25nm of material removal for each step.

Figures 1a-d show a series of ECCI images corresponding to the first four FIB milling sections. Many dislocations appear as dots threading out of the surface with bright/dark contrast (some indicated by circles in Figure 1a). Some isolated curved line segments are also visible (circled in Figure 1b). Over a milling depth of 75nm, the dislocations remained in the field of view and no noticeable beam damage was observed. Closer inspection revealed that the relative positions of some of the dislocations changed, as shown by the insets for the same area in figures 1a and d. A dislocation identification-3D reconstruction image analysis algorithm has been development. The position and nature (line segments or dots) of dislocations in Figure 1a have been identified using this algorithm, as indicated in Figure 2. This position identification is currently being used to reconstruct 3D tomographic maps of the dislocations through the ion milled volumes.

References:

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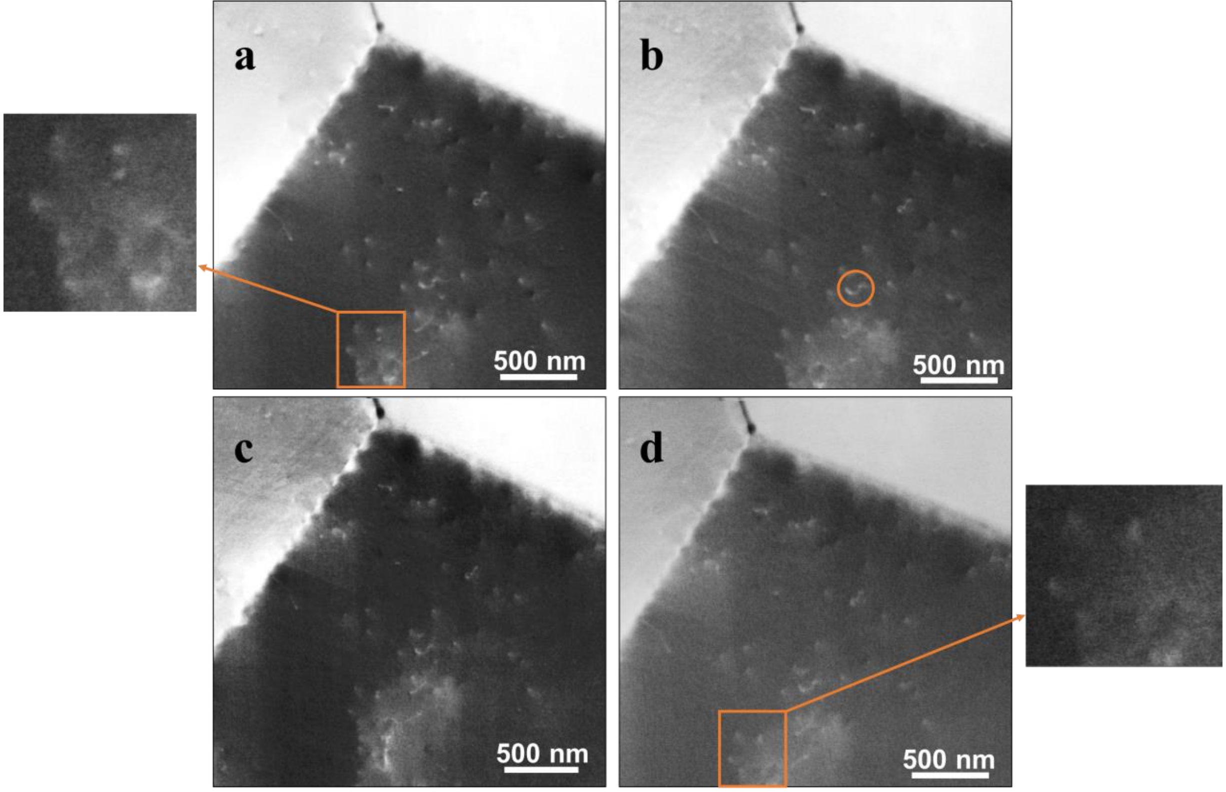

Figure 1. ECC images of the a) as-deformed/un-milled surfaces and subsequent FIB milled sections for b) 25 nm, c) 50 nm, and d) 75 nm. Insets illustrate slight changes in the relative positions between the un-milled surface and 75 nm milled surface, revealing the 3D positions of the dislocations.

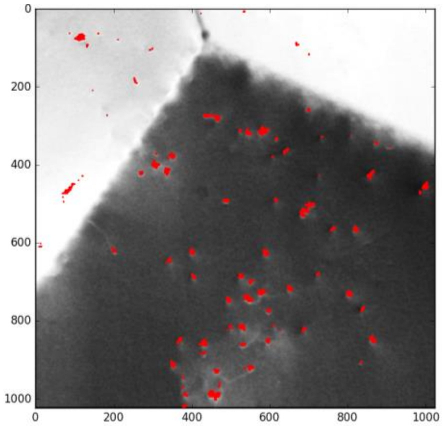

Figure 2. Positions of dislocation in figure 1a determined using an automated dislocation identification algorithm.