

HIERARCHICAL CHARACTERIZATION OF DEFORMATION HETEROGENEITIES IN BCC CRYSTALS

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ABSTRACT- Deformation behavior of body-centered cubic (BCC) metals is being investigated by white beam x-ray microdiffraction to characterize the dislocation structure that results from uniaxial compression experiments. The measurements were performed on molybdenum single crystals and a tantalum bicrystal as part of a hierarchical characterization effort. Results show heterogeneities in the deformed structure and misorientation maps consistent with results obtained from Orientation Imaging Microscopy (OIM). Additionally, the technique allows for the determination of the active glide systems as well as of the dislocation densities in function of the position in the sample.

INTRODUCTION: BCC materials are of scientific interest because of the anomalous deformation behavior noted in the literature for the past 40 years (Christian [1983]). In order to fully understand it, we have used the emerging technique of Synchrotron white beam X-ray microdiffraction to map heterogeneities, residual stress and local distortion in molybdenum single crystals and a tantalum bicrystal oriented for single slip and loaded in compression. White beam microdiffraction serves a critical role within the hierarchy of such detailed characterization; stand-alone TEM characterization is too inefficient to be useful, and optical and atomic force microscopies cannot relay information on the internal strain state. The advantage of this technique over TEM is the ability to probe over large areas (100 microns), and still have the spatial resolution (1 micron) to distinguish changes in the local dislocation structure. The heterogeneity of the plastic deformation is apparent in the

observation of localized streaking of the Laue reflections (Fig. 1.). While spot positions in the diffraction pattern define grain and subgrain orientation, their shapes convey information on the dislocation structure and distribution. The results of our preliminary investigation will be presented here and compared with OIM and TEM characterizations.

PROCEDURES, RESULTS AND DISCUSSION: The data were collected at the X-ray microdiffraction beamline (7.3.3) of the Advanced Light Source (ALS) in Berkeley. The beamline focuses the Synchrotron white beam (6-14keV) from a bending magnet using a pair of elliptically bent Kirkpatrick-Baez mirrors to achieve a spot size of approximately $1\mu m \times 1\mu m$ at the sample surface. Details of the apparatus and experimental setup can be found elsewhere (see MacDowell [2001] and Tamura [2002]). The Mo samples studied consist of single crystals sized $5mm \times 5mm \times 15mm$ oriented for single slip and strained to a maximum of 3%. The Ta bicrystal consists of two single crystals diffusion bonded together to create a 90° twist boundary and then compressed to 30-40% strain (Campbell [2004]). The Laue patterns are automatically indexed and analyzed using software developed at ALS to determine the orientation and strain. The misorientation between any two grains as well as orientation variations within single grains can be determined with a precision of 0.01°. An unstrained crystal was used as a reference for the strain measurements of the deformed crystal, with a strain sensitivity of about 2×10^{-4} .



Figure 1: μ XRD Laue pattern from the strained tantalum bicrystal sample at a location far from the boundary between the 2 crystals.

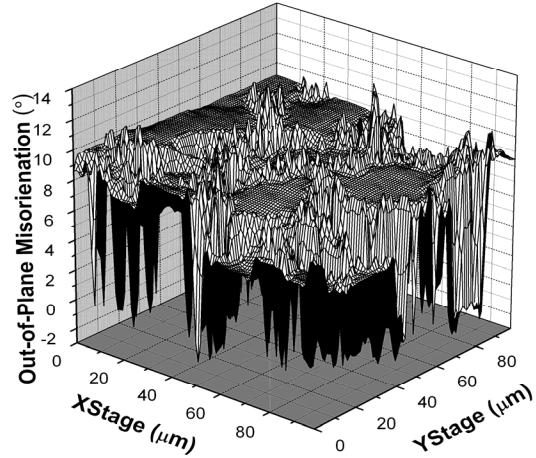


Figure 2: μ XRD map of the out-of-plane misorientation of the normal direction for the tantalum bicrystal far from the boundary.

The Laue patterns from the Ta bicrystal sample, (Fig. 1), show extensive

streaking characteristic of large plastic deformation and large dislocation density in the sample. The orientation maps of the bicrystal, as seen in Fig. 2, indicate a cell-like structure of dense dislocation walls. This deformation structure is consistent with previous OIM studies of similarly oriented and deformed single crystals (Schwartz [1999]). The cell boundaries correspond to localized high misorientation zones. Close to the boundary, rotations on the order of 10° are found, while the well-developed cell structure is found far from the boundary, with misorientations between 5°-11° at the cell boundaries. Moreover, sub-cell structures separated by high dislocation densities walls (up to $6(10)^9 \text{ cm}^{-2}$) have been detected. Simulations of the shape of the Laue reflections are part of an on-going effort to map the active glide systems in the samples. The results are being used as a proof-of-principle test to understand the more complicated dislocation structures of the Mo single crystals.

CONCLUSIONS: The investigation by white beam X-ray microdiffraction shows the capabilities of the technique to discern heterogeneity in the deformation structure from strained samples which complement other microscopy studies. Further work is underway to determine the nature of the dislocation structure. Future TEM work will allow correlation between the x-ray microdiffraction results and direct observations of the dislocation structure.

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