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Determination of Deformation Inhomogeneity in Polycrystalline Ni under Uniaxial Tension with 3D X-ray Microscope

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Subject Categories: Dislocations

Abstract

Deformation substructures due to intergranular interactions have significant effects on materials evolution such as recrystallization. Measurement of local misorientations introduced due to the formation of substructures is essential to understand deformation mechanisms. In this paper, the evolution of lattice rotations within submicron volume elements of neighboring grains in a polycrystalline Ni sample was studied by polychromatic 3D x-ray microscopy (P3DM) as a function of plastic strain. Individual grains exhibit distinct rotation patterns due to grain-to-grain interactions. Variations in rotation as a function of depth were also detected for all the grains measured. The results provide direct feedback for strain gradient plasticity model.

Introduction

There has been a long-term interest in deformation inhomogeneity on both the inter- and intra-granular scale. The recently commissioned polychromatic three-dimensional X-ray microscope (P3DM) [1, 2] with submicron beam size is a tool particular suitable for the study of deformation features in polycrystalline materials in local scale; X-ray diffraction is sensitive to the local unit cell orientation. In the case of Laue diffraction, streaked Laue pattern carry information on the formation of geometrically necessary boundaries (GNBs) and geometrically necessary dislocations (GNDs).

Characterization of the peak shapes and intensity distributions can be made in terms of the GNDs and GNBs that provide experimental feedback for strain gradient plasticity theory [3, 4]. Strain gradient direction, dislocation density and the probable activated dislocation slip systems can all be determined from the experimental information.

Here a study of the evolution of crystallographic orientation in neighboring grains in a Ni polycrystal on mesoscopic level has been carried out using the synchrotron X-ray microbeam technique. Measurements of the subgrain rotation under deformation are reported as a function of depth beneath sample surface. Analysis of the diffracted Laue patterns within the framework of strain gradient plasticity will be discussed.

Experimental Details

Rotations within individual grains in a Ni polycrystalline tensile sample as a function of depth and plastic strain were determined. The sample is 99.96% pure Ni with an average grain size of 200 μm and the dimensions of the sample are shown in Figure 1. The sample was annealed in a vacuum at 800°C for 4 hours. The experiment was carried out on beamline ID-34E at the Advanced Photon Source using a polychromatic X-ray microbeam. Laue patterns diffracted from four neighboring grains using a beam with cross-section of 0.46×0.55 μm^2 were measured as a stepwise function of plastic deformation at 0%, 10% and 15%. The beam penetrated ~ 30 μm beneath the surface normal.

Figure 1 gives the dimensions of the samples together with the sample coordinate system. Laue images diffracted from the sample were collected by an x-ray sensitive CCD. The orientation map collected at 0% strain over a region of 4×0.5 mm^2 at a step size of 5 μm around the center of the sample is overlaid on the overall sample profile (Figure 1).

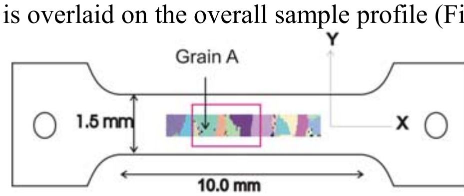

Figure 1. Dimensions of the tensile sample. The red box highlighted the four grains measured in the experiment.

Four grains, labeled A – D, highlighted by a red rectangle in Figure 1, were selected for detailed rotation measurements with increased deformation. Laue patterns were collected at the center of each grain at 0%, 10% and 15% strains. The Laue peaks are streaked at 10% and 15% deformation. Indexing of the streaked Laue patterns is impossible, as the changes of orientation due to dislocation motions through the integrated depth of the gauge volume (~ 30μm) are much larger than the angular resolution of the CCD. Depth-resolved profiling, developed by Larson et al, was carried out at these four positions to determine the local Laue diffraction contributed at specific depths beneath the surface.

Results and Discussions

Figure 2 shows the Laue image collected from Grain A after 15% strains. The image is a depth-integrated pattern. The streaking of the patterns indicates the formation of GNDs and GNBs. The Laue reflections for all grains indicate the presence of streaks, however the streaked pattern of each grain is different as it is dependent on the distinct dislocation activities within the grains.

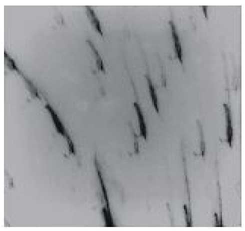

Figure 2. Depth-integrated Laue image for grain A after 15% strains.

Depth-resolved information was obtained by reconstructing depth-specific images collected with the differential-aperture method[2]. The peaks in the reconstructed images from the deformed sample are not as perfect as those found at 0 % strain. This suggests the deformation microstructure is finer than the 1 μm depth resolution. The orientation tensor of each sampling volumes was determined from the reconstructed images. The angular precision is within ~ 0.02° for the undeformed measurements. As the Laue peak shapes deteriorate due to deformation, the angular precision reduces and an average resolution of 0.1° is found for the analysis on the data from the 15 % strain.

The shape of the observed Laue streaks is interpreted by simulations of the diffracted Laue patterns and determines the scalar dislocation density distribution η. Deformation of the grains was described by the formation of GNDs and GNBs. For Ni, an FCC material, edge dislocation lines (ξ) are typically parallel to <112> with <110> Burgers vector (**b**) and [111] glide planes. Depth-resolved images were simulated with a series of slip systems combinations. The slip systems were then determined by comparing the simulations with the experimental results.

Figure 3 depicts the best-fit simulation pattern for grain A at 5μm in depth with **b** // $[01\bar{1}]$ and ξ // $[2\bar{1}1]$. Splitting of "streaked" patterns was observed as depth increased. This indicates that at least two different slip systems combinations are present through the depth of the grains. For grain A, simulations show that combination of two slip systems of **b** // $[01\bar{1}]$ and ξ // $[21\bar{1}]$and **b** // $[0\bar{1}1]$ and ξ // $[\bar{2}1\bar{1}]$ give a better fit to the experimental data.

Acknowledgement

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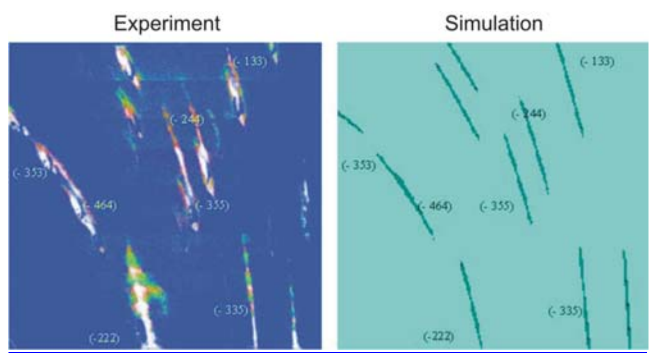

Figure 3. Comparison between experimental and simulated Laue patterns for grain A after 15% strains at the depth of 5μm.

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