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Evolution of deformation structures under varying loading conditions followed *in situ* by high angular resolution 3DXRD

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ABSTRACT

With high angular resolution three-dimensional X-ray diffraction, individual subgrains are traced in the bulk of a polycrystalline specimen and their dynamics is followed *in situ* during varying loading conditions. The intensity distribution of single Bragg reflections from an individual grain is analyzed in reciprocal space. It consists of sharp high-intensity peaks arising from subgrains superimposed on a cloud of lower intensity arising from dislocation walls. Individual subgrains can be distinguished by their unique combination of orientation and elastic strain. The responses of polycrystalline copper to different loading conditions are presented: during uninterrupted tensile deformation, formation of subgrains can be observed concurrently with broadening of the Bragg reflection shortly after onset of plastic deformation. With continued tensile deformation, the subgrain structure develops intermittently. When the traction is terminated, stress relaxation occurs and number, size and orientation of subgrains are found to be constant. The subgrain structure freezes and only a minor clean-up of the dislocation structure is observed. When changing the tensile direction after pre-deformation in tension, a systematic correlation between the degree of strain path change and the changes in the dislocation structure quantified by the volume fraction of the subgrains is established. For obtaining the subgrain volume fraction, a new fitting method has been developed for partitioning the contributions of subgrains and dislocation walls.

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1. Introduction

During plastic deformation of metals, dislocations are stored in the material and organize into heterogeneous dislocation structures characteristic of the material and the deformation conditions. As the formed dislocation structure influences the mechanical properties of the metal, understanding of the origin and evolution of ordered dislocation structures is relevant for predicting their flow stress and work-hardening rate (e.g. [1]).

The dislocation structures formed during deformation of copper have been studied extensively [2–6]. Dislocations organize in ordered structures consisting of almost dislocation-free regions (subgrains) separated by regions of high dislocation density (dislocation walls); the scale of the structure decreases with increasing flow stress. The specific morphology depends on the deformation conditions and the orientation of the crystalline lattice [6]. Changes in the deformation conditions require the dislocation structure to reorganize and adapt to the new deformation conditions, after e.g. strain path changes [7,8]. The processes involved in dislocation structure evolution and adaptation are still not well understood, partly because no experimental techniques have been available to study the dynamics involved. The experiment described in this paper aims to further our understanding of these phenomena by studying the evolution of dislocation structures *in situ* during varying loading conditions.

Traditionally dislocation structures are studied with either transmission electron microscopy [2–6] or classical X-ray line profile analysis [9–13]. Transmission electron microscopy is able to produce high resolution images of the dislocation structures in direct space, but has the disadvantage that bulk grains cannot be studied *in situ* because macroscopic samples must be destroyed to produce the thin foils necessary for transmission electron microscopy. Investigating the evolution of dislocation structures *in situ* in thin foils is not representative for the behavior in the bulk due to image forces from the free surfaces. Classical X-ray line profile analysis has the disadvantage of averaging over many grains as the measured diffraction signal originates from different grains even with different orientations. Recently, two synchrotron radia-

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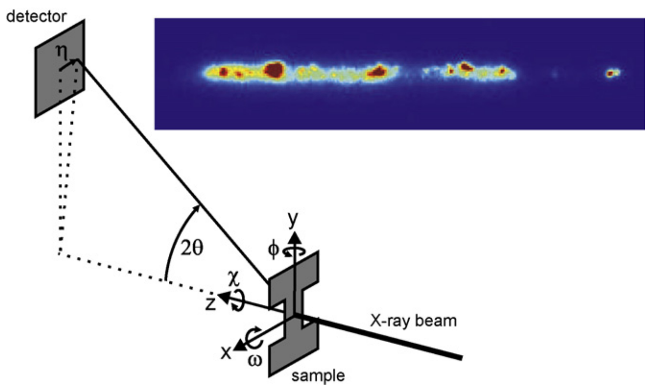

Fig. 1. Sketch of the setup with far distant detector and part of a two-dimensional detector image of a 4 0 0 reflection showing peaks of high intensity superimposed on a low-intensity cloud (from [19]).

tion based techniques for three-dimensional characterization have been established. Both instruments provide bulk information, but the spatial resolution of the 3D X-ray diffraction (3DXRD) microscope [14] does not allow for direct observation of the deformation structure, and the spatial scanning technique of the 3D X-ray crystal microscope [15] limits the possibility of investigating the dynamics of the structural changes. In the present work, high angular resolution 3DXRD [16] is used to study dislocation structures by obtaining high resolution three-dimensional reciprocal space maps of reflections from individual bulk grains in copper while loading (differently pre-conditioned) samples in tension.

2. Experimental technique

2.1. Setup

At beamline 1-ID-XOR at the Advanced Photon Source (APS, Argonne National Laboratory) a special setup for obtaining three-dimensional reciprocal space maps of individual bulk grains with high resolution is established [16]. An X-ray energy of 52 keV gives a penetration depth of about 500 μm in copper, and makes it possible to obtain diffraction patterns from bulk grains. A narrow beam with a height of 25 μm and a width of 30 μm ensures that only a small volume of the sample is illuminated, which is necessary for studying individual grains in order to avoid overlap with reflections from other grains. The narrow beam is conditioned by vertical focusing (giving a vertical divergence of 17 μrad) and horizontal slits. Focusing increases the flux and thereby reduces the acquisition time. High angular resolution is achieved by combining a narrow-bandwidth monochromator ($\Delta E/E = 7 \times 10^{-5}$) and a large sample-detector distance (3.6 m). A two-dimensional detector with a pixel size of 80 μm is mounted above the beam and centered at a height of a 4 0 0 reflection (for copper) as shown in Fig. 1. A two-dimensional image is acquired while rocking (rotating with constant angular velocity) the sample through a small angle interval around the horizontal axis perpendicular to the scattering plane (the ω-axis in Fig. 1). By combining several images a three-dimensional map is obtained where two dimensions are given by the horizontal and vertical directions on the detector and the third dimension arises from subsequent rocking intervals. The resolution of the method is $\sim 1 \times 10^{-3}$ Å^{-1} in the two detector directions (determined by detector pixel size, beam divergence and beam energy spread) and similar in the third direction (determined by the chosen rocking angle interval, $\sim 1 \times 10^{-3}$ Å^{-1} for 0.007° or $\sim 2 \times 10^{-3}$ Å^{-1} for 0.015°). A detailed description of the setup can be found in [17,18].

2.2. Experimental procedure

For *in situ* deformation, tensile samples are mounted in a custom made load frame with a strain gauge glued onto the gauge area. For each sample a suitable grain is selected, and one particular 4 0 0 reflection is mapped at different strain levels as the sample is deformed in tension. A large area two-dimensional detector near the sample (and centered with respect to the primary beam) is used for finding suitable grains which fulfill four criteria [19]: (1) The [4 0 0] direction must be nearly parallel to the tensile axis. (2) The chosen 4 0 0 reflection must be well separated from reflections from other grains. (3) The grain must be completely illuminated by the beam which requires that the grain size is smaller than the beam size. The grain size is estimated by scanning the grain through the beam and measuring the diffracted intensity as function of position. (4) The grain must be in the bulk of the specimen and must not be near the surface. The position of the grain with respect to the sample surface is determined by a simple geometric method. Some grains are excluded because the scan through the beam shows a profile with two distinct intensity maxima indicating the presence of a twin in the grain, others are excluded because their orientation spread is too large which would make the time required for a full mapping too long.

After a suitable grain is selected, the near detector is moved out of the X-ray path, and the sample is oriented so that the 4 0 0 reflection from the grain is observed on the far detector. With this detector two-dimensional images of the 4 0 0 reflection are obtained while rocking the sample around the ω-axis perpendicular to the main scattering plane. When the rocking interval is large enough to cover the entire orientation spread of the grain, smooth intensity distributions are obtained. For smaller rocking intervals, however, a more detailed structure of the Bragg reflection is revealed and the detector images show characteristic features related to the dislocation structure in the grain. As shown for a detector image obtained by rocking over an interval of 0.015° in Fig. 1, the images contain localized areas with high-intensity peaks superimposed on a slowly varying cloud of lower intensity (but enhanced compared to the background). This characteristic feature is observed for all reflections from grains in tensile deformed copper. The high-intensity peaks are produced by almost dislocation-free regions of the crystal (subgrains) while the spread-out cloud is caused by regions with a high dislocation density (dislocation walls) [16,17,20].

Finally, three-dimensional reciprocal space maps are obtained by combining a number of two-dimensional detector images (such as shown in Fig. 1), each acquired by exposing the sample while rocking it through a small interval around the ω-axis.

2.3. Data representation

Such three-dimensional intensity distributions are difficult to visualize, but the information can be conveniently presented in two complementary projections: (i) radial peak profiles and (ii) projections of the intensity distribution onto the azimuthal plane.

Radial peak profiles are obtained by integrating over the directions perpendicular to the diffraction vector (the azimuthal directions). They characterize the intensity distribution along the scattering vector

$$q_y = \frac{4\pi}{\lambda} \sin\theta. \tag{1}$$

The evolution of elastic lattice strains

$$\epsilon = -\frac{\delta q_y}{q_y} \tag{2}$$

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