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Evolution of dislocation density distributions in copper during tensile deformation

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Abstract

The evolution of dislocation storage in deformed copper was studied with cross-correlation-based high-resolution electron backscatter diffraction. Maps of 500 μm × 500 μm areas with 0.5 μm step size were collected and analysed for samples deformed in tension to 0%, 6%, 10%, 22.5% and 40% plastic strain. These maps cover ∼1500 grains each while also containing very good resolution of the geometrically necessary dislocation (GND) content. We find that the average GND density increases with imposed macroscopic strain in accord with Ashby's theory of work hardening. The dislocation density distributions can be described well with a log-normal function. These data sets are very rich and provide ample data such that quantitative statistical analysis can also be performed to assess the impact of grain morphology and local crystallography on the storage of dislocations and resultant deformation patterning. Higher GND densities accumulate near grain boundaries and triple junctions as anticipated by Ashby's theory, while lower densities are rather more spread through the material. At lower strains (⩽6%) the grain-averaged GND density was higher in smaller grains, showing a good correlation with the reciprocal of the grain size. When combined with a Taylor hardening model this last observation is consistent with the Hall–Petch grain size effect for the yield or flow stress.

Keywords: EBSD; Geometrically necessary dislocations (GNDs); Dislocations; Polycrystals; Plastic deformation

1. Introduction

The introduction of grain boundaries through thermo-mechanical processing (solidification, deformation, recovery, recrystallization and grain growth) imparts significant improvements in mechanical strength as described empirically by Hall [1] and Petch [2]. In general, polycrystalline materials consisting of numerous grains with various orientations provide higher strength compared to single-crystalline materials [3] and are utilized in most engineering systems. It is also well known that the generation, movement and storage of dislocations governs plastic deformation [4]. In polycrystals, the nature of deformation at the microscale is controlled by the balance between the local stress state and the local resistance to deformation, which enables either the activation of new dislocation sources or the mobility of existing dislocations. The introduction of interfaces limits the mean free path of mobile dislocations, thus encouraging dislocation–dislocation interactions. Furthermore local force equilibrium and geometrical shape compatibility must be maintained at a grain boundary, a requirement that results in local phenomena such as pile-ups due to strain incompatibilities and to changes in elastic and plastic properties induced by elastic anisotropy and a change in grain orientation.

On a very local scale, all dislocations are necessary. From a mechanics point of view, however, it is useful to classify them following Ashby's descriptions as either statistically stored dislocations (SSDs) or geometrically

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necessary dislocations (GNDs) [5]. In this theory, SSDs are formed due to mutually trapped dislocations existing as forms of dipoles and multipoles, whereas GNDs are generated as excess dislocations within a Burgers circuit to satisfy geometrical compatibility. Ashby proposed that the SSDs in single crystals play a dominant role in work hardening for the more homogeneous deformation of single crystals by acting as obstacles to hinder the movement of additional dislocations on active slip planes. However, for inhomogeneous deformation in polycrystals, GNDs are generated rapidly and at some point will exceed SSD density and thus dominate work-hardening processes.

Ashby's model begins by considering that individual grains in a polycrystal deform independently (i.e. without considering compatibility and equilibrium conditions) and then captures grain–grain interactions by the introduction of GND arrangements to fill-in or hollow-out voids and overlaps that would otherwise be created near grain boundaries [6]. In other words, GNDs are generated to connect the deformation in individual grains within polycrystals so as to ensure the compatibility requirements.

This model has yet to be proven, mostly due to the statistical nature of the problem which requires large numbers of dislocations to be analysed across many grains. The advent of diffraction in the transmission electron microscope has enabled theories of plasticity to be developed due to the direct observation of dislocations (e.g. [4,7]). However, transmission electron microscopy (TEM) is prohibitive for a general statistical study, simply due to time-consuming sample preparation and analysis which limits the number of areas that can be studied. X-ray or neutron diffraction experiments capture the general statistical nature of the problem well (e.g. the evolution of crystallographic texture) but they tend to lack detail about the nature of grain-to-grain behaviour due to their lack of spatial resolution.

There are two developments in this field which may shed more light on the problem, such as recent developments using high-resolution X-ray systems at synchrotron beam lines, either through differential aperture X-ray microscopy to reveal cell structures [8,9], far-field high-energy X-ray microscopy to separate cell interiors and cell boundaries [10], and near-field high-energy X-ray microscopy to generate large 3-D grain data sets with relatively high angular resolution [11]. The spatial resolution of synchrotron X-ray beams has improved to the submicron level and preliminary results show that dislocation cell structures can be investigated. However, a systematic study on plastic deformation in polycrystals has not yet been reported.

The combination of Hough-based electron backscatter diffraction (EBSD) [12,13] for mapping grain orientation and morphology, together with cross-correlation-based EBSD for high angular resolution of GND density, lattice rotation and strain gradients [14,15] provides a tool well suited to this problem. By utilizing electron diffraction in the scanning electron microscope, surfaces of bulk samples can be studied with high spatial resolution (~20 nm) [16] and relatively easy sample preparation. In order to assess deformation in large areas, a recent study by the authors [17] has found that even cross-correlation of highly binned patterns (e.g. 8×8) enables an adequate angular resolution (~0.1°) to reliably recover the stored GND densities in copper strained to 10% under tension. In comparison, no similar study using a Hough-based approach, which has a reduced angular misorientation resolution (1–0.5°) and therefore an increased noise floor [18], has been reported.

Dislocation density can be analysed using EBSD according to Nye's theory [19] in which rotation gradients within a Burgers circuit are linked to the stored GND content. High-resolution (HR)-EBSD measures rotation about three orthogonal axes on the surface of a sample and therefore the production of a map can be used to generate six lattice rotation curvature components. As Pantleon notes, these curvatures can be linked directly to five components of the Nye tensor and one difference [20]. From these six rotation curvature components a physically based L1-based line energy minimization scheme can be used to extract dislocation densities and report a description of the stored dislocation content. Early work using conventional EBSD to measure GND density was pioneered by Adams et al. on bi- and polycrystalline aluminium samples [21,22]. Various plastic deformation problems such as strain gradient increase in the vicinity of indentations [23] and interphase boundaries [24] were addressed using EBSD-measured GND density. Field et al. extended this GND density measurement to a significantly larger number of grains in polycrystalline aluminium [25–27] and steel [28]. Statistical analysis was conducted to study the general behaviour of GNDs. However, due to the relatively low angular resolution (~0.5°) [18], the GND density can only be recovered in moderately and severely deformed samples. A complete description of GND density evolution with strain is lacking as is a rigorous statistical analysis of GND density with respect to microstructural features.

Prior work with HR-EBSD has been limited by the desire to capture high angular resolution information and therefore only small areas have been mapped. Britton et al. presented the first polycrystalline analysis of stored dislocation content with this route and similar maps since have only contained tens of grains [29–31]. This has resulted in limited statistical analysis, as only a few grains could be studied and the statistics on the effects of neighbouring grains was poor.

We present a systematic study of GND density of five samples deformed in tension to 0%, 6%, 10%, 22.5% and 40% plastic strain. In order to capture information concerning dislocation storage with respect to microstructure we have exploited the fact that 4×4 binned patterns provide adequate information for cross-correlation-based EBSD measurements of stored dislocation content [17]. In our previous work, it was found that using a step size of 0.5 μm and 4×4 binning level provides an excellent combination of spatial and angular resolution (~0.03°) and the associated GND density noise floor was estimated

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