Acta Materialia 117 (2016) 35–42

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Geometrically necessary dislocations favor the Taylor uniform deformation mode in ultra-fine-grained polycrystals

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ARTICLE INFO

Article history:
Received 20 March 2016
Received in revised form
27 June 2016
Accepted 28 June 2016
Available online 10 July 2016

Keywords:
Geometrically necessary dislocations
Statistical dislocations
Severe plastic deformation
Ultrafine-grain
Polycrystal plasticity
Misorientation angle distribution

ABSTRACT

The density of geometrically necessary dislocations (GND) obtained from the lattice curvature was studied in commercially pure copper up to extreme large strains (von Mises strain of 63). Its evolution shows an increase to a maximum at a strain of about 2, then decreases until reaching the stationary limiting stage of grain refinement at a von Mises strain of about 14. At the same time, the total dislocation density is also decreasing. It is shown that the variation in the GND density correlates with the difference between the correlated (first neighbor grains) and the non-correlated (random neighbor) misorientation angle distributions. The low quantity of GND at extreme large strains is a consequence of the near Taylor-type homogeneous behavior of the polycrystalline ultrafine-grained structure.

1. Introduction

Material behavior can be described at meso-, micro- and nano-structural levels. The first level is the meso-level – the poly-crystalline nature of the material – which is characterized by the grain morphology and grain orientation distribution. The second level, the micro level, is the microstructure within the grains, determined by the grouping of faults, such as dislocation cells, microbands, disclination groups, etc. At the nano-level, we look into the structure of single dislocations, stacking faults and grain boundary characteristics. When a material is deformed plastically, the structure can change at all levels, especially at extremely large plastic deformations. Thus, it is important to understand the microstructural changes that are taking place when the material is deformed. Here, we aim to capture some deterministic changes due to severe plastic deformation at the meso and micro levels.

As an effective way to change material properties, especially mechanical strength, research into severe plastic deformation (SPD) techniques [1–4] has been accelerated. During SPD, large hydrostatic stresses stabilize the material flow while the end-shape of the workpiece remains constant. In creating a near nano-structured material, the most important feature of SPD is the fragmentation process of the grains which involves building new grain boundaries with increasing misorientations. Such boundaries become necessary because of the increasing difficulties to accommodate the neighboring grains with different orientations. Those dislocations, that are needed for the geometrical accommodation, are called geometrically necessary dislocations (GNDs) as first examined by Ref. [5]. They are part of the total dislocation density (ρ_{total}), which is composed of two parts: GND (ρ_{GND}) and 'statistical' ($\rho_{stat.}$) densities:

$$\rho_{total} = \rho_{stat.} + \rho_{GND} \quad (1)$$

'Statistical' dislocations are statistical in the sense that they do not produce significant misorientation [6]. An example is dislocations that form the walls of the dislocation cell structure. Such a dislocation wall creates misorientations in the range of 0.5° and the sign of misorientation changes from one wall to the next. Such dislocation-cell walls are called incidental dislocation boundaries while the GND walls were called geometrically necessary boundaries in Ref. [6]. The latter can display misorientations larger than

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http://dx.doi.org/10.1016/j.actamat.2016.06.062
1359-6454/

15°. In principle, all individual dislocations are GNDs because they assure a local deformation with respect to their surroundings, so they are geometrically necessary. However, dislocations exist in large number in the material, so groups of dislocations may show collective properties. Some of these properties can be identified by making Burgers circuit that encompass many dislocations. If the net Burgers vector is nearly zero, one can say that that group of dislocations does not show a polarity, while if the net Burgers vector is large, the group is polarized. The polarization means not only a large net Burgers vector but includes significant orientation difference of the group-domain with its surroundings. Lattice curvatures are typically produced by such GND groups. When there is no lattice curvature, statistical dislocations may still exist in large number, without creating net Burgers vectors.

The total dislocation density can be measured by different techniques, such as counting individual dislocations in electron microscopy images, or from broadening of the diffraction peaks obtained by X-ray [7]. The measurement of the GND component is more complex and became available only since mapping of orientations developed as a standard technique through Electron Back Scatter Diffraction (EBSD) [8,9], in Transmission Microscopy by Automated Crystal Orientation Mapping [10–13] or by Transmission Kikuchi Diffraction [14]. Measuring the orientations within small neighboring material volumes enables the calculation of the lattice curvature from which the GND density can be derived using the dislocation density tensor [15,16]. Based on this method, several studies gave information on the quantity of GNDs in deformed metals [17–20]. It has also been shown that the GNDs group into walls, similar to statistical dislocations [16]. GND densities were also estimated theoretically as part of a recent grain fragmentation model and compared to experiments [21]. That modeling is based on the grain-rotation-induced lattice curvature and predicts that the density of GNDs first increases then decreases at larger strains. This effect is due to the texture formation because lattice curvature is reduced when grain orientations approach the ideal positions. The lattice curvature based grain fragmentation model captures only the effect of lattice rotation, while other physical effects that can modify the GND density arising at extreme large strains, for example, dynamic recrystallization with grain boundary movements, were not incorporated.

To date, there is limited understanding of the evolution of the GND density for large strains, although this quantity indicates the degree of heterogeneity in the strain pattern that develops during the large plastic deformation of polycrystals. Knowledge of the plastic strain heterogeneity is fundamental in polycrystal modeling because the evolution of the crystallographic texture depends strongly on the modeling approach. The simplest model is the Taylor approach which assumes homogeneous deformation and, therefore, excludes the existence of GNDs. The most sophisticated polycrystal model is the Self Consistent Viscoplastic (VPSC) model where the grains are allowed to deform differently from the macroscopic strain [22,23]. This model contains a tuning parameter (α), which permits to control the degree of strain heterogeneity. It was introduced by Molinari and Toth [24] in the so-called interaction equation of the VPSC model. When the α parameter is varied, the following polycrystal plasticity approaches can be recovered: $\alpha = 0$: Static (also called the Sachs model), $\alpha = m$: Tangent, $\alpha = 1$: Secant, $\alpha = \infty$: Taylor model (m is the strain rate sensitivity of slip). Using this modeling, recent polycrystal texture simulations showed that, at very large plastic strains, the behavior of the polycrystal approaches the Taylor mode [25–27] because α values of about 20 were needed for the simulations. Therefore, a decrease in the GND density is indirectly predicted. This modeling result is now confirmed in the present report by experimental results obtained by EBSD on copper deformed by SPD. It is also shown that the GND density as a function of large plastic strain correlates with the difference between the correlated (first neighbor grains) and the non-correlated (random neighbor) misorientation angle distributions.

2. Experimental

Copper samples were deformed by High Pressure Tube Twisting (HPTT) [28], Equal Channel Angular Extrusion (ECAE) [1], or rolling; the experimental conditions are summarized in Table 1. The equivalent von Mises deformation ranged from 0.8 to 63. For EBSD measurements, the samples were mechanically ground and polished to 4000 grit using SiC paper, and then electro-polished in a solution of 25% orthophosphoric acid, 25% ethanol and 50% distilled water at 10 V for 30 s at room temperature of ~25°. EBSD was undertaken in a Leo-1530 field emission scanning electron microscope with an operating voltage of 20 kV, a probe current of about 5 mA and working distance of 20 mm. The grain boundaries were identified using a minimum misorientation angle of 5° between adjacent pixels, the obtained GND densities were calculated using the ATOM software [29].

The calculated GND density obtained from EBSD measurement depends on the measurement step size p, i.e. the pixel size. The relation between the density values and p appears almost linear and depends on material and processing conditions. Therefore, an important question is the choice of the step size for GND calculation. For this purpose a reference step size p_{ref} has to be defined to which all measurements (on the same material) can be referenced using a linear relationship. In general, the size of the electron beam is about 20 nm, however, the size of the diffracting area is larger; it can be estimated to be about 50 nm for copper. Therefore, a reference value of $p_{\mathrm{ref}} = 50$ nm was used here for copper and all measurements undertaken with a different pixel size were corrected to this reference pixel size.

The EBSD measurements provide the average crystallographic orientation within a pixel, so that using the orientations of adjacent pixels, the lattice curvature can be calculated. From the lattice curvature, five components of the Nye dislocation density tensor ($\boldsymbol{\alpha}$) can be obtained from 2D mapping [15]: α_{12}, α_{13}, α_{21}, α_{23}, α_{33} (measurement is on plane 3). The GND scalar density can be defined as the entry-wise norm of the Nye dislocation density tensor ($\boldsymbol{\alpha}$) divided by the Burgers vector length:

$$\rho_{GND}^{(2D)} = \frac{1}{b}\sqrt{\alpha_{12}^2 + \alpha_{13}^2 + \alpha_{21}^2 + \alpha_{23}^2 + \alpha_{33}^2} \tag{2}$$

In order to estimate ρ_{GND} for the 3D case, we assume $\boldsymbol{\alpha}$ isotropic and obtain:

$$\rho_{GND} = 3\rho_{GND}^{(2D)}\Big/\sqrt{5} \tag{3}$$

3. Experimental evolution of GND density

The GND densities were calculated from our EBSD measurements according to the procedure presented in Section 2, and are plotted in Fig. 1. These results were obtained for several strain paths, not just for monotonic ones; see Table 1 for sample conditions. Clear trends are observed in the results: the GND density first increases up to a von Mises strain of about 2, then it begins to decrease to reach a constant value at extreme large strains. The decrease is about a factor of 2 with respect to the maximum value.

It is important to compare the evolution of the GND density to the total dislocation density; results obtained by X-ray line profile analysis [30] are also plotted in Fig. 1. Initially the GND density is