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Resolving the geometrically necessary dislocation content in severely deformed aluminum by transmission Kikuchi diffraction

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ABSTRACT

In this paper, severe plastic deformation (SPD) is applied to commercially pure aluminum. Monotonic high pressure torsion (HPT) processing is employed at room temperature, and the microstructure of samples deformed up to an equivalent strain of 50 is investigated by electron backscatter diffraction (EBSD). The distribution pattern and the density of geometrically necessary dislocations (GNDs) are evaluated by examination of transmission Kikuchi diffraction (TKD) maps. Three different methodologies are utilized for assessment of the GND density. It was observed that two distinct stages of grain fragmentation and steady-state occur during processing. During the first stage, a severe grain refinement was observed as the average grain size decreased from ~85 μm to ~1 μm at an equivalent strain of 10. Quantification of the density of dislocations in both deformation regimes showed that, independent of the choice of model, the GND density is greater in the fragmentation stage than in the steady-state stage. This observation was linked with the prevalence of the continuous dynamic recrystallization (CDRX) phenomenon in each stage. Furthermore, a significant presence of GNDs in the steady-state stage was characterized. Formation of microstructure, grain refinement and saturation of grain size are discussed in the light of statistics of GNDs.

1. Introduction

In modern high pressure torsion (HPT), Bridgman's concept is utilized to deform a disk shaped sample by placing it between two anvils and employing torsional straining while a massive compressive load is applied [1]. This deformation technique is a continuous process, meaning that, unlike many other severe plastic deformation (SPD) processes, repetitive reinsertion of the sample is not required during processing [2]. Because of the high hydrostatic pressure, a practically unlimited magnitude of strain can be imposed in a single operation. In this way, bulk nano-structured and ultrafine-grained (UFG) metallic materials can be efficiently manufactured by heavy straining [3,4]. Consequently, the mechanisms driving microstructural evolution at elevated strains can be investigated.

Dependent on the level of deformation, two distinct stages of microstructural evolution can be distinguished in the HPT process: in the first stage significant grain fragmentation is taking place, whereas in the last stage the grain size saturates [5]. During the fragmentation process of the grains, new grain boundaries with increasing misorientations appear. The presence of these boundaries is necessary since they accommodate the developing misorientation across neighboring crystallite volumes [6]. Such boundaries are called geometrically necessary boundaries (GNBs), and a different selection and number of activated slip systems is observed on either side of the boundary. Accordingly, their constituent dislocations are known by geometrically necessary dislocations (GNDs).

Apart from GNDs, statistically stored dislocations (SSDs) contribute to the total dislocation density. While the misorientation angles across GNBs are large with a wide spread, a mutual trapping of SSDs leads to the formation of ordinary cell boundaries, with a relatively small misorientation and a narrow spread [7]. The formation of misorientations is modelled for both kinds of boundaries emphasizing the process of separation of dislocations of opposite sign. It is shown that fluctuations in the density of mobile dislocations lead to the formation of ordinary cell boundaries, whereas, even in the absence of statistical fluctuations, an additional contribution arises from a different activity of the slip

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systems on either side of the GNBs [8].

Since a local deformation field appears within the vicinity of each and every individual dislocation, all single dislocations are geometrically necessary. However, once a network of dislocations is treated as a continuum, a clear trend is observed. At large length scales any net geometric effect that SSDs would have, is counterbalanced by nearby dislocations of opposite sign within the Burgers circuit. The presence of GNDs, however, is characterized by a net Burgers vector, resulting in the gradual increase of lattice curvature and the emergence of an orientation difference [9]. It is believed that wherever non-uniform plastic deformation occurs, the GND density increases, and these additional GNDs contribute to hardening by acting as obstacles to mobile dislocations [10]. Therefore, the GND density may elucidate the heterogeneity of the local strain tensor and its development as a function of imposed strain.

The residual elastic lattice distortion caused by dislocations can be gauged by orientation contrast microscopy [11]. Provided a material model is available that connects the local orientation gradients with the dislocation content, the GND density can be computed. This relationship may be determined by the compatibility assumption of the elastic and the plastic deformation, leading to the fundamental relation of the dislocation theory [12]:

$$\nabla \times \boldsymbol{\beta}^{e} = -\nabla \times \boldsymbol{\beta}^{p} = \boldsymbol{\alpha} \tag{1}$$

whereby $\boldsymbol{\beta}^e$, $\boldsymbol{\beta}^p$ and $\boldsymbol{\alpha}$ are the elastic lattice distortion, the plastic lattice distortion and the Nye tensors, respectively. The Nye tensor operates on a plane normal to produce the net Burgers vector for a circuit on that plane, and is related to the dislocation content of individual slip systems by [13]:

$$\boldsymbol{\alpha} = \sum_{t=1}^{N} \rho^{t} \boldsymbol{b}^{t} \otimes \boldsymbol{\nu}^{t} \tag{2}$$

whereby ρ^t, $\boldsymbol{b}^t$ and $\boldsymbol{\nu}^t$ are the dislocation density, Burgers vector and line vector of each dislocation system t, respectively. A dislocation system, here, is defined as a unique combination of Burgers vector and line vector for a pure edge or screw dislocation. Dependent on the number of resolved terms in the Nye tensor, the crystallographic symmetry and the type of material model, the GND content can be estimated.

Owing to the development of a refined microstructure, often with a high density of crystallographic defects, conventional techniques are less applicable to characterization of SPD processed metals. As opposed to the finest obtainable spatial resolution by EBSD, i.e. 20–50 nm [14], transmission electron microscopy (TEM) can reach a spatial resolution of 2–3 nm [15]. The higher spatial resolution of TEM and the higher angular resolution of Kikuchi diffraction patterns in EBSD are combined in a recently developed technique, called Transmission Kikuchi Diffraction (TKD) [16–18]. In TKD, a TEM foil is used as a specimen in a scanning electron microscope and the Kikuchi diffraction pattern generated by the forward scattered electrons transmitted from the bottom of the foil is used for the indexation of the crystal structure [16]. The spatial resolution of the TKD depends on sample thickness, the atomic number of the material and the energy of the incident electrons [19]. Therefore, for severely deformed metallic samples, an effective characterization tool is offered which provides a significantly better spatial resolution in comparison to conventional EBSD.

In this article, a commercially pure aluminum alloy is deformed by HPT. The deformed samples, up to an equivalent strain of 50, were characterized by EBSD. The number of grains contained in each EBSD scan varied from 10^3 to 10^6 at different strain amplitudes. In order to obtain a better understanding of the deformation mechanisms, TKD technique was utilized. While EBSD served for a global analysis, TKD was used for a local analysis. The GND density is quantified by examination of the TKD maps, on the basis of three different methodologies. Each model is first explained, and the formation of microstructure, the grain refinement and the saturation of grain size are further discussed.

2. Material and sample processing

An initially cold rolled sheet of commercially pure aluminum with 1.2 mm thickness was heat-treated at 500 °C for 30 s, resulting in the formation of a fully recrystallized microstructure with an average grain size of 85 μm. Disks with 15 mm diameter were extracted from the sheet and deformed by HPT processing at room temperature. The torsional straining was applied with a constant rotation speed of 1 rpm, while a nominal pressure of 2.5 GPa was maintained. Revolutions of 1/8, 1/4, 1/2, 1, 2 and 5 corresponding to equivalent strains of up to 50 were imposed. The deformed samples were stored in a freezer at −20 °C before microstructural investigation.

For the EBSD measurements, the finely polished disks were electropolished by A2 Struers electrolyte, under a voltage of 48 V during 20 s at 22 °C. For TKD, the samples were first ground to a thickness of 150 μm and then 3 mm disks were punched out of the sample. The samples were further thinned using a twin-jet electro-chemical polisher in a solution of 1/3 nitric acid and 2/3 methanol, until a central hole appeared, according to well-established procedure for TEM foil preparation. The inner edge of the sample is transparent for the electrons, which corresponds to a thickness between 40 and 80 nm. An EDAX-TSL EBSD system attached to an FEI environmental scanning microscope (Quanta 450 with a field emission gun) was used for both the EBSD and TKD measurements. EBSD was performed at an accelerating voltage of 15 kV, at a working distance of 16 mm and a tilt angle of 70°, whereas the TKD was performed at an accelerating voltage of 30 kV, at a working distance of 6 mm under a tilt angle of −10°. A beam current of 2.3 nA corresponding to FEI spot size #5 (for the final aperture of 30 μm) was used for the EBSD measurement. These parameters allow acquisition of the diffraction patterns in a square scan grid mode with direct indexing, providing acquisition speeds between 50 and 120 patterns per second. The location of observation is on the top surface of the disks, at a constant radius of 3 mm from the center, which corresponds to equivalent strains of 1, 2, 5, 10, 20, and 50. EBSD and TKD measurements were performed with step sizes of 100 and 20 nm, respectively.

The EBSD and TKD data were analyzed with the commercial TSL-OIM data analysis software version 6.1. For TKD, a cleanup procedure of grain confidence index standardization with a grain tolerance angle of 1° was employed on data points. The EBSD data were additionally processed by a grain dilation correction. The number of grains contained in each EBSD scan varied from 10^3 to 10^6, which is sufficient to ensure a statistically representative sample.

3. The GND density evolution: methodology and results

The GND content was assessed based on examination of the TKD maps, shown in Fig. 1. In these maps, boundaries with rotation angles between 2 and 5°, 5–15° and higher than 15° are indicated in red, white and black lines, respectively. A pixel to pixel criterion is used for calculation of misorientations, meaning that the difference between each pixel and its neighbors is gauged. The evolution of the fragmentation process during large plastic deformation can be clearly seen. The emergence of white lines as extensions of red lines, and black lines as extensions of white lines is an indication of gradual misorientation of low angle grain boundaries (LAGBs) to high angle grain boundaries (HAGBs). In the following sections, details of each utilized approach for the GND estimation are presented.

3.1. Lower bound approach

The residual elastic lattice distortion caused by dislocations could be measured by diffraction techniques, and subsequently be used to recover the dislocation content of the material. Eq. 1 provides the material model required for this correlation, leading to the estimation of the Nye tensor. The Nye tensor, however, has at most nine terms while