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Statistical analysis of dislocations and dislocation boundaries from EBSD data

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ABSTRACT

Electron BackScatter Diffraction (EBSD) is often used for semi-quantitative analysis of dislocations in metals. In general, disorientation is used to assess Geometrically Necessary Dislocations (GNDs) densities. In the present paper, we demonstrate that the use of disorientation can lead to inaccurate results. For example, using the disorientation leads to different GND density in recrystallized grains which cannot be physically justified. The use of disorientation gradients allows accounting for measurement noise and leads to more accurate results.

Misorientation gradient is then used to analyze dislocations boundaries following the same principle applied on TEM data before. In previous papers, dislocations boundaries were defined as Geometrically Necessary Boundaries (GNBs) and Incidental Dislocation Boundaries (IDBs). It has been demonstrated in the past, through transmission electron microscopy data, that the probability density distribution of the disorientation of IDBs and GNBs can be described with a linear combination of two Rayleigh functions. Such function can also describe the probability density of disorientation gradient obtained through EBSD data as reported in this paper. This opens the route for determining IDBs and GNBs probability density distribution functions separately from EBSD data, with an increased statistical relevance as compared to TEM data. The method is applied on deformed Tantalum where grains exhibit dislocation boundaries, as observed using electron channeling contrast imaging.

1. Introduction

Dislocations play an utmost important role in many physical phenomena such as plastic deformation, recovery and recrystallization, which are responsible for microstructural evolutions during processing of metals and alloys. Hence, an accurate description of dislocations structures in deformed materials is a key step for understanding, modelling and thus to be able to predict those phenomena and the resulting microstructures.

Since many decades, dislocations are classified into redundant dislocations termed Statistically Stored Dislocations (SSDs) and non-redundant dislocations termed Geometrically Necessary Dislocations (GNDs) [1,2]. Dislocations with cumulative effect allowing the accommodation of the lattice curvature due to the non-homogenous plastic deformation are defined as GNDs [1–3]. Dislocations stored in arrangement which do not lead to a significant rotation of the crystalline lattice (tangles, dipoles …) are SSDs, their net Burgers vector is almost zero [4]. Since each dislocation actually induces a slight lattice curvature at the dislocation local scale, each dislocation could in principle be defined as GND. So, it is clear that the separation of dislocations in SSDs and GNDs strongly depends on the observation scale and on the accuracy of the technique used to measure the crystal lattice disorientations (i.e. misorientation angle).

For materials with medium to high stacking fault energy, dislocations have the ability to change their slip plane, by cross-slip mainly, and thus to acquire a 3D mobility. This mobility allows for forming well-organized dislocation structures. Such non-random dislocation structures have been intensively characterized by Transmission Electron Microscopy (TEM) in many materials, e.g. Cu [5], Al [5–8], Ni [7,9], 304 L austenitic steel [7] and Fe [10]. Two types of dislocation boundaries have been reported: dense dislocation walls with high dislocation density and cell boundaries with lower dislocation density. As disorientation increases with dislocation density, those two types of dislocation boundaries are usually associated with higher and lower disorientations, respectively. Kuhlmann–Wilsdorf and Hansen [11] termed dense dislocation walls as Geometrically Necessary Boundaries (GNBs) and Incidental Dislocation Boundaries (IDBs). The formation of GNBs is deterministic as it results from different slip activities on each side

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of the boundary. The formation of IDBs is stochastic because it results from of statistical mutual trapping of glide dislocations [12]. For a detailed definition of GNB and IDB the reader is invited to refer to [4].

Hughes et al. [8] proposed a distribution function to describe the probability density of the disorientation of IDBs and of GNBs. Later on, Pantleon and Hansen [13] observed that the Rayleigh distribution function, described in Eq. (1), better fits the probability density of the disorientation of IDBs and of GNBs. This was argued with a theoretical analysis [12,13] and based on previous experimental observations for FCC (Cu [5], Ni [7,9], Al [5–8], austenitic steel [7]) and BCC (Fe [10]) materials [6,8].

$$f(\theta) = \frac{\theta}{\sigma_\alpha^2} \exp\left(-\frac{\theta^2}{2\sigma_\alpha^2}\right), \quad (1)$$

with θ the disorientation and σ_α the standard deviation. The mean disorientation of such a distribution can be calculated as:

$$< \theta >= \sigma_\alpha \sqrt{\pi/2}. \quad (2)$$

Fitting experimental IDBs and GNBs disorientation distributions with such a Rayleigh function provides a quantitative description of dislocations structures. In addition the overall disorientation distribution of IDBs and GNBs can be described by a linear combination of those Rayleigh distribution functions [12,13], see Eq. (3). Both contributions can then be retrieved from the overall distribution so that IBDs and GNBs can be quantified (i.e. the disorientation distribution of each can be determined), even if they are not distinguished in the experimental data.

$$f(\theta) = C\left[\frac{\theta_{GNB}}{\sigma_{GNB}^2} \exp\left(-\frac{\theta_{GNB}^2}{2\sigma_{GNB}^2}\right)\right] + (1-C)\left[\frac{\theta_{IDB}}{\sigma_{IDB}^2} \exp\left(-\frac{\theta_{IDB}^2}{2\sigma_{IDB}^2}\right)\right], \quad (3)$$

where θ_{IDB} and θ_{GNB} are the disorientations of IDBs and GNBs respectively, σ_{IDB} and σ_{GNB} are the standard deviation of the Rayleigh distribution for IDBs and GNBs respectively, C and (1-C) are the fraction of GNBs and IDBs respectively. The main advantage of Eq. (3) is that it makes it possible to separate IDBs and GNBs if the experimental data are not distinguished.

However, to our knowledge, this procedure was only applied to experimental data obtained from TEM measurements. The main drawback of TEM is the local nature of analysis; results are very accurate but with poor statistical relevance. The aim of the present paper is to propose a procedure to perform a similar quantitative analysis of dislocations structures using Electron BackScatter Diffraction (EBSD) data. Experimental probability density distributions of disorientations (including both IDBs and GNBs) can be obtained from EBSD maps, provided that a special care is taken to the acquisition settings, notably with regards to the step size. The disorientation probability density distributions of IDB and GNB separately and the fraction of each are then retrieved using Eq. (3), and compared to former trends obtained from TEM data in the literature.

First, the studied material (cold-deformed pure tantalum), the experimental conditions and data are presented. Then dislocation analysis from EBSD data is discussed. The main drawbacks of using EBSD data for quantitative analysis are related to the influence of the measurement noise and of the EBSD mapping step size [14–20]. A method based on the one originally proposed by Kamaya [21], described in the Section 3.1, is applied to reduce those drawbacks. The advantages and limitations of this approach are discussed based on the comparison of quantitative results obtained for a series of samples submitted to an increasing level of strain. A statistical method is then proposed for the analysis of dislocation boundaries from EBSD data.

2. Material and experimental conditions

High purity Tantalum (> 99.995% wt) has been chosen as a model material in this work, to assess the influence of the applied plastic deformation on the development of dislocation structures. Tantalum is a BCC material with Bürgers vector magnitude of 2.86×10^{-10}m. In order to assess the influence of the strain level by performing a single mechanical test, double cone samples [22] were submitted to compression at room temperature (Fig. 1a), which leads to a well-controlled strain gradient along the radius. A finite elements simulation of the compression experiment was done and led to the equivalent plastic strain field presented in Fig. 1b. With the considered dimensions of the double cones samples the maximal equivalent plastic strain obtained at the center is $\varepsilon_{VM} = 0.73$.

A cross-section of the deformed sample (CD-RD plane) was prepared by mechanical polishing up to 4000 grit SiC paper. Then, a colloidal silica solution with an average particle diameter of 20 nm was used for mechanical – chemical polishing. EBSD maps were acquired using a QUANTAX EBSD system from Bruker company (with e-Flash^{HR} EBSD detector and ESPRIT software package), mounted on a Zeiss Supra40 FEG SEM operated at 20 KeV. The EBSD maps were acquired at different places of the sample (shown by white squares on Fig. 1b) with an acquisition step size of 1.41 µm over a rectangular grid of 1.55 mm × 1.16 mm area. This measurement step size was chosen as a good compromise between the need of a statistically relevant measurement area and a spatial resolution adapted to the different microstructures investigated. Deformation substructures go finer and finer when increasing the strain level. Here the choice has been made to keep step size constant for all measured microstructures, to avoid introducing additional artifacts. Since this is a critical parameter when analyzing deformation substructures, this point will be further commented while discussing the relevancy of the approach.

EBSD maps are presented in Fig. 2. It is worth mentioning that special care was paid to the sample preparation of all samples and that exactly the same EBSD acquisition settings were applied for all maps. This is important since the measurement noise level (which also has a great influence on the point-to-point misorientations which will be analyzed in the following) is sensitive to the surface quality and to the acquisition settings. As the Von Mises strain increases from 0 to 0.53, grains develop smooth and then steeper orientation gradients (revealed by continuous color gradients on Figs. 2a-d), without any well-defined substructures visible at this scale. At $\varepsilon_{VM} = 0.73$, intragranular substructures start appearing, as revealed by the localised color changes; grains start fragmenting into smaller features. The size of those features is still much larger than the chosen step size.

Two Electron Contrast Channeling Imaging (ECCI) micrographs obtained on deformed tantalum samples at different magnifications are presented in Fig. 3. Intragranular lamellar structures are observed. Those are made of dislocation boundaries, consisting of IDBs and GNBs.

3. Disorientation gradient calculation from EBSD data

3.1. Presentation of the method

EBSD technique allows measuring the crystal orientation at each measurement point on the sample surface. Hence, the disorientations $\theta_{i,j}$ can be calculated between any two measurement points i and j. The presence of dislocations in a deformed crystal may induce a measurable lattice rotation (hereafter referred to as intragranular misorientation). The contribution of the elastic field to the local misorientations can be considered negligible [23] so that the local misorientation can be directly linked to, or converted