

Determination of dislocation density in an Inconel 600 nickel alloy by XLPA and automated EBSD

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Abstract. There are two widely used examination methods to study microstructure of polycrystalline materials, namely the automated electron backscatter diffraction (EBSD) and the X-ray diffraction line profile analysis (XLPA). Both methods are suitable to determine quality and quantity of crystallographic defects or irregularities within crystalline structures. However, the EBSD method can be used to estimate the density of only geometrically necessary dislocations (GND), while the XLPA method can give the total dislocation density. A novel software was developed for the determination of GND density from misorientations measured between the neighbouring pixels in EBSD images. Therefore EBSD method is a local, while XLPA method is a non-local procedure to determine dislocation densities. In fact, the calculated GND density depends on the applied step size of EBSD measurements, namely GND densities increase with decreasing the step size. So this leads to a question of how large applied step size of EBSD measurements gives a good approximation for the real GND density in a given polycrystalline material.

1. Introduction

In a previous paper [1] dislocation densities were determined through by using both EBSD and XLPA measurements in ferrous lath martensite. However, the ferrous lath martensite morphology differs from the microstructure of usual polycrystalline materials: it has a characteristic multilevel microstructure. A parent austenite grain contains several packets (the group of laths with the same habit plane). Each packet is divided into parallel blocks and a block is further subdivided into laths. The martensite lath is a single crystal of martensite which contains high density of lattice defects, such as dislocations. The size of single martensite laths is very small (typically few micrometers), therefore they cannot be seen clearly by optical microscope. The blocks and packets are separated by high-angle grain boundaries with the misorientation larger than 15°, while between the laths the misorientation angle is smaller (~10°) [2-5].

It was found that the smaller the step size the higher the dislocation density. As an example, for the smallest (35 nm) and largest (500 nm) applied step sizes the dislocation densities were about 6 and 30×10^{14} 1/m², respectively. This tendency was explained by the fine subgrain structure in the laths, revealed by transmission electron microscopy (TEM). TEM pictures demonstrated that the laths consist



of subgrains in the size range between 50 and 100 nm. This was in good agreement with the average value of crystallite size (71 nm) determined by XLP. Since the subgrain size was under 100 nm, the more reliable dislocation density could be acquired with the smallest step size (35 nm).

Nevertheless, in common polycrystalline materials, the microstructure does not contain any smaller units within crystallites and sizes of the crystallites may vary from tens to hundreds of micrometers. Therefore determination of a reliable GND density through EBSD measurements seems to be difficult. In order to study the effect of step size on GND density, EBSD measurements were performed with different step sizes and these results of GND densities were compared the total dislocation density determined by XLP.

2. Experimental material and methods

EBSD and XLP measurements were performed on an EBSD reference sample given and prepared for EBSD measurements by EDAX-TSL. This sample is a 6.35 mm diameter rod made from Inconel 600 nickel alloy with a recrystallized microstructure. Its composition according to our measurements is as follows: Ni ~70%, Cr ~20% and Fe ~10%.

EBSD measurement were carried out on a Philips XL30 scanning electronmicroscope equipped with an EDAX-TSL type EBSD system and an OIM Analysis 6.2 software configuration. X-ray diffraction measurements were performed by a special high resolution double crystal diffractometer using CoK α 1 radiation ($\lambda = 0.1789$ nm).

3. Results and discussion

3.1. Determination of total dislocation density by XLP

The total dislocation density was determined by XLP using the CMWP fitting procedure. The parameters of the microstructure obtained by XLP are shown in Table 1.

Table 1. In this table q is describing the edge/screw character of dislocations, ρ is the dislocation density, m and σ are the median and variance of coherently scattering domain size, and $\langle x \rangle$ is area-weighted mean crystallite size.

ρ [10^{13} m^{-2}]	q [-]	M^* [-]	m [nm]	σ [-]	$\langle x \rangle$ [nm]
3(\pm 1)	2.1(\pm 0.1)	3.0(\pm 0.3)	97	0.28	120(\pm 20)

The area-weighted mean crystallite size is about 120 nm for the sample. XLP is sensitive to the coherently scattering domain size, so not only the high angle grain boundaries are detected, but the low-angle boundaries as well [6, 7]. Subgrains with low angle domain boundaries, as well as dislocation walls are the structures the XLP is sensitive to, in contrast with the EBSD technique which cannot resolve these low angle grain boundaries. This is why the sub-grain size determined by diffraction methods like XLP is always smaller than that determined by EBSD.

XLP yields a total dislocation density of $3 \times 10^{13} \text{ 1/m}^2$ in this sample. As it will be seen later this value is higher than GND values obtained by EBSD. However, it should be noted that the method based on EBSD technique gives only the density of the geometrically necessary dislocations, while XLP provides the total dislocation density (including both geometrically necessary and statistically stored dislocations).

XLP also gives a parameter describing the edge/screw character of dislocations. The experimental value of parameter q is 2.1. Within the experimental error limits it is in good agreement with the arithmetic average of the theoretical values calculated for pure edge and screw dislocations, 1.6 and 2.3, respectively. This observation suggests that the character of the dislocation structure is mixed.

3.2. Methodology of GND density determination from EBSD misorientations

Determination of density of GNDs is possible via mapping the misorientation relations in a certain sample. Orientation of a “point” on the sample being investigated can be measured by EBSD technique. The advantage of this method is that arbitrary areas (e.g. a single grain) can be selected for the measurements thus local dislocation density can be earned. On the other hand, only superficial investigation can be carried out. In this section the main steps of derivation of dislocation density from misorientation relations is described.

Orientation of a lattice point can be characterized by the Euler-angles (ϕ_1 ; ϕ_2 ; ϕ). As EBSD measurements carried out in separate points on the surface of the sample, Euler-angles can be assigned to each measured point. In fact, scanning the surface with the primer electron beam means that orientation (and other properties) are measured in thousands of points on the surface of the sample. In this way, all neighbouring points can be characterized by their orientation deviation, i.e. the misorientation.

Following the guidelines of Pantleon et al. misorientations of adjacent measuring points are calculated in quaternionic representation [8, 9, 10]. From these mathematical objects, Nye’s dislocation density tensor can be determined as the follows.

$$\alpha = \begin{bmatrix} \alpha_{11} & \alpha_{12} & \alpha_{13} \\ \alpha_{21} & \alpha_{22} & \alpha_{23} \\ \alpha_{31} & \alpha_{32} & \alpha_{33} \end{bmatrix} \quad (1)$$

In Eq. 1, α_{ij} are unambiguous functions of the misorientation and the distance between two measuring points, i.e. the resolution of the measurement. From Nye’s tensor GND density can be directly determined via Eq. 2:

$$\rho^* = \frac{1}{b} (|\alpha_{12}| + |\alpha_{13}| + |\alpha_{21}| + |\alpha_{23}| + |\alpha_{33}|), \quad (2)$$

where ρ^* is the dislocation density, b is the length of Burgers-vector (in this case $|b| = 2,49 \text{ \AA}$). Since the scanning is two-dimensional measuring points do not have neighbours in the z -direction (perpendicular to the plane of the sample). Hence, Nye-tensor is an incomplete matrix with five elements, and the dislocation density obtained in this way called – more precisely – quasi-dislocation density. After calculating the local ρ^* values, the global dislocation density for the measured area can be obtained by averaging.

3.3. Determination of GND density by EBSD

In order to calculate GND densities through EBSD measurements, at first a misorientation range should be determined. This range with a higher and a lower limit contains the misorientations considered for calculations. A higher misorientation limit should be avoided for taking into account grain boundaries (as high angle misorientations) and misidentified points (tiny grains or single pixels with high angle boundaries inside a larger grain mostly caused by structural faults). Fig. 1 shows a misorientation distribution for the sample obtained by EBSD with 1.8 \mu m step size. It reveals that the majority of misorientations what can be taken in account to calculate GND densities are smaller than 5° .

Fig. 2 shows a misorientation map of the same EBSD measurement with low ($<5^\circ$) and high ($>5^\circ$) angle boundaries. (This kind of map shows misorientation of each point with different colours.) It reveals that low angle boundaries can be seen only within the individual grains, while the majority of high angle boundaries can be seen between the adjacent grains (as high angle boundaries) and inside the grains as twin boundaries or misidentified points. Therefore, only misorientations smaller than 5° can be used for determination of GND density.

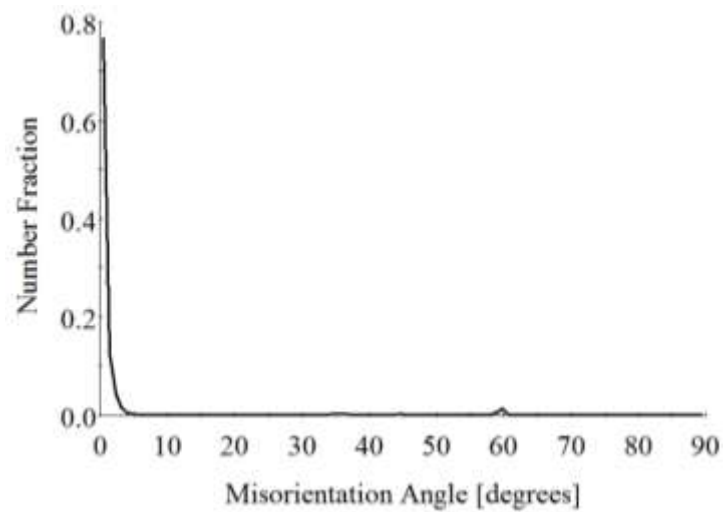


Figure 1. Misorientation distribution of the sample obtained by EBSD with 1.8 μm step size.

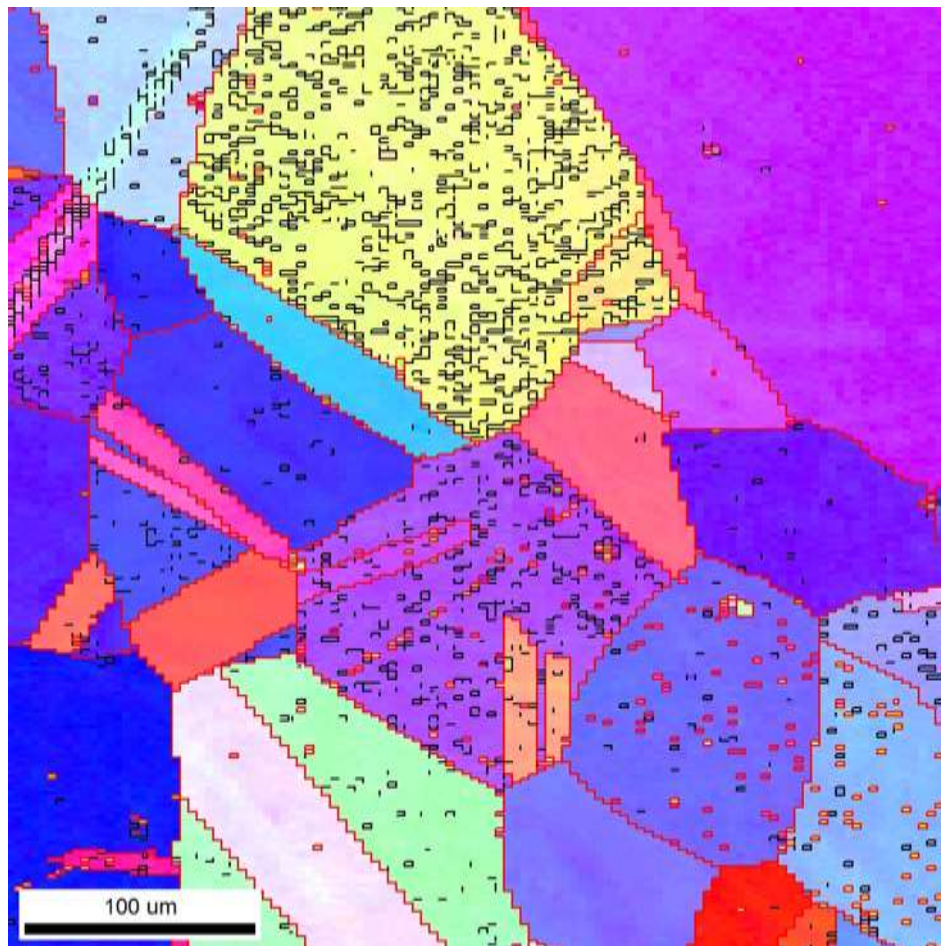


Figure 2. Misorientation map of the sample obtained by EBSD with 1.8 μm step size with low ($<5^\circ$, black lines) and high ($>5^\circ$, red lines) angle boundaries.

However, there is an uncertainty in the determination of the Euler angles from the Kikuchi patterns obtained by EBSD which can result an error in the calculation of the dislocation density from the misorientations. In order to estimate the error in the misorientation angles the same measurements were

carried out on an undeformed single crystalline Si wafer. In this single crystal orientation does not change, therefore differences between the crystallographic orientations of the neighbouring EBSD image pixels should be practically zero.

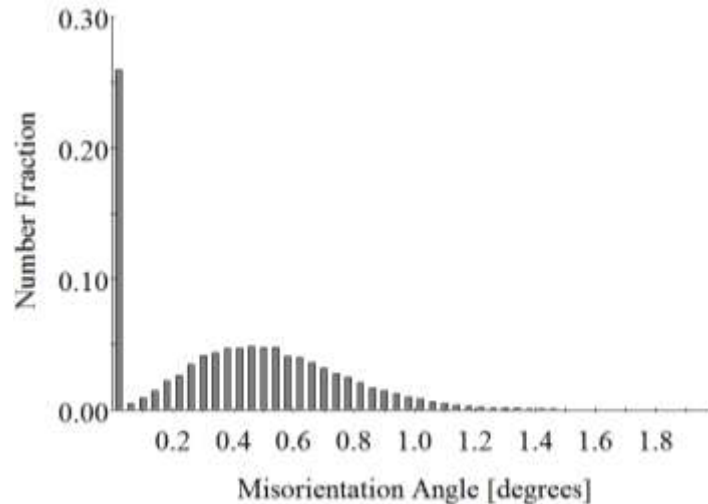


Figure 3. Misorientation distribution of a Si single crystal obtained by EBSD with 2.0 μm step size.

Fig. 3 shows the misorientation distribution for this Si single crystal obtained with an applied step size of 2.0 μm . This distribution reveals that certain misorientations can be observed under 1.5° even if the material is practically defect-free.

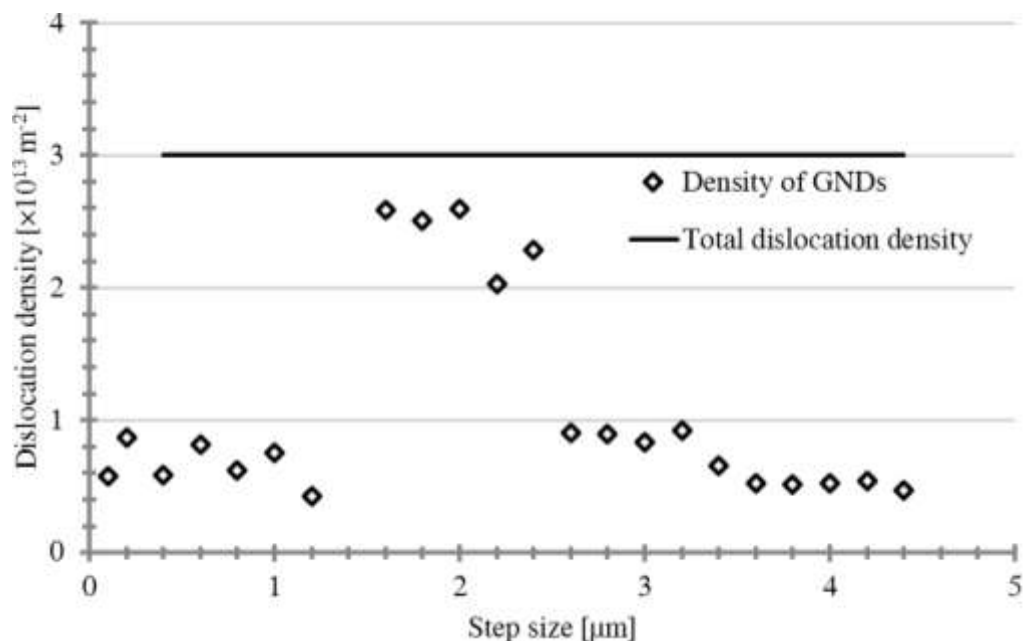


Figure 4 Density of GNDs (and total dislocation density) versus applied step size.

Therefore, misorientations higher than 1.5° can only be used in the calculation of the dislocation density. The other thing why a lower limit should be applied is the high fractions of small misorientations under $\sim 0.1^\circ$ (see Fig. 5) what would increase unreasonably the calculated dislocation densities.

Namely, if both misorientations between a pixel and its two neighbours (at the right side and below the studied pixel) are smaller than 1.5°, the dislocation density is taken as zero in that pixel. If any of the two misorientations is higher than 5°, the pixel is ignored from the dislocation density calculation. For all other pixels the dislocation density was calculated from the misorientations, as described in the

section above. Fig. 4 shows the calculated dislocation densities determined for the whole studied area as a function of applied step size calculated from EBSD data.

It shows that calculated densities of GNDs are between 0.5 and $1 \times 10^{13} \text{ 1/m}^2$ up to $\sim 1.2 \text{ }\mu\text{m}$ step size, then grow up to $\sim 2.5 \times 10^{13} \text{ 1/m}^2$ and decreases to $\sim 0.5 \times 10^{13} \text{ 1/m}^2$. The small calculated dislocation densities at $< 1.5 \text{ }\mu\text{m}$ step sizes can be understood from the Fig. 5. It reveals that while the majority of misorientations are under 1.5° at $4.4 \text{ }\mu\text{m}$ step size, until then almost all the misorientations are smaller than 1.5° at $0.4 \text{ }\mu\text{m}$ step size. The smaller the step size, the more number fraction of misorientations will be smaller than 1.5° , therefore they are ignored during calculation of GND density, thus it will also be small.

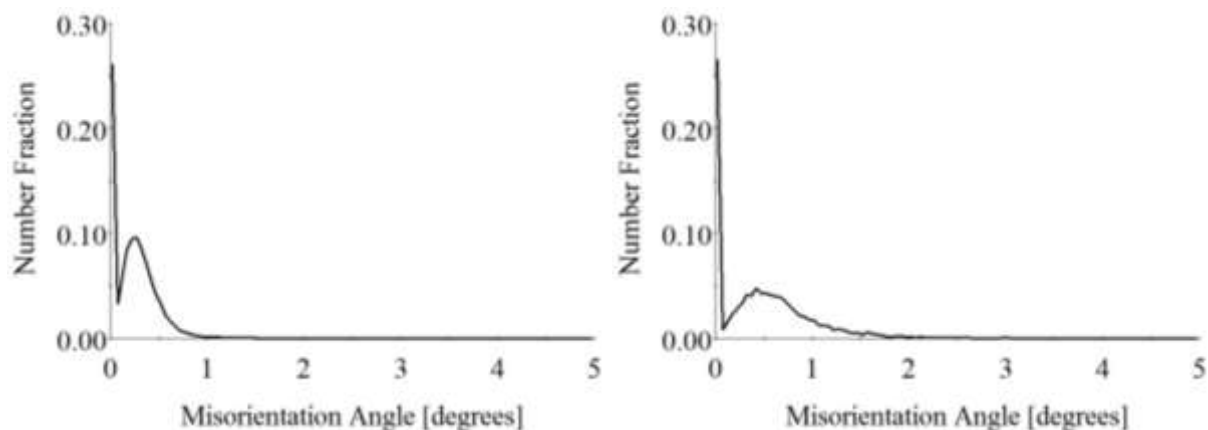


Figure 5. Misorientation distribution for the Ni sample obtained by EBSD with 0.4 (left) and $4.4 \text{ }\mu\text{m}$ (right) step size.

Fig. 4 also reveals that the calculated dislocation densities are smaller ($\sim 0.5 \times 10^{13} \text{ 1/m}^2$) at larger (2.4 - $4.4 \text{ }\mu\text{m}$) step sizes. This is because the larger the step size the more misorientations are omitted for calculating GND densities during scanning. Accordingly, it seems that step sizes between 1.6 and $2.4 \text{ }\mu\text{m}$ would give reliable results for GND density in a common polycrystalline material. These GND densities are smaller (70-85%) than the total dislocation density and their values are close to it.

4. Conclusions

Regarding our experimental results contradictions are to be observed. On the one hand, they are appropriate from the point of view that calculated GND densities are smaller than the total dislocation density at every step sizes. On the other hand, however, based on these measurements it is difficult to specify an optimal step size for calculating correct GND density in common polycrystalline materials. Two main reasons can be considered as explanations for that:

- In fact, the total dislocation density ($3 \times 10^{13} \text{ 1/m}^2$) in the sample examined by XLPA is very low. A sample with much more total dislocation density (due to cold working) should be more suitable for this kind of measurements. However, it would make sample preparation for EBSD to be difficult.
- It seems to be another problem that the uncertainty in the determination of the Euler angles from the Kikuchi patterns (1.5° , determined by EBSD on Si single crystal) is too large. Especially, when it is compared to the upper limit of calculation (5°). It can be as high as 30% of that. It means that a major proportion of the misorientations smaller than 5° should be ignored during determination of GND density.

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