1. Introduction

Material behavior can be described at meso-, micro- and nano-structural levels. The first level is the meso-level — the polycrystalline 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 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 ($\rho_{\text{total}}$), which is composed of two parts: GND ($\rho_{\text{GND}}$) and 'statistical' ($\rho_{\text{stat}}$) densities:

$$\rho_{\text{total}} = \rho_{\text{stat}} + \rho_{\text{GND}}$$

'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...
was introduced by Molinari and Toth [24] in the so-called inter-

density is indirectly predicted. This modeling result is now

tates developed as a standard technique through Electron Back

Scatter Diffraction (EBSD) [8,9], in Transmission Microscopy by

tions undertaken with a different pixel size were cor-

measurements undertaken with a different pixel size were cor-

as the entry-wise norm of the Nye dislocation density tensor (3).

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
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 calcula-
tion. 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 \( p_{ref} = 50 \text{ nm} \) was used here for copper and all
measurements undertaken with a different pixel size were cor-
rected 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
(\( \alpha \)) can be obtained from 2D mapping [15]: \( \alpha_12, \alpha_{13}, \alpha_{21}, \alpha_{23}, \alpha_{31} \)
(measurement is on plane 3). The GND scalar density can be de-
developed as a standard technique through Electron Back

Scatter Diffraction (EBSD) [8,9], in Transmission Microscopy by

Scattering and 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 move-
ments, were not incorporated.

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GND density for large strains, although this quantity indicates the
degree of heterogeneity in the strain pattern that develops during
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plastic strain heterogeneity is fundamental in polycrystal modeling
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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
(\( \alpha \)), which permits to control the degree of strain heterogeneity. It
was introduced by Molinari and Toth [24] in the so-called inter-
action equation of the VPSC model. When the \( \alpha \) 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 a 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 dif-
fERENCE 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 pol-
ished 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 \(-25^\circ \text{ C} \). EBSD was un-
dertaken in a Leo-1530 field emission scanning electron micro-
scope 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
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(\( \alpha \)) can be obtained from 2D mapping [15]: \( \alpha_{12}, \alpha_{13}, \alpha_{21}, \alpha_{23}, \alpha_{31} \)
(measurement is on plane 3). The GND scalar density can be de-
developed as the entry-wise norm of the Nye dislocation density tensor (\( \alpha \))
divided by the Burgers vector length:

\[
\rho_{\text{GND}}^{(2D)} = \frac{1}{5} \sqrt{\alpha_{12}^2 + \alpha_{13}^2 + \alpha_{21}^2 + \alpha_{23}^2 + \alpha_{31}^2} \tag{2}
\]

In order to estimate \( \rho_{\text{GND}}^{(3D)} \) for the 3D case, we assume \( \alpha \) isotropic
and obtain:

\[
\rho_{\text{GND}} = 3 \rho_{\text{GND}}^{(2D)} / \sqrt{5} \tag{3}
\]

3. Experimental evolution of GND density

The GND densities were calculated from our EBSD measure-
ments 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 condi-
tions. 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
nearly as high as the total density, meaning that most dislocations are GNDs. At larger strains the total dislocation density becomes twice as high as the GND. However, at a strain of about 4, the total dislocation density decreases and levels off. The decrease in the total density is nearly equal to the decrease in the GND density, suggesting that the origin of the decrease in total dislocation density can be related to the decrease of the GND density.

4. Misorientation distributions and their relation to GNDs

The orientation difference between neighboring grains can be represented by the angle of misorientation, which is the smallest rotation angle that can bring one crystal orientation into the other. If there is no orientation preference between neighboring grains, and the orientation distribution of the polycrystal is random, the misorientation angle distribution is represented by the so-called Mackenzie distribution [31] (Fig. 2) for cubic crystals. Deviations from this distribution can arise for two reasons:

i: a possible non-random orientation distribution of the grains of the polycrystal, or

ii: an orientation correlation is building up between neighbor grains.

The first effect is due to the evolution of the crystallographic texture, while the second is observed during plastic deformation due to the strain heterogeneities that necessarily exist in a polycrystal [32]. Grain fragmentation during large plastic strain is also a mechanism that produces correlations between neighboring grains because the orientations of the fragments of a grain are necessarily related to the mother grain.

When a polycrystal is deformed, strain heterogeneities appear within the grains, mostly near the grain boundaries. They are necessary to accommodate the deformation between neighboring grains. The strain heterogeneity manifests itself by lattice curvatures leading to large orientation differences within the same initial grain. As the original grain boundaries become unidentifiable after a certain strain, the grains of the polycrystal have to be redefined; EBSD orientation imaging is usually employed for this purpose.

4.1. The next-neighbor grain-to-grain misorientation distribution

In order to determine grain boundary misorientations, the

<table>
<thead>
<tr>
<th>Identification number</th>
<th>Deformation mode</th>
<th>Number of maps used</th>
<th>Note</th>
<th>Eq. von Mises strain</th>
<th>Detected grain number</th>
<th>Step size in EBSD [(\mu m)]</th>
<th>Grain Size [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 ECAE 2 1 pass route A 1.15</td>
<td>1 pass route A 1.15</td>
<td>20,225</td>
<td>0.3</td>
<td>1.15 855–2388</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 ECAE 3 4 pass route A 4.60</td>
<td>4 pass route A 4.60</td>
<td>988</td>
<td>0.1</td>
<td>26,454 667–1432</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3 ECAE 1</td>
<td>2 pass route A 2.30</td>
<td>4773</td>
<td>0.2</td>
<td>459 + 487 + 291 388–695</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 ECAE 1</td>
<td>4 pass route A 4.60</td>
<td>12,475</td>
<td>0.2</td>
<td>390–910</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 HPTT 2 30° exterior 0.86</td>
<td>1.73</td>
<td>3471</td>
<td>0.1</td>
<td>480–546</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6 HPTT 3</td>
<td>42,957</td>
<td>0.1</td>
<td>30° middle 2.31</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7 HPTT 2</td>
<td>29,519</td>
<td>0.1</td>
<td>30° interior 3.75</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 HPTT 2</td>
<td>27,918</td>
<td>0.1</td>
<td>60° middle 4.62</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9 HPTT 2</td>
<td>30° interior 27,111</td>
<td>0.1</td>
<td>7.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 HPTT 2</td>
<td>36,388</td>
<td>0.1</td>
<td>500° exterior 14.43</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11 HPTT 1</td>
<td>12,814</td>
<td>0.046</td>
<td>500° interior 62.35</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12 Rolling 1</td>
<td>36,159</td>
<td>0.048</td>
<td>80% rolling 0.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13 Rolling 1</td>
<td>365</td>
<td>0.2</td>
<td>50% rolling 0.135</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14 Rolling 2</td>
<td>400–910</td>
<td>0.1</td>
<td>90% rolling 2.68</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>15 Rolling 2</td>
<td>253–300</td>
<td>0.08</td>
<td>1293</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>16 Rolling 2</td>
<td>153–189</td>
<td>0.08</td>
<td>350–930</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
'ATOM' software [29] was employed to obtain the Next Neighbor Misorientation Distribution (NNMD) [33]. First, the misorientation angle of each pixel with its neighbors was examined and when it exceeded the 'grain tolerance angle'; (set here to $5^\circ$), a boundary was defined between the two pixels. Once all pixel-based boundaries were defined, a flood-fill procedure was applied to search for sub-areas delimited by a closed boundary; such sub-areas are defined as grains. Their average orientations are defined by Fig. 2. Disorientation distributions between neighbor grains as a function of large strains for commercially pure copper. The sample numbers refer to the experimental conditions in Table 1.
averaging over the orientations of the pixels composing the grain. Finally, the NNMD between neighboring grains was constructed using these average grain orientations.

There is a clear evolution of the NNMD during large plastic deformation, which is well documented for some materials, such as copper and aluminum, see e.g. in Refs. [34,35]. In the initial stage, a large peak appears at lower misorientation angles. At a later stage more and more grains develop large angle misorientations. Finally, at extremely large strains, the distribution approaches the Mackenzie one [34]. For a given material and constant strain path, the evolution of the NNMD can be used to estimate the plastic strain imposed on the material [34]. We found, however, that the NNMD does not become identical to the Mackenzie distribution and a constant difference can usually be seen. This difference can be attributed to the texture of the material which also causes a difference from the Mackenzie distribution, even in the case when there is no strain heterogeneity.

4.2. The non-correlated misorientation distribution

At a given deformed state of the polycrystal, one can obtain the experimental NNMD. The NNMD can be called a “correlated” misorientation distribution because it is constructed from first neighbor misorientations. One can also calculate misorientation distribution between grains that are not neighbors. Such a misorientation function was introduced by Cottam and Curtis [36] and can be called a random pair misorientation distribution (RPMD). It can be calculated from the orientation distribution function (ODF) of the polycrystal by computing the misorientation of a given grain with other grains that are chosen randomly. For this purpose 3000 grains were selected from the measured texture of the polycrystal. When calculating the RPMD it is necessary to consider that a given grain can have around 10–14 neighboring grains, so the construction of the RPMD involved the random selection of 12 grain orientations.

Fig. 2 shows the NNMDs and the corresponding RPMDs for six deformation states of the material; at von Mises equivalent strains of 0.86, 1.15, 3.7, 4.6, 14.4 and 62.4. For the two lowest strains, the RPMDs are similar to the Mackenzie distribution, however, for the very large strains the RPMD are significantly different. The difference is accentuated at high misorientation angles, where the RPMD has higher intensities compared to the random case. This is due to the texture of the material; some of the main ideal orientations of shear textures are actually 60° away from each other (for example; the A/A, and B/B ideal orientations [37]).

4.3. The relation between GNDs and the misorientation functions

If a random pair misorientation distribution is produced by plastic deformation, there is no interaction between grains; any orientation can be a neighbor for a given grain, and thus, no accommodation is needed for a grain to be neighbor to any other grain. Consequently, there are no GNDs; the deformation of the polycrystal can start without GNDs. It is then plausible that the GND density is related to the difference between the NNMD (correlated distribution) and the RPMD (non-correlated distribution) of the polycrystal.

Let \( N(g) \) be the next neighbor misorientation distribution (NNMD) and \( R(g) \) the corresponding RPMD. For quantifying the difference between NNMD and RPMD, a scalar quantity is defined as follows:

\[
R = \sqrt{\int [N(g) - R(g)]^2 \, dg} \quad (4)
\]

Both \( N(g) \) and \( R(g) \) are normalized density probability functions (their integral is 1). \( R \) is plotted as a function of the deformation in Fig. 1. Initially it shows an oscillatory evolution, then it decreases monotonically to a value 10 times smaller than initially. Interestingly, the variation is very similar to the evolution of the GND density. This effect will be discussed in more detail in the following section.

5. Discussion

As deformation begins the GND density is increasing, which is understandable because by increasing strain, the magnitude of the deformation heterogeneity increases, which requires more and more GNDs. What is not expected, however, is that the GND density will arrive at a maximum and then decreases at very large strains. The magnitude of the decrease is about a factor of two, which is very significant. Finally, the GND density arrives at a limiting constant value. It is important to point out that the precision of the EBSD measurement makes the limiting GND value quite uncertain, so even a higher rate of decrease could be occurring.

The decrease in GND density can be related to the grain fragmentation process that takes place at large plastic strains. The main mechanism for grain fragmentation is the grouping of GNDs into walls. The misorientation angle of these walls progressively increases with strain because more and more GNDs are pumped into the walls by the ongoing plastic deformation. When this angle is sufficiently high, the GND wall becomes the boundary of two new grains. Therefore, the GNDs forming this wall cannot be further counted as GNDs. This effect leads to a decrease in the GND density.

Another reason for a significant decrease in the GND density can be that the formation of GND walls is taking place in such a manner that the degree of heterogeneity decreases. The driving force for such a self organization phenomenon is the expected decrease of the total enthalpy of the system, similar to the formation of dislocation cell structures by statistical dislocations. This self organization process then progressively leads to the elimination of the deformation heterogeneities that initially exists between neighboring grains, i.e. changing the deformation mode from heterogeneous to more and more homogeneous behavior. This was also found by polycrystal plasticity simulations in Refs. [25–27] where it was shown that, in the ultrafine grain size regime, the polycrystal behavior approaches uniform Taylor behavior.

The low value of the GND density in the limiting stage does not mean that there is no need for GNDs at large strains. Indeed, in this stage the average grain size remains constant, see Fig. 1 and Ref. [38] and the grain shapes do not follow the imposed deformation, rather they are observed to be only slightly elongated [38] or equiaxed. The evolution of grain shape is illustrated in Fig. 3 for increasing strain. At a von Mises strain of 1.1, the shape follows that expected from the macroscopic deformation (ECAE, one pass). At a strain of 4.6, the shape is much more elongated, however, to lesser extent than expected after four passes in ECAE. Arriving at a von Mises strain of 14.4, the grain size becomes very small and the grains appear equiaxed, which is unexpected. This situation is maintained for larger strains; a steady state develops. This means that a continuous dynamic recrystallization (DRX) process is taking place meaning that grain boundaries are actually moving in the steady state. This leads to an increase in the size of certain grains. When they become too large, a subdivision process will start by new emerging GND walls in these grains. In the limiting stage of grain subdivision, the increase in grain size by dynamic recrystallization is compensated by the grain subdivision process. The first process is removing GNDs while the second is increasing their density, so an equilibrium state in grain size becomes possible.

The occurrence of DRX is evident from the grain shape
arguments. Starting from a strain of about 1, the average grain size decreases by the formation of subgrains that have misorientations more than 5° with respect to their mother grains. By further straining the grain size decreases continuously. This mechanism is a grain subdivision process which can be considered as a continuous DRX (called CDRX in Ref. [39]). In CDRX, new grain boundaries are made by grouping of GNDs. During CDRX grain boundary motion is also possible, especially towards such neighboring grains where the dislocation density is larger. Indeed, a neighboring grain can have a large dislocation density for two reasons. One is the difference in orientation which requires a larger number of active slip systems and hence a larger dislocation density. Another reason for higher density can be that a neighboring grain is not fully compatible with its next neighbor for the imposed deformation, so large GND density develops in that grain. Such grains are candidates for elimination by the moving grain boundaries. The result of this process is not only a change in the grain shape but also a decrease in the average GND density. In this mechanism the driving force is the energy gain due to the elimination of GNDs. Grain boundary motion can also be stress driven, which is indeed very high in the limiting stage of grain fragmentation because of the ultra fine grain size. In stress driven grain boundary motion, the grain boundary moves perpendicular to the applied shear stress. Such a mechanism decreases the aspect ratio of the grain, which can compensate for an increase due to plastic deformation induced shape change.

The misorientation density function between neighboring grains is extensively used in this work. Correlated and uncorrelated versions of these distributions were calculated and shown to be effective in linking them to the GND density; the difference between the correlated and uncorrelated misorientation distributions (called $R$) vary in the same way as the GND density (Fig. 1). This newly found property of the misorientations can be useful in future studies.

As discussed above, the large decrease in the GND density was related to the CDRX process that can take place at large strain by subdivision of grains and by moving grain boundaries. In a previous study of the misorientation distribution it was shown that the maximum intensity of such distribution correlates with the distance between the centers of the neighboring grains for plastic deformation [32]. A unique power law was found with a common exponent of about $-2.0$ between these quantities, independently of material and level of strain. It was also shown in Ref. [32] that this power law disappears if the material is recrystallized. Therefore, it becomes that the material state can be identified by carrying out such a misorientation analysis of the microstructure. The same analysis was then applied to the present case for four levels of deformation of our copper material; at von Mises strains of 1.15, 4.62, 14.43 and 62.35. The result is displayed in Fig. 4. As can be seen, for the two lower levels of deformations, the same exponent was obtained as in Ref. [32]. However, for the larger strains, von Mises strains of 14.43 and 62.35, the power law is not valid, instead of a straight line a curved line appears which is characteristic of static recrystallization (see in Ref. [32]). Indeed, such large strains were not studied in Ref. [32]. In static recrystallization of copper, new grains appear with high grain boundary motion. Therefore, this analysis shows that starting from a von Mises strain of about 14, a similar DRX phenomenon is taking place in the copper material leading to a constant nearly equiaxed grain structure, as it is shown in Fig. 3.

Finally, it is interesting to note that the grain subdivision process is taking place in such a way that the grain-to-grain misorientation correlation is perfectly transforming at extreme large strains and approaching the random case. The meaning of this result is that the DRX process is taking place in such a manner that grains that have higher GND densities (because of their incapacity of similar deformation as their neighbors) will be eliminated by the moving grain boundaries. Therefore, the DRX process is taking place in a selective way and the grain boundary speed is larger in the direction of grains that are badly accommodating to their neighbors. This process leads to a low level of GND density and a more
homogeneous deformation of the polycrystal, as was predicted in earlier polycrystal deformation modeling [25–27].

6. Conclusions

This paper is the first report on the evolution of GND density at very large strains in commercially pure polycrystalline copper and reaches the following conclusions:

1. With increasing plastic strain, the GND density first increases to a maximum during large strain. In the first stage, the dislocation population is predominantly composed of GNDs. After the maximum there is a progressive decrease towards a constant value at extremely large strains.

2. The large decrease of the GND density can be related to the grain fragmentation process taking place at large strains. A two stage dynamic recrystallization process forms the microstructure at large strains: Initially the grain fragmentation process takes place by continuous DRX which ends at a steady state where the average grain size remains constant. In this stage the DRX process is characterized by the moving of grain boundaries together with an ongoing grain fragmentation process.

3. The grain-to-grain misorientation distribution evolves towards the so-called non-correlated one at extreme large strains, which is the same as obtained for random orientation pairs.

4. A new scalar quantity was defined as the difference between the correlated and non-correlated misorientation distributions of the deformed polycrystals and was shown to vary in a manner similar to the GND density.

5. The GND decrease leads to a reduction of heterogeneities in the local plastic deformation involving a change to a near homogeneous Taylor type behavior of the polycrystal in the limiting stage.

Acknowledgements

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