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THE SCALING OF MISORIENTATION ANGLE DISTRIBUTION AT STRAIN-INDUCED BOUNDARIES IN COPPER DEFORMED BY TENSION UNDER VARIOUS CONDITIONS

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Abstract. In the paper, polycrystalline copper deformed by tension under different conditions of loading has been studied using electron backscatter diffraction. The microstructure of areas located on the longitudinal section of the specimens deformed until fracture was examined. The fragmentation of initial grains in case of deformation at room temperature was observed whereas at 400°C, considerable dynamic recovery and recrystallization significantly influenced the microstructure formation. A procedure for computer analysis of the orientation maps has been put forward, which allows separating recrystallized regions from the non-recrystallized ones and further analyzing the misorientation statistics of strain-induced boundaries. A scaling behavior of the strain-induced misorientation distributions was shown to take place. The mechanism of strain-induced boundary evolution was proved to remain unchanged for all studied deformation conditions, in spite of recovery and recrystallization occurring at elevated temperatures.

Keywords: polycrystalline copper, plastic deformation, recrystallization, microstructure, electron backscatter diffraction

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МАСШТАБНАЯ ИНВАРИАНТНОСТЬ РАСПРЕДЕЛЕНИЯ УГЛОВ РАЗОРИЕНТИРОВКИ НА ГРАНИЦАХ ДЕФОРМАЦИОННОГО ПРОИСХОЖДЕНИЯ В МЕДИ, ДЕФОРМИРОВАННОЙ РАСТЯЖЕНИЕМ В РАЗЛИЧНЫХ УСЛОВИЯХ

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Аннотация. В работе исследована поликристаллическая медь, деформированная растяжением в различных условиях (использован метод дифракции обратнорассеянных электронов). Проанализирована микроструктура участков, расположенных на продольном сечении образцов, деформированных до разрушения. В случае деформации при комнатной температуре, наблюдали фрагментацию исходных зерен, тогда как при 400 °C на формирование микроструктуры значительное влияние оказывали динамический возврат и рекристаллизация. Нами предложена процедура компьютерного анализа ориентационных карт, позволяющая отделять рекристаллизованные области от нерекристаллизованных и анализировать статистику разориентировок на границах деформационного происхождения. Доказано, что механизм эволюции разориентировок остается неизменным для всех изученных условий деформации, несмотря на динамический возврат и рекристаллизацию при повышенной температуре.

Ключевые слова: поликристаллическая медь, пластическая деформация, рекристаллизация, микроструктура, дифракция обратнорассеянных электронов

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Introduction

During plastic deformation, a significant number of dislocations remain trapped inside original grains, leading in medium to high stacking fault energy metals to the formation of multiple strain-induced boundaries [1, 2]. These boundaries have low misorientation angles at an early stage of the microstructure evolution. The level of misorientation, however, continuously increases with strain so that high-angle boundaries appear at later stages [2, 3]. The distribution of boundary misorientation angles is important for evaluation of strength properties [4 – 7]. Besides, it can provide important evidence concerning the physical mechanism of the grain refinement during deformation [2 – 4].

The exhibition of microstructure evolution depends, in particular, on the deformation temperature. During low-temperature deformation, the phenomenon called fragmentation [2] takes place, namely, the subdivision of original grains into volumes, which mutual misorientations gradually increase in the process of deformation. An increase in temperature promotes dynamic recovery leading to a reduction of dislocation density and formation of lower-energy dislocation substructure [1]. With a further rise in temperature, a dynamic recrystallization (DRX) occurs: both the discontinuous DRX, which involves the formation of new grain nuclei and their subsequent growth at the expense of surrounding substructure, and the continuous DRX, when the new fine grains develop without the nucleation stage by a gradual increase in subgrain misorientations [7 – 9]. At the same time, it remains unclear whether temperature increasing influences the mechanism of grain subdivision [9].

A scaling behavior in the boundary misorientations has been found by D. A. Hughes and co-authors [3]: it turned out that the misorientation angle-distribution determined at various strains was invariant with respect to the average misorientation angle. Such a scaling has a physical significance, since it indicates that a physical mechanism remains unchanged when changing external conditions.

In the present study, this approach has been used in order to clarify to what extent a change in the conditions of deformation influences the mechanism of grain subdivision. In this concern, the misorientation distribution of strain-induced boundaries were examined in polycrystalline copper deformed in tension under various conditions.

Materials and methods

Cylindrical copper specimens were tensile strained until fracture in three ways:

(*i*) at a strain rate of $3{\cdot}10^{-2}$ s^{-1} at room temperature (specimen I),

(*ii*) at the same strain rate but at 400 °C (specimen II),

(*iii*) under a constant stress of 120 MPa at 400 °C (specimen III).

The fracture of specimen III happened after half an hour of deformation. For the following examination, the necked specimen has been cut along the tensile axis direction, and regions for the Electron Backscatter Diffraction (EBSD) analysis were chosen in several places within the neck, on the longitudinal section near the central axis of the specimen. Local strains ε in those places were calculated from the local diameter D of the necked specimen using the equation

$$\varepsilon = 2{\cdot}\log(D/D_0),$$

where D_0 is the initial diameter.

For every specimen studied, the boundary misorientations were analyzed in two regions corresponding to strains $\varepsilon \approx 0.7$ and 1.0. The EBSD analysis was carried out on SEM LYRA 3 XMN RL using Oxford HKL AZtec™ software; further processing of orientation maps was performed by means of MTEX software [10]. Orientation maps shown in what follows are the inverse pole figure (IPF) maps plotted with respect to the tensile direction (TD).

In the case of specimen III, a considerable part of material turned out to be recrystallized. Since we were interested in the misorientation distribution of strain-induced boundaries, it was necessary first of all to separate the non-recrystallized material from the recrystallized one. To do this, we used grain average misorientation (GAM) derived by averaging kernel average misorientations over a grain [11]. The latter, in its turn, was calculated as an average of misorientations between a given point and its nearest neighbors. When using GAM for the separation, it was assumed that dynamically recrystallized grains did not have a deformation substructure and hence differ by a low value of GAM. An example is shown in Fig. 1. One can see that the recrystallized grains, which easily can be distinguished on the orientation map by a uniform orientation and multiple annealing twins (Fig. 1,*a*), are characterized by relatively low GAM values (Fig. 1,*b*). In the given example, one can exclude recrystallized microstructure by eliminating grains with GAM less than 0.3 deg·μm^{-1}. The distributions of boundary misorientation angle (Fig. 1,*c*) obtained for recrystallized and non-recrystallized regions separated in this way confirm correctness of the separation: the first distribution contains the high-angle peaks corresponding to the annealing twins of the first order (60°) and second one (about 39°), whereas the latter contains the low-angle peak corresponding to the strain-induced dislocation boundaries.

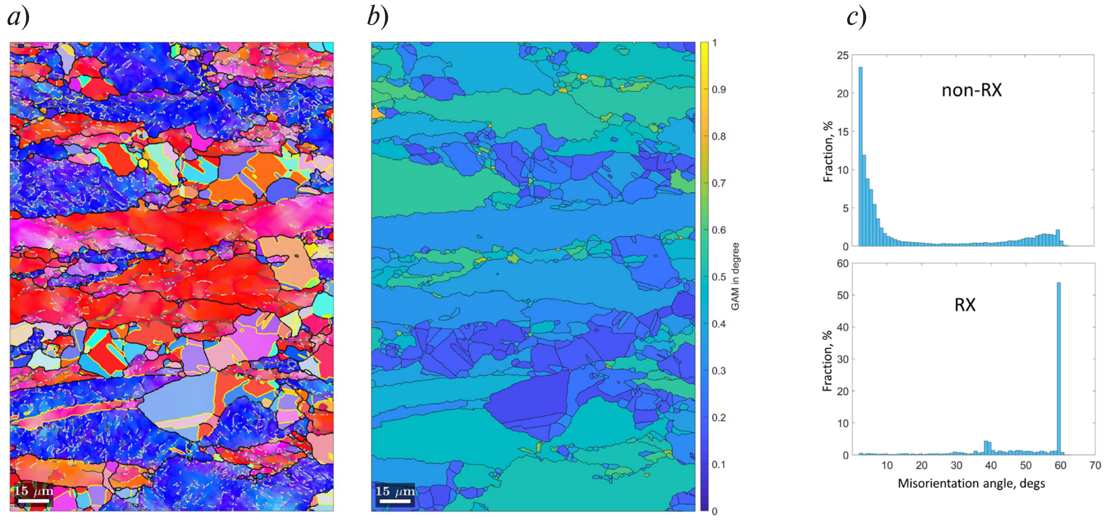

Fig. 1. Inverse pole figure (IPF) (*a*) and grain average misorientation (GAM) (*b*) maps of the same region of specimen III and misorientation angle histograms (*c*) obtained from recrystallized (RX) and non-recrystallized (non-RX) parts of this region.

The boundaries (Bs) on the IPF map are shown: low-angle Bs ($2° < \theta < 5°$) by light grey color; mediate angle Bs ($5° < \theta < 15°$) by dark grey; random high-angle Bs ($\theta > 15°$) by black); $\Sigma 3$ Bs satisfying Brandon criterion by yellow

Results and discussion

Microstructure evolution. Fig. 2 shows representative examples of the microstructure evolved in the deformed specimens. One can see that a grain-scale orientation heterogeneity and multiple low-angle boundaries develop inside original grains in all specimens. Besides, the orientation dependence of the microstructure, which has been described earlier [12], is observed regardless of deformation conditions. At the same time, apparent differences in the microstructures are observed. In particular, a fraction of [001]-oriented material in the specimens deformed at 400°C is considerably larger than that in specimen I. The boundaries of [001]-grains in specimen II are serrated (Fig. 2,*e*) suggesting the occurrence of local grain boundary migration, which usually accompanies DRX. Only a small amount of fine recrystallized grains can be found in specimen II, total area occupied by them remains negligible (about 1 %). However, in specimen III, deformation of which proceeds at the same temperature but for a longer period, DRX develops to a much greater extent (Fig. 2,*c*, *f*). The area fractions occupied by recrystallized grains are about 5% at $\varepsilon = 0.70$ and about 20° at $\varepsilon = 1.05$.

In Fig. 3, the misorientation distributions are presented in terms of the boundary length per unit area. This way of presentation allows not only to determine relative frequencies of boundaries as function of their misorientation but also to characterize accumulation of strain-induced boundaries during deformation. One can see that the length of boundaries increases considerably in the course of deformation at room temperature within the range of strains examined (Fig. 3,*a*), both in the low-angle (less than 15°) and high-angle (more than 15°) ranges. With increasing temperature of active deformation, accumulation of strain-induced boundaries slows down (Fig. 3,*b*), supposedly due to dynamic recovery, which promotes more uniform slip and

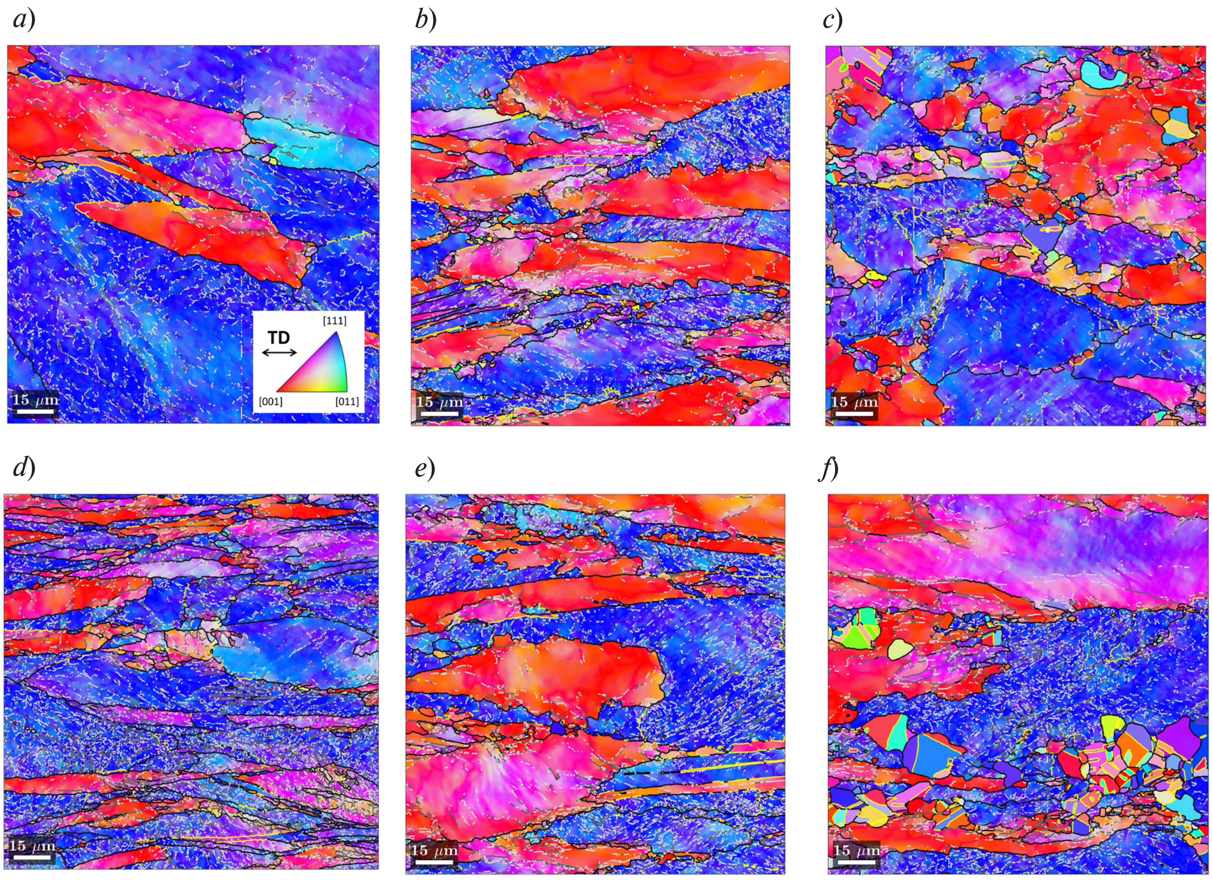

Fig. 2. IPF maps for typical microstructures evolved in the studied specimens I – III: in I, for ε = 0.70 (*a*) and 1.00 (*d*); in II, for ε = 0.65 (*b*) and 1.00 (*e*); in III, for ε = 0.70 (*c*) and 1.05 (*f*).

Standard stereographic triangle, which defines coloring of IPF maps, is inserted in Fig 2,*a*; the tensile direction (TD) is also shown. Color scheme of boundaries on the IPF maps is the same as the one given in Fig. 1

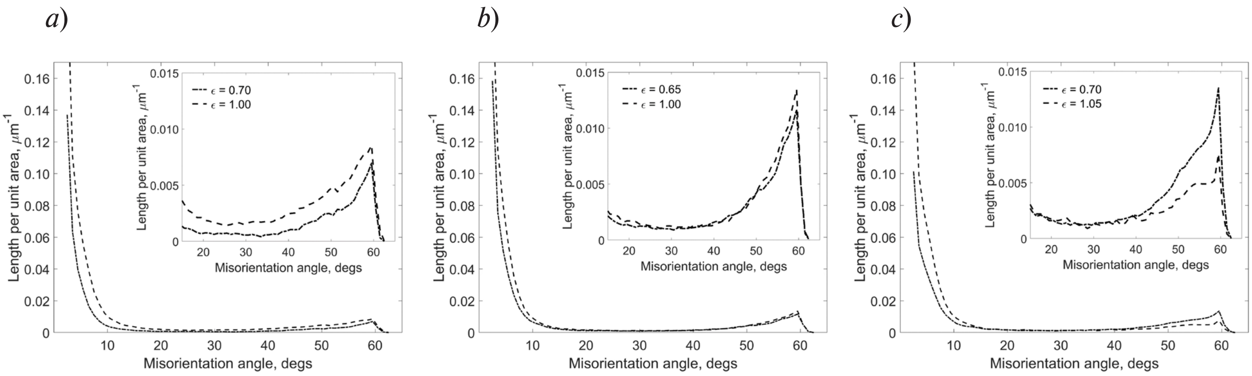

Fig. 3. Distributions of boundary misorientation angles in copper specimens I (*a*), II (*b*), III (*c*), deformed in tension to various strains under different conditions.

In the insets: Enlarged images of the high-angle parts of the graphs counterbalances strain hardening [1]. In specimen III, the recrystallized part of material was excluded from consideration when calculating the distribution shown in Fig. 3,*c* using the procedure described in the previous section. Nevertheless, a boundary length even decreases with increasing strain for the boundaries corresponding to the high-angle peak. Note that this peak is due to boundaries of original annealing twins distorted as a result of deformation. One can suggest that those twins become hardly fragmented in the course of deformation, and hence, accumulate increased stored strain energy, and, for this reason, new recrystallized grains consume them predominantly.

Application of scaling hypothesis to EBSD data. Two kinds of strain-induced boundaries are distinguished [3]:

geometrically necessary boundaries (GNBs) separating regions with different slip system activity;

incidental dislocation boundaries (IDBs) formed by a statistical trapping of dislocations.

The first kind is also called “fragment boundaries” [2], while the second one is “cell boundaries”. The IDBs remain low-angle with strain (the average misorientation is about 2° even after the strains from about 1 to 2), while the average misorientation of GNBs increases significantly [3, 4]. It was shown by the transmission electron microscopy (TEM) [3] that IDBs and GNBs, when considered separately, follow unique distributions f_1 and f_2, respectively. The latter show scaling behavior at small and mediate strains. However, at $\varepsilon \approx 1$ or more, this regularity happens to be violated for GNBs. According to recent study [13], new high-angle boundaries appear at this stage, whose misorientations fall far away beyond the range of scaled distribution f_2, and follow another unique distribution – f_3. As a result, a total distribution of strain-induced boundary misorientations consists of three partial distributions: f_1 (IDB), f_2 (GNB) and f_3 (see it in Fig. 4). The boundaries producing distribution f_3 are in essence also geometrically necessary since they separate regions with different combinations of operating slip systems.

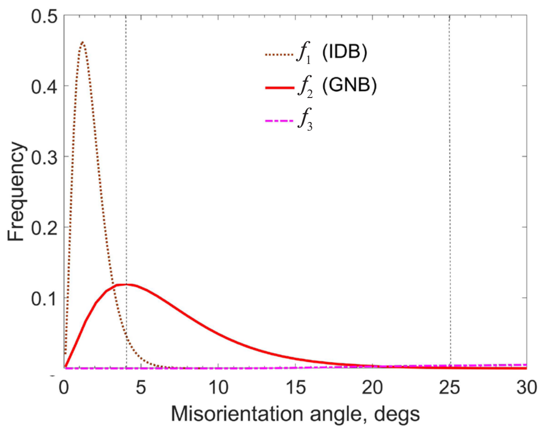

Fig. 4. Partial distributions f_1, f_2 and f_3 constituting total misorientation angle distribution of strain-induced boundaries in copper deformed by compression to strains $\varepsilon \approx 1$ [13].

Vertical lines indicate the bounds of the interval used to test scaling behavior of boundary misorientations

The scaling of GNBs related to distribution f_2 was proved using TEM through their visual separation from IDBs based on different morphological features. The EBSD technique does not allow one to make such a separation. However, one can try finding an approximate solution of this problem from their crystallographic characteristics. To do this, one should isolate an angular range [θ_{min}, θ_{max}] where distribution f_2 is presented with minimal overlapping with f_1 on the left and f_3 on the right, and then calculate the probability density $p(\theta/\theta_{av})$ for this range. Here θ_{av} is the average misorientation angle over the given angular interval.

Based on the available data (see Fig. 4), the contribution of IDBs into the overall angle distribution is minor if θ_{min} = 4° is chosen. On the other hand, the contribution of high-angle strain-induced boundaries (distribution f_3) is negligible for θ_{max} = 25°. A contribution of original grain boundaries is also insignificant within this interval for the large-grained copper investigated in the present study. Obviously, the choice of 25° as an upper bound is rather arbitrary, but its slight variation has no significant effect on the distribution of probability density $p(\theta/\theta_{av})$.

The normalized angle distributions obtained as described above are shown in Fig. 5. It is seen that a scaling of misorientations takes place for three specimens studied, suggesting that physical mechanism of the grain subdivision remains unchanged as the conditions of deformation change.

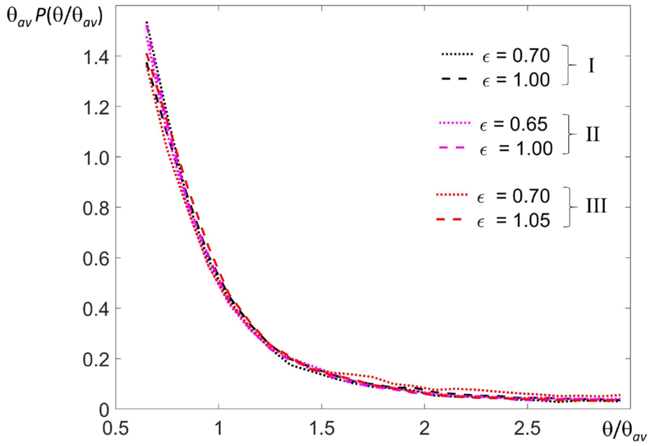

Fig. 5. Scaling behavior of misorientations across GNBs for specimens I, II and III deformed under different conditions

Conclusions

A tensile experiment has been conducted with polycrystalline copper deformed under three different conditions: at a constant strain rate at room temperature (23°C) and at 400°C as well as at a constant stress at 400°C. The evolution of misorientation across strain-induced boundaries were investigated based on EBSD analysis of regions located on the longitudinal section of the specimens. An analysis of the results obtained allows us to make the following main conclusions.

1. The exhibitions of a grain subdivision observed in the examined specimens differ with the deformation conditions. Gradual fragmentation of the original grains occurs at room temperature within the range of strains examined. At the same time, dynamic recovery and recrystallization influence the microstructure and texture evolution in specimens deformed at 400°C considerably.

2. A scaling behavior of strain-induced misorientation takes place. This approves that in spite of recovery and recrystallization effects, the mechanism of strain-induced boundary evolution remains unchanged. Therefore, at 400°C, which is about half the melting point of copper, this evolution is controlled by the micromechanics of polycrystalline material, just like the grain fragmentation at room temperature.

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