# FEATURES OF STRUCTURE OF COPPER TWO-DIMENSIONAL POLYCRYSTALS OBTAINED BY RECRYSTALLIZATION METHOD AND NATURE OF ITS CHANGES IN PROCESS OF PLASTIC DEFORMATION

## E.E. Badiyan, A.G. Tonkopryad, T.R. Zetova, R.V. Shurinov, S.V. Talakh, A.V. Dergacheva V.N. Karazin Kharkiv National University, Kharkov, Ukraine E-mail: Evgeny.E.Badiyan@univer.kharkov.ua

Influence of conditions of recrystallization on the structure of polycrystalline samples of copper and type of boundaries is investigated. It is experimentally shown that depending on the degree of deformation preceding recrystallization annealing, all the samples may be divided conditionally into three types – "monocrystals" with the twins crossing all over the sample; coarse-grained samples containing boundaries of the general type and special type (including twinning); fine-grained samples – of the general type. In the grains of such samples special boundaries including twinning and boundaries of the general type are found. It is shown that during plastic deformation of samples various mechanisms of tension relaxation are realized depending on the type of structure and, finally, determine their mechanical characteristics.

#### **INTRODUCTION**

It is well known that regularities of plastic deformation development of crystalline solids, and finally, their mechanical characteristics, are defined by not only structural and substructural heterogeneity of samples before their deformation, but also by mechanisms of its changing during deformation process. First of all, the question is about the boundaries between the areas of a sample with various crystallographic orientation which are formed in the course of deformation. Formation of new boundaries may result in changes of the ratio between translation and rotational modes of plastic deformation and, finally, and of regularities of plastic deformation development in general. Emergence of the boundaries of deformation origin which are a barrier to moving dislocations may reduce the degree of plastic deformation by sliding. The degree of disorientation of separate areas of the sample may lead, on the one hand, to considerable increase in plasticity of the sample [1-3], on the other hand, the contribution to plasticity may give also changes of crystallographic orientation of separate areas of sample favorable for sliding.

The variety of ways to change substructural and orientation heterogeneity in the course of plastic deformation has been revealed at the investigation of aluminum the two-dimensional polycrystals containing, on the whole, boundaries of the general type [1]. It is experimentally shown that in two-dimensional polycrystals, in the form of grains containing only penetrating boundaries, perpendicular to sample surfaces due to the lack of constraint in the direction, any orientation effects are shown most brightly [1] mJ/cm<sup>2</sup> [2] in copper samples, unlike aluminum samples, practically are always found twins of growth and, therefore, twinning boundaries the role of which isn't obvious in deformation processes. All this provokes a special interest for carrying out investigations of interrelations between structural, substructural and orientation changes and regularities of plastic deformation in copper samples.

#### MATERIALS AND INVESTIGATIONS METHODS

To prepare the samples, copper foil (99.97%), 200  $\mu$ m thick was used, the average size of grains was  $\approx$ 0.1 mm. The samples, size  $70 \times 10$  mm<sup>2</sup>, were cut out from a sheet of foil. For having samples with various grain size the so-called method of "critical deformations" (which consists in primary annealing at temperature of 700 °C for two hours; sample deformation value of 2...7%; subsequent recrystallization annealing) was used at temperature of  $\approx 1000$  °C for five hours. Before the last annealing the both surfaces of samples were polished. Annealing of samples were carried out in a vacuum furnace. The depth of vacuum was ~  $10^{-2}$  Pa. Samples boundaries became apparent by means of spirit solution of concentrated nitric acid. The time of etching was of 3...10 s. All the samples were deformed in conditions of monoaxial stretching with constant speed of deformation  $2 \times 10^{-5} \text{ s}^{-1}$  at a room temperature. For all samples of *in situ* with the period 0.02 s in the course of deformation the color orientation maps (COM) were registered [5, 6], allowing to define the characteristics of substructural and orientation heterogeneity. If necessary, the visualization technique [7, 8] of color shades on COM allowing to reveal the slightly disoriented areas on the sample surface was used. For a part of samples, in certain areas, their crystallographic orientation was determined by Laue's method and the complete certification of grains boundaries was obtained. These areas of researches are specified in microphotos.

#### **EXPERIMENTAL RESULTS**

Color orientation maps of the surface of polycrystalline samples obtained by recrystallization method are given in Fig. 1.

The increased fragments of these structures are given in Fig. 2. All the samples can be divided into 3 types conditionally. The value of preliminary deformation was the only parameter which changed to obtain such a variety of structures. So, for the samples (their surface structure is given; it makes -2% Fig. 1 *e*, Fig. 1 *a* -4%, Fig. 1,*b*,*c*,*d*-6...7%.



Fig. 1. The microstructure of copper samples obtained by recrystallization depending on the value of preliminary deformation: a)  $\varepsilon = 4\%$ ; b, c, d)  $\varepsilon = 6...7\%$ ;





Fig. 2. Microphotos of the increased fragments of the surface of copper samples (the structure is given on Fig. 1,a,c,d)

The samples of the first type contain only twinning boundaries (Fig. 1,a, Fig. 2,a) crossing all over the surface. In Fig. 1,b,c,d and Fig. 2,b variety of structure of samples of the second type is given. They contain twins differing in form, sizes and orientation of grain boundaries of special and general type. The increased fragment of this structure containing boundaries of grains and twinning boundaries 2 is shown in Fig. 2,b. Boundary certification according to X-raying data showed that the boundary 2 is coherent twinning boundary  $\Sigma$ 3, 60°, [111], boundary 1 – boundary of the general type, and grain boundary 3 - similar to the special type.

Samples of the third type (Fig. 1,e and Fig. 2,c) contain fine-grained structure with boundaries of the general type. The average size of such grains makes  $\approx 1$  mm. Almost in each grain (Fig. 2,c) twins breaking in the grain body are found. It should be noticed that all the samples (their structure is shown in Fig. 1 and Fig. 2), are through.

In the course of plastic deformation of copper samples of the 1st type the emergence of extrusion (Fig. 3,a, b,c) and intrusions (Fig. 3,c) is found, their form being various, but all of them are localized near the twins so that at least one of boundaries of extrusion or intrusion was perpendicular to twinning boundary.



Fig. 3. Fragments of surface of copper sample (Fig. 1,a) after its deformation for 12%. Near twinning boundaries, extrusions (a, b, c) and intrusions are found (c)

The feature of emergence of extrusion (Fig. 4,a) on one of the sample surfaces at plastic deformation of copper two-dimensional polycrystals containing only twinning boundaries is that on the opposite surface of the sample (thickness of 200  $\mu$ m) there are always intrusions, Fig. 4,c. The interferograms of the both surfaces obtained by means of Linnik's interferometer testify to being of extrusions and intrusions. The interferogram of the sample surface area on a boundary "grain body – extrusion" is given as an example on Fig. 4,b.

Formation of extrusions and intrusions, as a rule, occurs in the course of fatigue tests of samples and depends on easiness of cross sliding which is always complicated in copper because of the low value of defect packing energy. The place of their localization – steady strips of sliding [9]. To explain the phenomena of emergence of extrusions and intrusions at plastic deformation of copper samples, apparently, it is necessary to consider also the phenomena of sliding in secondary systems. For the area of extrusion (intrusion) emergence the crystallographic orientation of axis of stretching of the sample was defined and it was close to the direction  $[\overline{1}12]$ . At that orientation of axis of stretching at the same time the primary system of sliding  $(\overline{1}11)[011]$  at plastic deformation is carried out according with secondary, interfaced system of sliding  $(111)[\overline{1}0\overline{1}]$  too. Thus, specific crystallographic orientation of axis of stretching of the sample, its thickness and penetrating twinning boundary being in a place, may be a possible reason of extrusion emergence on the surface of sample and at the same time on the opposite side of intrusion.



Fig. 4. Increased fragments of the surface of a copper sample deformed for 12%, extrusion on one side – (a), and intrusion on the opposite side – (c) and the interferention picture of area between the boundary grain body and extrusion (b).



Fig. 5. Fragments of the surface of copper sample given on Fig. 1 (d), after deformation for 14%

Samples of the second type possess coarse-grained structure. The most part of grains contain twins various in form, sizes and orientation. The other grains do not contain twinning boundaries. Plastic deformation in such samples is realized by development of dislocation sliding (Fig. 5,a) or rotational effects (1) (see Fig. 5,b). In certain grains phenomena of dislocation sliding through twinning boundaries (see Fig. 5,c) is revealed. Plastic deformation of the 3rd type-samples containing grain boundaries of the general type is characterized by formation of rotational structure (1) (Fig. 6,a) and development of dislocation sliding (see Fig. 6,b). In these samples because of grain boundaries of the general type which are an obstacle for moving dislocations, and intensive development of sliding in each grain, the highest values of strength  $\sigma_{\rm B}$  and the maximum plastic deformation ( $\varepsilon_{\rm max}$ ) take place before destruction of samples in comparison with the samples of the 1st and 2nd type (table).

| Mechanical | characterist | tics of the | studied | samples in |
|------------|--------------|-------------|---------|------------|
| compariso  | n with samp  | oles of the | 1st and | 2nd type   |

| Type of simple | Conditional flow limit $\sigma_{0,2}$ , MPa | Strength $\sigma_{\rm B}$ , MPa | Maximum<br>deformation<br>before destruc-<br>tion<br>ε <sub>max</sub> , % |  |  |
|----------------|---|---------------------------------|---|--|--|
| Ι              | 35  | 100                             | 6   |  |  |
| Π              | 30  | 125                             | 12  |  |  |
| III            | 60  | 480                             | 21  |  |  |



Fig. 6. Microphotos of various areas of the surface of copper sample given on Fig. 1 (e) after its deformation for 10%, a – emergence of rotation (1), b – development of dislocation sliding

## CONCLUSIONS

It is experimentally shown that the changes of recrystallization conditions of copper foil 200 µm thick is possible to change in the samples not only the grain average size, but also the type of all the boundaries which are through. Conditionally all the variety of structures can be divided into three types. The samples of the first, "monocrystals", type containing only twinning boundaries crossing the whole sample. Only finegrained samples belong to the second type  $(d \approx 10 \text{ mm})$ . The structure of these samples is the most diverse. They contain twins differing in form, sizes and orientation of grain boundaries of special and general type. The third type includes fine-grained samples  $(\overline{d} \approx 10 \text{ mm})$ , all the boundaries belonging to the general type. Practically in each grain of such samples twins are found. Such variety of structure in copper two-dimensional polycrystals results, at plastic deformation, in specific ways of tension relaxation and finally in changes of mechanical characteristics of samples. So, in the samples of the first type in the course of their plastic deformation at the room temperatures near twinning boundaries there are extrusions and intrusions - the ways of tension relaxation being not proper to copper. In the samples of the 2nd type rotational changes and the effect of transparency of twinning boundaries for sliding of dislocations are found. In the samples of the third type within each of grains an intensive sliding is observed. Plasticity of such samples increases simultaneously with increasing of strength.

#### REFERENCES

1. Е.Е. Бадиян, А.Г. Тонкопряд, О.В. Шеховцов, Р.В. Шуринов. Ориентационные изменения и развитие трещин в процессе пластической деформации двумерных поликристаллов алюминия // *МФиНТ*. 2008, т. 30, №3, с. 361-371.

2. В.В. Рыбин. Большие пластические деформации и разрушение металлов. М.: «Металлургия», 1986. 224 с.

3. В.В. Рыбин. Закономерности формирования мезоструктур в ходе развитой пластической деформации // Вопросы материаловедения. 2002, №1(29), с. 11-33

4. И.И. Новиков. Дефекты кристаллического строения металлов. М.: «Металлургия», 1983, 232 с.

5. E.E. Badiyan, A.G. Tonkopryad, O.V. Shehovtsov, R.V. Shurinov, T.R. Zetova. Optical Technique for the In Situ Study of Orientation and Structure Changes Accompanied the Plastic Deformation of Polycrystalline Specimens of Aluminum // *Inorganic Materials*. 2011, N 15, p. 1663-1666.

6. Патент 93021 Україна. Спосіб визначення кристалографічної орієнтації зерен на поверхні полікристалічного зразка / Є.Ю. Бадіян та інші. 2010, Бюл. №24.

7. Е.Е. Бадиян, А.Г. Тонкопряд, О.В. Шеховцов, Р.В. Шуринов, Т.Р. Зетова, Е.С. Казачкова. Визуализация субструктурной и ориентационной неоднородности в отдельных зернах поликристаллических образцов // Заводская лаборатория. Диагностика материалов. 2014, т. 80, №8, с. 37-40.

8. Патент 104249 Україна. Спосіб візуалізації орієнтаційної неоднорідності та морфології поверхні монокристала або окремих зерен полікристала / Є.Ю. Бадіян та інші. 2014, Бюл. №1.

9. В.Е. Панин, Н.С. Сурикова, Т.Ф. Елсукова, В.Е. Егорушкин, Ю.И. Почивалов. Наноструктурированные фазовые границы в алюминии при циклической интенсивной пластической деформации // Физическая мезомеханика. 2009, т. 12. №6, с. 5-15.

Article received 11.12.2015

## ОСОБЕННОСТИ СТРУКТУРЫ ДВУМЕРНЫХ ПОЛИКРИСТАЛЛОВ МЕДИ, ПОЛУЧЕННЫХ МЕТОДОМ РЕКРИСТАЛЛИЗАЦИИ, И ХАРАКТЕР ЕЕ ИЗМЕНЕНИЯ В ПРОЦЕССЕ ПЛАСТИЧЕСКОГО ДЕФОРМИРОВАНИЯ

#### Е.Е. Бадиян, А.Г. Тонкопряд, Т.Р. Зетова, Р.В. Шуринов, С.В. Талах, А.В. Дергачева

Исследованы влияния условий рекристаллизации на структуру поликристаллических образцов меди и тип границ раздела. Экспериментально показано, что в зависимости от величины деформации, предшествующей рекристаллизационному отжигу, все полученные образцы можно условно разделить на три типа – «монокристаллы» с двойниками, пересекающими весь образец; крупнозернистые образцы, содержащие границы общего и специального типов (в том числе и двойниковые), и мелкозернистые образцы, все границы зерен которых общего типа. Внутри зерен таких образцов обнаруживаются специальные границы, в том числе двойниковые и общего типа. Показано, что в процессе пластического деформирования образцов реализуются, в зависимости от типа структуры, различные механизмы релаксации напряженного состояния, в конечном итоге определяющие их механические характеристики.

## ОСОБЛИВОСТІ СТРУКТУРИ ДВОВИМІРНИХ ПОЛІКРИСТАЛІВ МІДІ, ЩО ОТРИМАНІ МЕТОДОМ РЕКРИСТАЛІЗАЦІЇ, І ХАРАКТЕР ЇЇ ЗМІНИ В ПРОЦЕСІ ПЛАСТИЧНОЇ ДЕФОРМАЦІЇ

#### *Є.Ю. Бадіян, А.Г. Тонкопряд, Т.Р. Зстова, Р.В. Шурінов, С.В. Талах, Г.В. Дергачова*

Досліджено вплив умов рекристалізації на структуру полікристалічних зразків міді та тип меж зерен. Експериментально показано, що залежно від величини деформації, яка передує рекристалізаційному відпалу, усі отримані зразки можна умовно розділити на три типи – «монокристали» з двійниками, що перетинають увесь зразок; крупнокристалічні зразки, що містять межі загального і спеціального типів (у тому числі двійникові), і дрібнозернисті зразки, усі межі зерен яких загального типу. Всередині зерен таких зразків виявляються спеціальні межі, у тому числі двійникові та загального типів. Показано, що в процесі пластичної деформації зразків реалізуються, залежно від типу структури, різні механізми релаксації напруженого стану, які визначають зрештою, їх механічні характеристики.