A STUDY OF THE STRUCTURE OF THE BOUNDARIES OF GRAINS IN TUNGSTEN ION WITH FIELD-EMISSION MICROSCOPE

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The structure of large-angle boundaries of grains in tungsten is studied with a field-emission ion microscope. It is shown that the misorientation of grains corresponds as a rule to the lattice of the coinciding points with a sufficiently high density of coincidences. In those cases when a boundary does not lie along a close-packed surface of a sublattice, a stepped structure is observed. The height of the steps varies from one atomic parameter in unannealed tungsten to several hundred in recrystalized tungsten. The conjugation turns out to be satisfactory also in those cases when the height of microsteps is not a multiple of interplanar sublattice spacings. For an arbitrary height of steps, the conjungation of adjacent-grain lattices can be ensured at the expense of a system of partial dislocations.

DIFFERENT physical properties of polycrystalline solids depend to a strong degree on the structure of the intergranular boundaries. Several models were proposed at different times for their description. Mott[1], on the basis of results of experiments on diffusion mobility of the boundaries, proposed that the intergrain boundaries consist of "islands" of good conjugation, surrounded by regions with a strongly disturbed structure. Smoluchowski[2] proposed to regard the regions of poor conjugation as clusters of dislocations. Li[3] attempted to describe the large-angle intergranular boundaries by a method similar to that used by Read and Shockley in the case of block boundaries. In view of the large dislocation density on large-angle boundaries, Li was forced to take into account the distortion of the form and the dimensions of the dislocation nuclei.

An alternative of the dense disposition of the dislocations in the boundary layer is a system of dislocation walls that ensure a smooth transition between grains. The energy of such boundaries is somewhat higher than that of single-layer boundaries, but the difference is very small, so that the possibility of formation of multiwall boundaries is admitted[4].

Recently, in connection with the uses of especially pure material for different purposes, interest in the investigation of "special" boundaries with anomalously large mobility has increased. Aust and Rutter have shown that mobile boundaries are characterized by a high density of the coinciding lattice points[5]. For boundaries that do not correspond fully to the model of the coinciding lattice points, it is possible to introduce a system of dislocations that compensates for the deviation from the conjugation condition[6-8]. However, even in this case the published data on the structure of the intergranular boundaries cannot always be reconciled with the indicated model.

In order to clarify the details of this structure of large-angle boundaries and to compare them with the indicated models, we undertook an ion field-emission microscope study of intergranular boundaries in cold-drawn tungsten wire subjected to controlled annealing.

MATERIAL AND INVESTIGATION PROCEDURE

The object of the investigation was cold-drawn tungsten 99.9% pure. Certain samples were annealed at a temperature 1400–600°C for 10–12 minutes in vacuum at a pressure of 10^{-6} mm Hg. The system was first evacuated to 10^{-6} mm Hg. and filled to atmospheric pressure with pure helium. The samples were made of wire by electrochemical polishing. During the course of the investigation, additional polishing was performed by the field at liquid-nitrogen temperature. A helium ion field-emission microscope with nitrogen cooling was used in the experiments. The pressure of the residual gases in the system, prior to filling with the helium, was $\sim 10^{-7}$ mm Hg, and the helium pressure was 4×10^{-3} mm Hg.

The disorientation of the grains was determined from the positions of the poles of the close-packed planes {110}, {211}, {321}, and {111}. The error in the determination of the disorientation angles was ±2°. The density of the boundary was determined accurate to ±4° from the displacement of the track of the boundary during the process of controlled evaporation by the field.

RESULTS AND DISCUSSION

Grain boundaries were observed in 28 out 126 samples. The orientation of the majority of the microcrystals prepared from the recrystallized wire corresponds to a [110] rolling texture. During the recrystallization process, the rolling texture was as a rule conserved. However, in some cases an appreciable deviation from the [110] orientation was observed; in particular, microcrystals with [320] axis were observed.

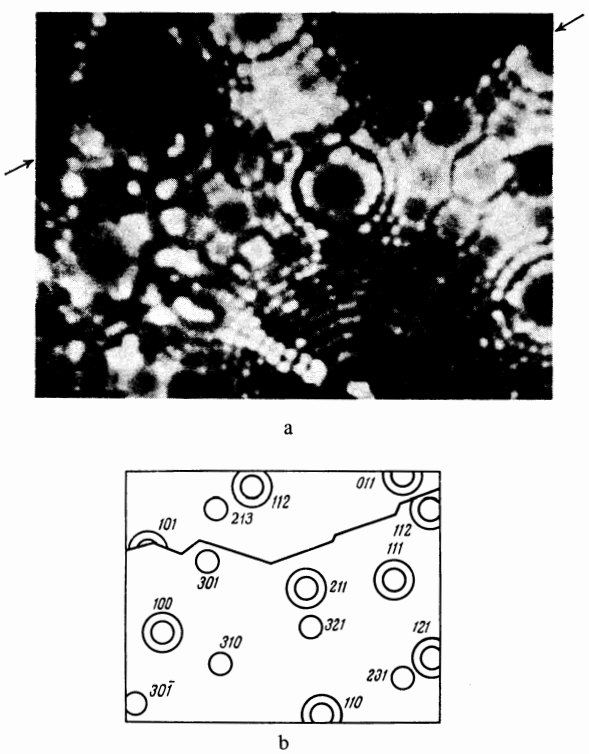
Figure 1a shows the field-emission image of the surface of a tungsten bicrystal containing a large-angle inclined boundary. The disorientation axis is [110] and the disorientation angle is $\omega = 40°$, the boundary lying within ±5° of the $(\bar{1}13)$ plane. Several microsteps of height from 3 to 30 Å are observed in a section of the

Reciprocal density of coinciding sites	Angle of disorientation relative to the [110] axis, ω	Closepacked planes of sublattices	Burgers vectors of twinning dislocations b/a
3	70.5	112	$^1/_6\langle 111\rangle$, $^1/_3\langle 111\rangle$
9	38.9	114	$^4/_9\langle 122\rangle$, $^5/_9\langle 122\rangle$
11	50.5	332	$^5/_{22}\langle 113\rangle$, $^3/_{11}\langle 113\rangle$
17	86.6	334	$^7/_{17}\langle 223\rangle$, $^{10}/_{17}\langle 223\rangle$
19	26.5	116	$^7/_{38}\langle 133\rangle$, $^6/_{19}\langle 133\rangle$
27	31.5	552	$^5/_{27}\langle 115\rangle$
33a	20.0	118	$^8/_{33}\langle 144\rangle$
33 b	59.0	554	$^{14}/_{33}\langle 225\rangle$

boundary approximately 300 Å long; these steps lie in the $(\bar{1}14)$ plane, which is the most closely packed in the sublattice with reciprocal coinciding-site density $\Sigma = 9$.

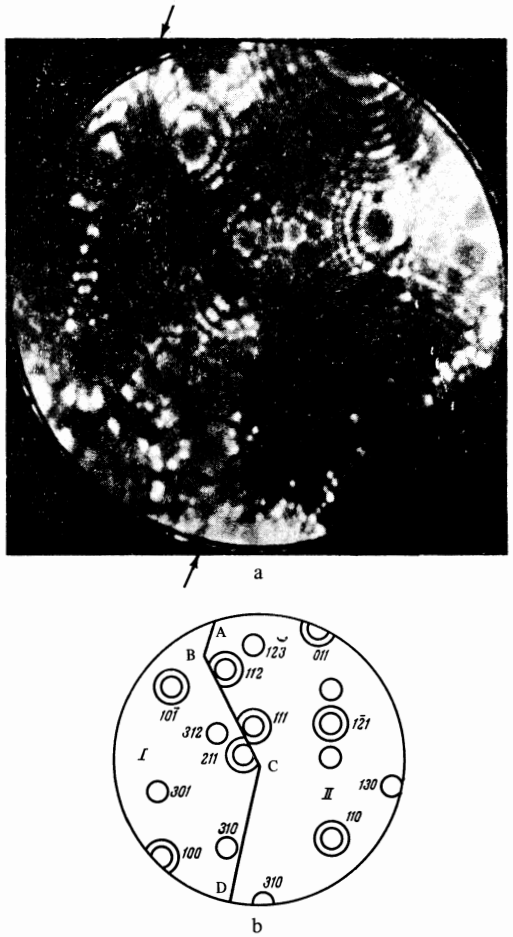
The width of the steps depends in general on the angle of inclination of the boundary to the close-packed plane of the lattice of the coinciding sites (see the table). In samples made of non-recrystallized wire, the width of the steps usually does not exceed 100 Å. Recrystallization makes the microsteps larger. By way of an example, Fig. 2a shows a microphotograph of a sample made of wire recrystallized at 1400°C. The disorientation can be described by a rotation through $\omega = 38°$ around the [110] axis, corresponding to a sublattice with $\Sigma = 9$. Only one step, with approximate height 150 Å, is located in the image field. The boundary sections AB and CD lie in the plane (114) parallel to the close-packed plane of the sublattice with reciprocal density of the coinciding sites on the boundary $\sigma = 1$.

The conjugation on the section BC, corresponding to the planes $(1\bar{1}2)$ of crystal I and $(5\bar{5}2)$ of crystal II (see Fig. 2b), is also sufficiently high, this being manifest in the absence of tracks of preferred evaporation by



a b

FIG. 1. Intergranular boundary (designated by arrows). The disorientation angle is 40° relative to the [110] axis. Microsteps with height from 3 to 30 Å are located along the boundary.



a b

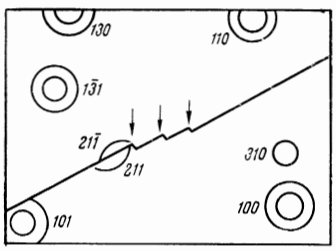
FIG. 2. Recrystallized tungsten bicrystal. Within the limits of the field of view, the boundary consists of two parallel sections (AB and CD) located in the $(1\bar{1}4)$ plane, and a transition section BC representing a step of height 150 Å. On section BC, the coincidence of the sites is characterized by $\sigma = 3$; on the remaining sections $\sigma = 1$.

the field in the region where the boundary emerges on the surface of the sample. Using the model of lattices with coinciding points, it can be shown that the section of the boundary under consideration is partially conjugated: every third site along the boundary plane belongs to both lattices ($\sigma = 3$).

The presence of relatively large terraces (up to several hundred Å) lying in the close-packed planes of the sublattice is characteristic of the majority of asymmetrical inclined boundaries in recrystallized tungsten. The steplike structure is observed also in those cases when the deviation from symmetrical orientation reaches 25–30°.

In nonrecrystallized tungsten one frequently encounters steps whose height is smaller than the corresponding interplanar distances of the sublattice. Thus, the microphotograph of Fig. 3a shows on the boundary of the microcrystals which are disoriented by an angle of 87° relative to the [110] axis three monatomic steps (designated by the arrows on Fig. 3b), lying in the $(3\bar{3}4)$ plane. Since the height of the terraces is smaller than the interplanar distance of the sublattice (9.03 Å) the boundary obviously cannot belong entirely to the sublattice.

a



b

FIG. 3. Unannealed tungsten bicrystal. Three monoatomic steps are designated by arrows on the scheme. The grain boundary lies in the plane $(3\bar{3}4)$. Each step corresponds to a partial dislocation $^{7}/_{17}a\langle 223\rangle$.

The section of the boundary lying in a plane parallel to the closely-packed plane of the sublattice but not coinciding with it, should correspond to a zero density of the coinciding sites (Fig. 4a). Nonetheless, experiment shows that conjugation of the boundaries containing steps of arbitrary height (including monatomic) turns out to be sufficiently high and does not differ from conjugation of the steplike boundaries with a height that is a multiple of the interplanar distances of the sublattice.

Crystallographically regular conjugation on the section of the boundary not lying in the close-packed plane of the sublattice can be obtained by tangential displacement of one of the lattices by a definite fraction of the parameter (see Fig. 4b). Such a displacement can be realized by partial (twinning) dislocations.

Using the method of Jaswon and Dove[9], we can find an expression for the corresponding Burgers vectors of the partial dislocations:

$$\mathbf{b} = \frac{n}{2\Sigma}(\epsilon^2\Sigma - na^2)^{1/2}[uv\omega],$$

where a is the lattice parameter, ϵ is the shortest distance between sites belonging to two close-packed planes, and u, v, and ω are the indices of the normal to the disorientation axis in the plane of the boundary, and n = 1, 2.

The table lists the values of the Burgers vectors for the grain boundaries with disorientation relative to the [110] axis through angles corresponding to the formation of sublattices with reciprocal site density up to $\Sigma = 33$.

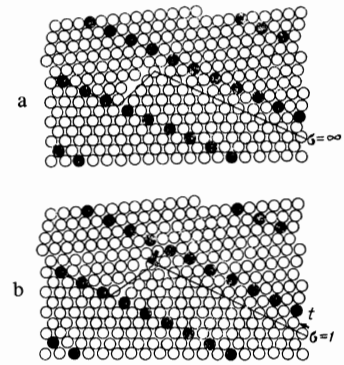


FIG. 4. a—arrangement of atoms near incoherent boundary ($\sigma = \infty$); b—restoration of coherence as a result of partial dislocation.

Most dislocations in the investigated samples were pure edge dislocations, with Burgers vectors approximately parallel to the surface of the samples. Therefore it is most frequently difficult to observe their emergence to the surface. In some cases, when the Burgers vector is inclined to the surface of the sample, or when the absolute value of the Burgers vectors is sufficiently large, it is possible to distinguish those places where such dislocations emerge to the surface. In Figs. 3a and b, such locations, corresponding to emergence of dislocations with Burgers vector (7/17) a ⟨223⟩, are designated by arrows. Thus, neighboring grains of polycrystalline tungsten deformed by rolling are disoriented through discrete angles corresponding to the lattice of the coinciding sites. The boundaries are steplike, but the heights of the steps differ from the interplanar distances of the sublattice. These steps are formed as a result of motion of partial dislocations. It is shown that during the course of annealing, the steps become larger, this being apparently due to accumulation of dislocations moving in neighboring planes when obstacles are encountered.

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