LOW-TEMPERATURE RECRYSTALLIZATION OF MICROCRYSTALS OF TUNGSTEN

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Tungsten deformed at liquid-nitrogen temperature was studied in a field ion microscope after being kept at room temperature. It is shown that in the deformed boundary region of the microcrystals, new recrystallization centers may arise at 600–700° C. The transverse size of the stable grain is 20–60 Å at a disorientation angle of 8–10°. The dislocation structure of the boundaries is discussed.

INTRODUCTION

In study of the recrystallization of plastically deformed materials, two mechanisms are discussed for the formation of recrystallization centers: the formation of nuclei inside strongly distorted regions of the deformed matrix, and growth of new crystals from blocks, fragments, or subgrains of the parent crystal, which has been broken up in the deformation.[1] It would be possible, by studying the initial stages of recrystallization, to discover which of these processes takes place; however, the numerous metallographic, x-ray, and electron-microscope methods have turned out not to be very effective, as the result of their poor resolution. Considerably greater possibilities are presented by use of the field ion microscope, whose resolution approaches the lattice constant of the metal.

It is known that low-temperature cold working leads to a reduction of the recrystallization temperature[2] and correspondingly to a reduction of the rate of crystallization. In studying the initial stages of recrystallization by direct observation in a field ion microscope, it was desirable to slow up the process; for this purpose the samples studied were subjected to deformation at liquid-nitrogen temperature.

TECHNIQUE

The samples consisted of grade VA-3 tungsten wire of purity 99.9%, deformed by bending at liquid-nitrogen temperature. Needle-shaped samples were prepared from the wire by electrochemical etching. In the microscope the surface of the point was cleaned and polished by evaporation in an electric field at liquid-nitrogen temperature until a stable shape was obtained. Thus, the previous history of the material studied in the field ion microscope consisted of deformation at liquid-nitrogen temperature, heating to room temperature, and a short (1 sec) heating to 100–200° C in fastening the specimen to a loop by spotwelding. The recrystallization annealing was carried out directly in the microscope in the absence of an electric field. After the annealing the specimen was again polished in an electric field at liquid-nitrogen temperature for the purpose of reestablishing a stable shape.

RESULTS AND DISCUSSION

In studying the surface of specimens prepared by the means described above, in most cases we observed distortions of the crystal structure of the material (intergrain boundaries, boundaries of blocks, dislocations, etc.). We frequently observed stable distortions of the shape of the emitting surface of the point, which were not removed by field evaporation even after annealing; this is evidently due to incorrect shape of the shank of the point, arising as the result of preferential electrochemical etching of strained portions in the process of preparing the point.

The incorrect shape of the shank is the cause of variation in the magnification of the image, which considerably hinders the interpretation of the microphotographs, in particular, the determination of the planes of the boundaries and the disorientation angles of the grains; however, for angles up to 60° the orientation relations can be established with a sufficient degree of accuracy (within 1–2°).

As a result of the nonuniformity of the deformation it was impossible to use data on deformation of the wire from which the samples were prepared to estimate the amount of cold working. Therefore it was necessary to compare the structure of the specimen before annealing with its structure after annealing and thus obtain qualitative characteristics of the recrystallization process.

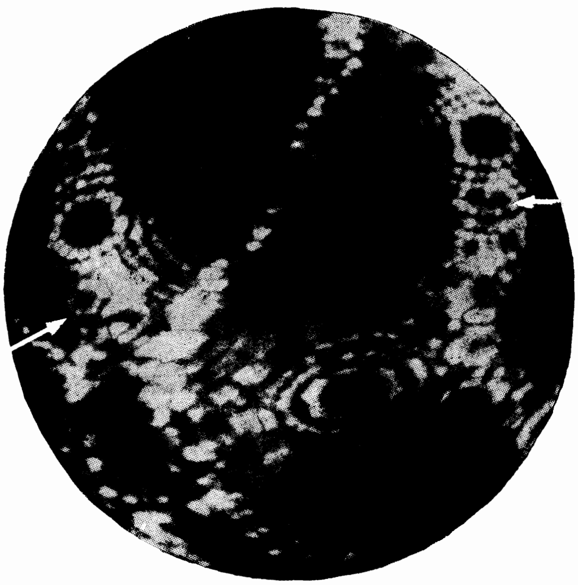

FIG. 1. Field ion microscope photograph of a specimen prepared from tungsten wire, deformed by bending at liquid-nitrogen temperature. The arrows indicate the trace of a grain boundary.

Figure 1 shows an ion-projection tube microphotograph of a tungsten sample not heated after deformation. In the central part of the surface of the point is observed a boundary (its trace is denoted by the arrows) between two adjacent grains, whose dimensions correspond approximately to the radius of curvature of the point, $r \approx 200$ Å. The disorientation angle (not more than 15°) is determined from the position of the identical (111) and (112) faces in each of the grains (see the diagram, Fig. 2b). One is struck by the unusual distortion of the picture. It is easy to show that in different places on this point the principal radii of curvature of the surface differ very strongly: thus, near the (110) faces of crystal A and the (211) faces of crystal B the principal radii of curvature differ by almost a factor of two. This is the result of nonuniform evaporation in the electric field, arising either from the presence of residual strains close to the theoretical strength of the material, or from the effect of an incorrect, nonconical shape of the shank. The distorting effect of the shank, however, can be excluded, since, after 10 sec of annealing at 700°C and electric-field polishing, the surface has already acquired

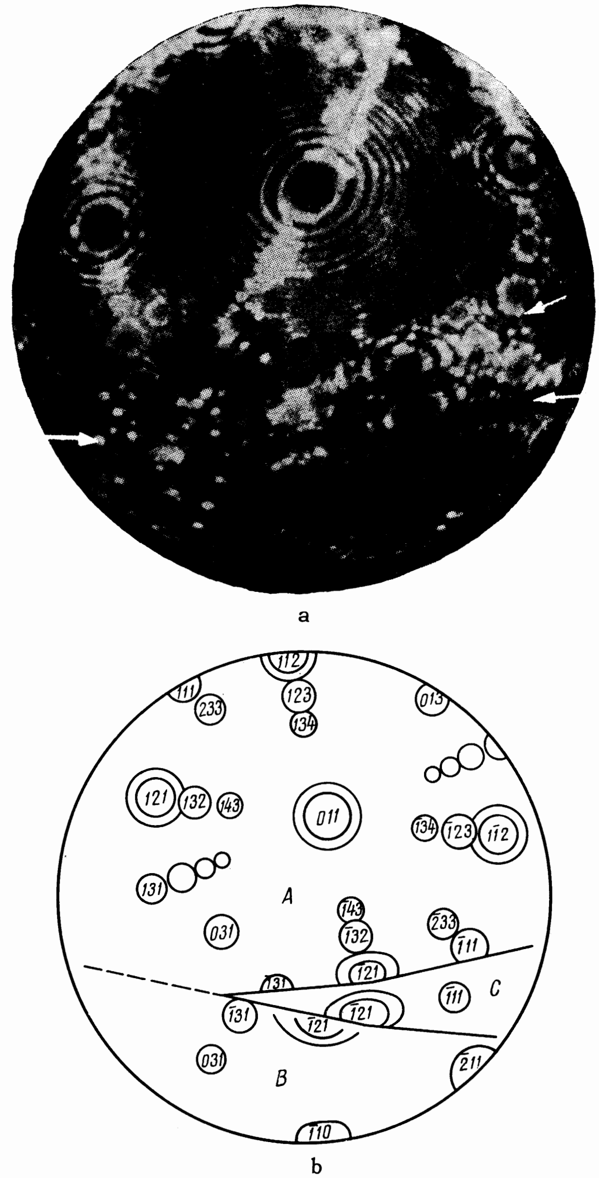

FIG. 2. a – The same specimen after annealing at 600–700°C for 10 sec. The trace of the boundary has shifted. A wedge-shaped recrystallization nucleus has formed at the boundary. b – Diagram of the location of the principal planes of the microcrystals. A and B – parent crystals. C – New wedge-shaped crystal.

an almost spherical shape. Removal of more than 400 atomic layers in the polishing process prior to annealing did not lead to appreciable improvement in the correctness of shape of the point surface. Thus, it was shown that in this case large residual strains were preserved in the specimen. This is also confirmed by the fact that before the annealing an anomalously high rate of evaporation was observed at liquid-nitrogen temperature in a field $E \approx 4.5 \times 10^8$ V/cm; after annealing, evaporation under the same conditions was not observed even at $E \approx 5.5 \times 10^8$ V/cm.

The recrystallization annealing of the specimen was carried out at 600–700 °C, since the cold working at liquid-nitrogen temperature permitted us to count on a considerable reduction in recrystallization temperature (the usual recrystallization temperature of tungsten is ~1200 °C).

Figure 2a is a microphotograph of the point of the same specimen after annealing for 10 sec, with subsequent cooling to liquid-nitrogen temperature and brief electric-field polishing. The trace of the boundary of grains A and B has shifted to a new location in the annealing process. Analysis of a series of successive microphotographs of the sample, obtained before and after annealing, shows that migration of the boundary took place.

In Fig. 2a we can distinguish three crystallites: a large one A, a small one B, and a wedge-shaped one C (see the diagram Fig. 2b). The boundary between the large and the wedge-shaped crystals is symmetrical and inclined, the plane of the boundary being close to (101). The absence of preferential electric-field evaporation of the surface of the point near the trace of this boundary can evidently be explained by the fact that the edge dislocations which form the boundary are approximately parallel to the portion of the surface being discussed.

The boundary between the wedge-shaped crystal C and the small crystal B is strongly etched and has a serrated shape. This we can consider as an indication that the dislocation lines come out to the surface at a large angle. This grain structure, and also the location of the $(\bar{1}21)$ face in the wedge-shaped crystal C and the small crystal B, indicate that this is a screw boundary with a $\langle 011 \rangle$ rotation axis; the disorientation angle is about 10°.

The boundary of the initial grains A and B is complex, containing inclined and screw components. At the vertex of the wedge-shaped nucleus this boundary transfers discontinuously to the two boundaries of the new crystal, each of which contains only one component: the boundary A-C is inclined, and the boundary B-C is screw-type. It can be assumed that in the annealing process the complex well of dislocations broke up into two simple walls, each of which contains one type of dislocation.

Various methods exist for determining the recrystallization parameters. The most sensitive method is the determination of changes in the mechanical properties, which permits observation of the early stages of recrystallization, bordering on such phenomena as the relaxation of residual strains and recovery. According to the data of Davis[3] and Aleksandrov and Mordyuk,[4] the stability of previously cold-worked tungsten begins to decrease at 600–700 °C only after 10–100 minutes of annealing. The value of the incubation period found by us ($\tau < 10$ sec) is below the statistical average and can be considered the result of special deformation conditions at the boundary of a given pair of grains.

In the process of further field evaporation, it was established that the wedge-shaped crystallite has a considerable extension in a direction perpendicular to the surface of the point. After etching away about 100 $(\bar{1}21)$ atomic layers, the shape and size of this crystallite did not greatly change. Thus, the wedge-shaped crystallite can be considered as a thin plate of volume at least 10^{-18} cm^3. The wedge shape of the grain suggests the idea of a relatively high growth rate along the separation boundary, i.e., in the region of localization of the most severe distortions.

Thus, we have been able to observe in deformed tungsten during the annealing process the origin, at the boundary of two grains, of a stable recrystallization nucleus with a rather large disorientation angle with respect to the parent grains.

The observation of the formation of recrystallization centers by growth of blocks already existing in the deformed matrix is difficult in the field ion microscope because of the small field of view, which in most cases turns out to be less than the average size of the blocks. However, in tungsten deformed at low temperatures, in a number of cases we have been able to observe layer-blocks of thickness up to 100 Å with orientations with respect to the parent matrix of less than one degree. Heating to 600–700 °C led to enlargement of the block to the dimensions of the field of view. We can suppose that in these cases we have observed the initial stage of formation of recrystallization centers by growth of blocks preserved in the deformed matrix.

The results obtained do not yet allow us to state a preference for one of the two recrystallization mechanisms; probably they both occur under different conditions.

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[1] See, for example, J. E. Burke and D. Turnbull, Progr. Metal Phys. **3**, N. Y., Interscience, 1952, p. 220. Russian transl., Usp. fiz. metallov **1**, 368 (1956).

[2] R. I. Garber, I. A. Gindin, V. S. Kogan, and B. G. Lazarev, DAN SSSR **110**, 64 (1956).

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[4] L. N. Aleksandrov and V. S. Mordyuk, FMM **12**, 249 (1961), translation: The Physics of Metals and Metallography **12**, No. 2, 77 (1961). L. N. Aleksandrov, Uchenye zapiski Mordovskogo gos. un-ta (Scientific Publications of the Mordvinian State University), 1964, No. 36, p. 67.

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