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Abstract

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THE EFFECT OF PLASTIC DEFORMATION ON THE FINE STRUCTURE OF MOLYBDENUM SINGLE CRYSTALS

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The study of high-purity single crystals of refractory metals is of considerable interest, since it makes it possible to assess more correctly various physicomechanical properties associated with the nature of the metal, excluding the influence of impurities and grain boundaries.

The high technological plasticity of single crystals of refractory metals makes it possible to obtain from them various semifinished products that find wide application in electronic devices, where metals of a high degree of purity are especially needed—metals capable of operating reliably and for long periods at high temperatures under cyclic conditions under the action of an electric current in vacuum or in alkali-metal plasma; metals that are plastic at low temperatures and resistant to recrystallization, vibration, etc. To obtain single crystals of refractory metals, an apparatus was designed and built in the Laboratory of Alloys of Refractory and Rare Metals of the A. A. Baikov Institute of Metallurgy (1), making it possible to grow single crystals of all refractory metals and alloys in the range of solid solutions by the method of zone melting with an electron beam in high vacuum.

In the present work, molybdenum single crystals were studied that had been obtained by electron-beam zone refining using a liquid-nitrogen trap from forged molybdenum rods 8 mm in diameter and 200 mm long, produced by double arc melting. The molten zone was moved from bottom to top at a rate of 5 mm/min; the vacuum was maintained at about 10^{-4}–10^{-5} mm Hg. Two passes of the zone were made.

The content in the single crystals of carbon and metallic impurities was less than 0.01% each (chemical and spectral methods), and of oxygen, hydrogen, and nitrogen—less than 0.001% (vacuum-fusion method), i.e., it was at the limit of accuracy of the analytical methods. The value of the residual electrical resistance (the ratio of the electrical resistance at 20° to its value at 4.2°K) was 900, as compared with 10–20 for vacuum-melted polycrystalline molybdenum.

The orientation of the single crystals with respect to the crystal axis was determined by the Laue back-reflection x-ray method. To study changes in the

Figure 1

Figure 1: Figure 1

Figure 2

Figure 2: Figure 2

substructure during deformation, the single crystals were rolled through grooves at room temperature with a reduction per pass of not more than 2%. The single crystals possessed high plasticity and, at ordinary temperature, withstood without fracture a total degree of deformation of more than 90%.

To reveal the microstructure, specimens of the single crystals were cut along definite crystallographic planes with an abrasive wheel 0.30 mm thick under intensive water cooling. The surface of the sections was subjected to electropolishing in a 10% aqueous alkali solution at a voltage of 30—40 V and to electrolytic etching at a lower voltage.

Figure 1a (see inset to p. 1118) shows the microstructure of a molybdenum single crystal on a plane parallel to (100); square figures are also visible there ···

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Fig. 1. Substructure of a molybdenum single crystal. 600×.
a —section plane (100), *b* —section plane (111)

Fig. 2. Change in the substructure of a molybdenum single crystal during deformation by manual rolling at room temperature. 200×.
a —initial substructure of the single crystal, crystal axis [113], *b* —deformation by 30%, $\alpha = 55°$; *v* —deformation by 40%, $\alpha = 40°$; *g* —deformation by 50%, $\alpha = 30°$; *d* —deformation by 75%, $\alpha = 10°$; *e* —deformation by 85%, $\alpha = 0°$

etching pits, which represent the points at which dislocations emerge on the polished plane. The microstructure of the (111) plane of a molybdenum single crystal is characterized by triangular etch figures (Fig. 1,b). The substructure of the plane parallel to (110) could not be revealed by this etching method. Other investigators have likewise not yet succeeded in doing this. Along with individual etch figures distributed over the entire polished section, boundaries of misorientation blocks, consisting of etch figures, were revealed. The misorientation angles are small, of the order of several minutes. The density of etch pits was of the order of 10^3 cm^{-2}, the same for the (100) and (111) planes.

The microhardness of a molybdenum single crystal was measured on the (100), (110), and (111) planes. The measurements were carried out on a PMT-3 instrument under a load of 100 g. Before measurement the polished surface was subjected to thorough electropolishing and electrolytic etching in order to remove the work-hardened layer. No hardness anisotropy was detected for the molybdenum single crystal. The following measurement results were obtained:

Polished plane	(100)	(110)	(111)
H_{μ}^{*}, kg/mm^2	157	156	157

It was of interest to study the change in the substructure of a molybdenum single crystal during deformation. The method of selective etching used in the present work makes it possible to reveal traces of active slip planes, which, as a rule, are the planes with the densest packing of atoms, i.e., $\{110\}$ for a bcc lattice.

Figure 2 (see insert to p. 1118) shows the microstructures of a molybdenum single crystal at various stages of cold deformation by rolling through grooves. On examining the micrographs one can notice the predominant arrangement of etch figures along two intersecting directions situated at a certain angle to the rolling direction. Evidently, these directions are traces of slip planes along which dislocations move during deformation; moreover, here there are two active intersecting slip systems, which is characteristic of the body-centered cubic lattice. Slip on two intersecting systems of planes was also observed by Maddin and Chen in studying the surface of stretched molybdenum single crystals (^{2}). The extent of the revealed regions of traces of slip planes depends on the number of accumulated dislocations, i.e., on the perfection of the single crystals and the degree of their plastic deformation.

It is also important to note that, with increasing degree of deformation, the position in space of the traces of the slip planes changes and approaches more and more closely the rolling direction, i.e., in the course of deformation there occurs a rotation of the slip planes in the direction of the acting force, which is very clearly visible in the microstructures (Fig. 2,b–e). This circumstance, in this form, as far as we know, has not previously been noted by anyone.

The angle α (Fig. 2,b–d) between the rolling direction (indicated in the figure by an arrow) and the position of the slip planes decreases from 55 to 10° as the degree of deformation in section increases from 30 to 75%. The change in the angle α with increasing degree of deformation can be represented by the following empirical formula: $\alpha = \frac{1}{K}(C - a)$, where α is the angle between the rolling direction and the position of the slip planes at a given degree of deformation a, C is the degree of deformation at which the slip planes are arranged along the rolling direction, i.e., when $\alpha = 0$, and K is an empirical coefficient of dimension [deg^{-1}], depending on the initial ori-

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* Microhardness values are averages of 7-9 measurements.

of the single crystal’ s orientation. In our case the formula has the form:
$\alpha = (85 - a)$ degrees.

Thus, in the process of deformation of molybdenum single crystals, they are fragmented into misorientation blocks, the boundaries of which are located in the active slip planes. With an increase in the degree of deformation, the blocks rotate in the direction of the acting force, and the misorientation of the blocks increases, which ultimately leads to the formation of a fibrous structure at a high degree of deformation.

It has been established that in a molybdenum single crystal produced by electron-beam melting, in the cast state electropolishing reveals a substructure along the planes (100), (111); along the plane (110) the substructure cannot be revealed. The etch figures have the form typical of a b.c.c. lattice: triangles for (111) and squares for (100). The hardness and density of etch pits on the planes (100) and (111) are identical: $H_\mu = 157$ kg/mm^2, and the density of etch pits is of the order of 10^3 cm^{-2}.

It has been established that in the process of cold plastic deformation of molybdenum single crystals, they are fragmented into misorientation blocks, whose boundaries are the active slip planes. During deformation the blocks rotate in the direction of the acting force; at the same time the misorientation of the blocks increases, and at deformation greater than 80% a fibrous structure is already observed.

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