FIELD-ION-MICROSCOPIC STUDY OF INTERSTITIAL PLASTICITY OF TUNGSTEN MICROCRYSTALS

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Plastic deformation of tungsten needle-like microcrystals is investigated at temperatures between 21 and 80°K. It is shown that under the action of stresses close to the theoretical tensile strength, plastic deformation related to the formation and diffusion of interstitials is possible. Disordering in microcrystals bombarded by low energy He ions is observed. The mechanism of formation of crowdions on nonconservative movement of dislocations is considered. Suppression of tungsten plasticity on lowering of the temperature to 20°K is ascribed to crowdion slowing down of the dislocations.

INTRODUCTION

INDENBOM[1] has shown that an appreciable mass transfer, connected with displacements of interstitial atoms in a crowdion configuration, is possible in crystalline materials at sufficiently low temperatures. The crowdion model of plasticity explains a number of effects accompanying plastic deformation and failure under the influence of a concentrated load under condition of limited dislocation mobility[2-4]. Low-temperature interstitial plasticity was observed in tungsten microcrystals subjected to tension at stresses close to the theoretical ultimate strength[5]. We have investigated by ion and electron field-emission microscopy the plastic deformation of tungsten in the temperature interval 21–80°K.

The investigations were made in a helium field emission microscope in which the specimens were cooled with liquid nitrogen and hydrogen. The single crystals were loaded by ponderomotive forces of an electric field; the mechanical stresses were determined from the field intensity near the investigated part of the sample surface. Some of the microcrystals were bombarded with helium ions of energy up to 2 keV to increase the concentration of the point defects. The bombardment was effected directly in the vacuum chamber of the microscope at 21°K and higher, by ions produced by interaction between the field-emission electrons and the atoms of the image-producing gas. The ion-beam parameters were calculated from the relations given in[6].

The rapid changes in the shape of the crystals were observed and registered with the aid of an electrostatic image-brightness amplifier with a current gain 10^5 and a resolution 30 lines/mm. The temperature of the needle tip was mònitored against the position of the maximum of the autoionization intensity maximum near the faces with high energy of evaporation by the field. The shift of the autoionization peak with increasing temperature T is due to the change of the conditions of the diffuse displacements of the polarized atoms of the gas towards surface sections of high curvature. The peaks shifts toward higher field intensities E in accord with the relation $E = (3k/\alpha)^{1/2}(T + \Theta)$, where α is the coefficient of polarization of the gas molecule, k is Boltzmann's constant, and Θ is the Debye temperature. The validity of this relation is confirmed by the agreement with the experimental data of Muller et al.[7] The intensity of the ion bombardment usually did not exceed 10^{-5} A/cm, so that no noticeable increase of the needle temperature was observed in the bombardment process.

After the bombardment, the samples were evaporated by the electric field until the microscopic projections produced by the ion bombardment were removed by radiation-stimulated surface self-diffusion in strong electric fields ($\sim 5 \times 10^7$ V/cm)[6,8].

RESULTS AND DISCUSSION

No traces of plastic deformation were observed in needle-shaped microcrystals with diameter less than 500 Å at the tip, in the temperature interval 21–80°K, when the electric field intensity was raised to a level correspondong to a pondermotive-force density $\sigma \approx 1500$ kgf/mm^2. Emergence of the interstitial atoms to the surface after loading by the electric field was observed in samples of large diameter and was accompanied as a rule by spoiling of the atomically smooth faceting of the needle. The series of microphotographs in Fig. 1 illustrates the change in the microtopography of a needle (diameter $d_{av} \approx 1000$ Å) during the course of exposure at $\sigma \approx 1000$ kgf/mm^2 and T = 78°K. Formation of increased-contrast bands and of helicoidal figures, which characteristic of dislocation plastic deformation, is observed on the surfaces of the samples, as well as the appearance of a large number of autoionization centers of increased brightness. The latter should be regarded as interstitial atoms[9] emerging to the surface during the course of plastic deformation.

The bombardment of the samples with helium ions of energy up to 2 keV alters radically the microstructure and the mechanical stability. Field evaporation of microcrystals bombarded with an ion flux of 10^{15}–10^{16} cm^{-2} reveals a large number of point defects. A direct determination of the defect concentration in the surface layers is made difficult by the fact that only a fraction

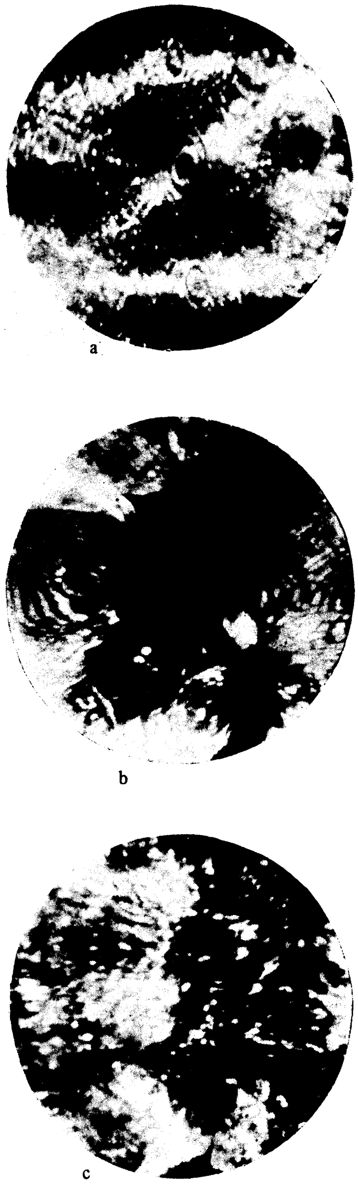


FIG. 1. Variation of the microrelief of the surface of a needle-shaped sample during the course of exposure at $\sigma \approx 1000$ kgf/mm^2, T = 78°. Micrograms a, b, and c were obtained at intervals of 30 min.

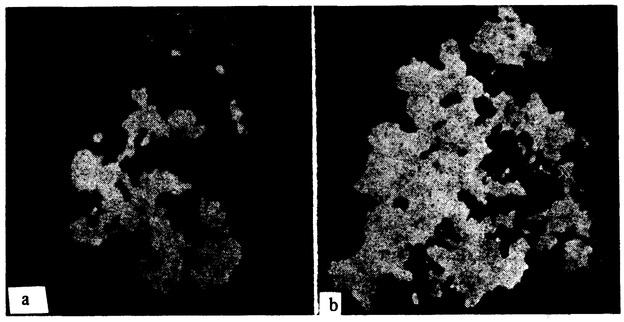


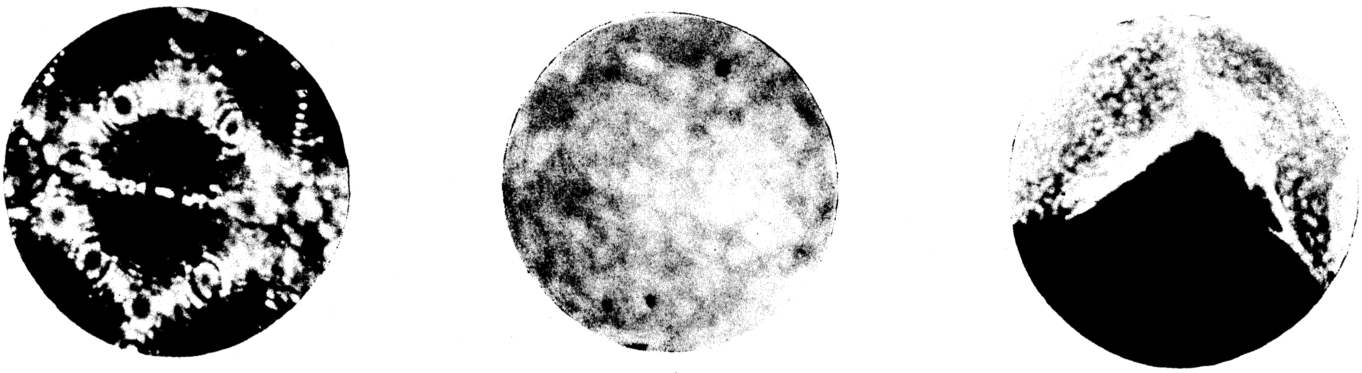
FIG. 2. Broadening of ion field emission image of bombarded tungsten microcrystal: a—microgram taken immediately after bombardment, b—after deformation at $\sigma \approx 500$ kgf/mm^2.

of the surface atoms takes part in the production of the ion field emission image. The concentration of the point defects can be estimated, following Muller[10], to be inversely proportional to the cube of the maximal Miller index of the faces formed in the evaporation of the crystal by the field. The absence of faces that are less closely packed than $\{321\}$ from the ion field emission micrograms indicates that concentration of the defects in the surface layers is higher than 10^{-2}. Raising the field intensity above the intensity of the image field in the helium ions is accompanied by the appearance of a large number of interstitial atoms and of their complexes on the surface of the needle, which are then evaporated by the field. The intensity of this process decreases somewhat when the field intensity is lowered to the auto-ionization threshold of helium ($E \approx 300$ mV/cm), corresponding to a stress 500 kgf/mm^2. In a better image field ($\sigma \approx 1000$ kgf/mm^2) the change in the sample shape is manifest in a jumplike broadening of the image (Figs. 2a and 2b). Such a process can be interpreted as a collective emergence of a large number of atoms to the surface, followed by evaporation of the most strongly projecting sections.

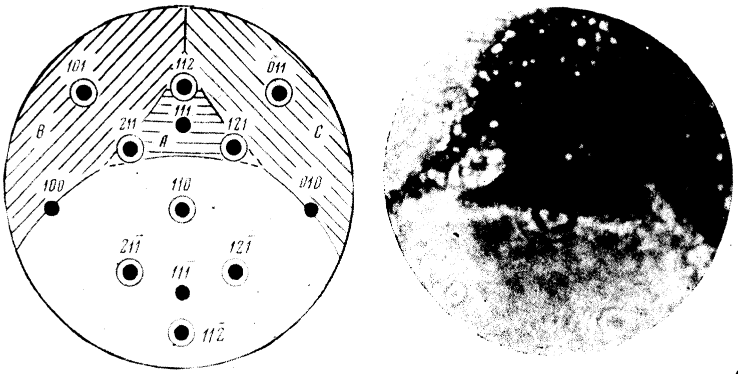
In those cases when the sample was deformed by the field after the removal of the microscopic projections and formation of an atomically-smooth surface, a shape change of crystallographic character is observed. Figure 3 shows a series of field-emission micrograms illustrating the change in the shape of an ideal single crystal (Fig. 3) after bombardment with a 10^{16} cm^{-2} flux of helium ions of ~1.5 keV energy; raising the working stress to a level close to the evaporation value ($\sigma \approx 1500$ kgf/mm^2) is accompanied by a jumpwise displacement of the part of the microcrystal bounded by the band-line segments $[01\bar{1}]$ and $[10\bar{1}]$ (Fig. 3c). The formation of a projection is revealed by the increase of the ion emission contrast and the loss of regularity of the microtopography of part of the sample surface. The region experiencing the projection is shown shaded on the stereographic projection (Fig. 3d). An analysis of the microphotographs shows that the produced microscopic projection has a partially faceted shape with a vertex near the emergence of the [111] pole. The projections of the edges on the surface are oriented approximately along the lines $[01\bar{1}]$, $[\bar{1}10]$, and $[10\bar{1}]$, which intersect at the [111] pole.

Since the surface reveals none of the jogs usually produced in dislocation plastic deformation of microcrystals[11], the deformation should be of interstitial character. The partial faceting of the microprojection means that the interstices emerge to the surface anisotropically. This can be explained, in analogy with the explanation of Rozhanskiĭ and Velednitskaya[4], as being due to the one-dimensional character of the crowdian displacement along the close-packed directions. It should be noted, however, that the anisotropy can also be connected to a considerable degree with the inhomogeneity of the elastic deformation of the needle. The elastic-deformation energy per atom of a sample located in a field of intensity E is approximately equal to[9]

$$W = (\Omega / 128\pi) E^4 / K,$$



a b c



d e

FIG. 3. Crowdion deformation of tungsten microcrystal at 80°K: a—defect-free crystal prior to bombardment, b—during the course of bombardment with helium ions, c—image and d—stereographic projection of microcrystal after deformation, e—after evaporation of 10 atomic layers by the field.

where Ω is the atomic volume and K is the modulus of hydrostatic compression. For tungsten in the best-image field (E = 500 mV/cm), the energy is W = 0.4 eV, amounting to ~50% of the activation energy of the interstice migration to recovery stage I [12]. Consequently, the ponderomotive forces of the electric field may exert an appreciable influence on the mobility of the interstices. This assumption is favored, in particular, by the fact that the most prominent sections of the produced projection (Fig. 3c) lie in regions corresponding to the maximum local electric field intensity at the surface of the undeformed crystal.

The interstice distribution revealed during the process of field evaporation is not uniform. The concentration of the interstitial atoms in the regions experiencing a displacement is much higher than the mean value and is conserved when 10^{12} atomic layers are evaporated by the field. In the undeformed region, the interstice concentration decreases by one order of magnitude when about ten (110) layers are removed. The inhomogeneity of the defect distribution is not due to the non-uniformity of the ion flux, as is evidenced by the relative homogeneity of the distribution of the ion field emission over the surface of the needle (Fig. 3b). It should be assumed that the increased interstice concentration in the region B – A – C had occurred in the course of the deformation. When layer after layer were evaporated by the field, we observed in this region a preferred removal of the sample material (Fig. 34), apparently as a result of the decrease in the density by supersaturation with vacancies when the interstices are formed.

Cooling the samples with liquid hydrogen (T ≈ 21°K) strongly increases the mechanical stability of the bombarded microcrystals. Comparison of the micrograms obtained with electron field emission during the bombardment (Fig. 4a) and with ion field emission after raising the voltage until the best image field was produced (Fig. b) shows that at T = 21°K there are no significant changes in the morphology of the surface while the stress is being raised to ~1000 kgf/mm^2. We can thus conclude that at hydrogen temperature no displacement of interstitial atoms through distances comparable with the dimensions of the investigated microcrystals (~10^3 Å) are produced.

The results confirm the previously assumed[3] crowdion diffusion produced when needle-like microcrystals are loaded at low temperature. The temperature region in which crowdion displacement was observed in the present study corresponds to recovery stage I [12,13]. The interstitial atoms that are not in crowdion configurations apparently migrate at higher temperatures. The agreement between the calculated

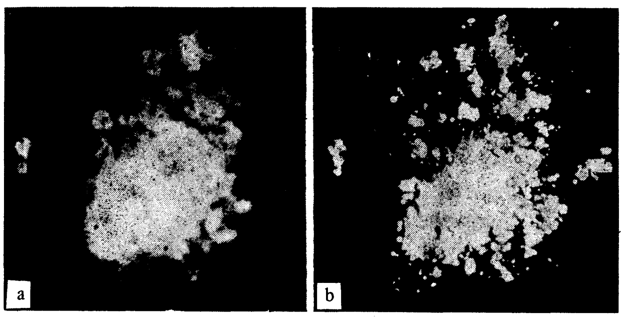


FIG. 4. Electron (a) and ion (b) field emission pictures of a crystal bombarded at 21°K.

values and the data obtained in[14] on the concentration of the displaced atoms produced when tungsten is bombarded with high energy electrons at a temperature 300°K indicates that there is no noticeable migration of these atoms at temperatures below recovery stage III.

On the basis of the ion field emission microscopy data on the dependence of the mechanical properties of tungsten crystals on the singularities of their microstructure, it was of interest to analyze the possible mechanisms of crowdion formation.

When tungsten is bombardment with helium ions of energy E_{He}, the maximum number of interstitial atoms produced in interactions with individual ions is

$$\frac{2Mm}{(m+M)^2}\frac{E_{He}}{E_d},$$

(where M and m are the masses of the tungsten and helium atoms, $E_d \approx 50$ eV is the threshold displacement energy) and reaches a value 2 at $E_{He} \leq 2$ keV, i.e., bombardment with low-energy ions leads to the appearance of only single defects. Thus, depleted bands and dynamic crowdions cannot be produced at energies close to the threshold displacement energy[15]. It must be assumed that only metastable crowdions can be produced in the irradiation process at relatively low momentum transfers close to the displacement threshold.

Low-temperature plastic deformation of the microcrystals are observed in very small volumes (10^{-18}–10^{-15} cm^3), and it is difficult to assume that in the investigated cases the interstitial atoms are produced at dislocation intersections. It is most probable that the interstices (including crowdions) are produced in nonconservative displacements of dislocations. Then the absence of traces of interstitial plasticity in microcrystals with diameter less than 500 Å can be attributed to their freedom from dislocations, due to the emergence of the dislocations to the surface under the action of the image forces. The site displacement that is characteristic of crowdions is realized in the cross section of the core of the dislocation jog relative to half of the nearest close-packed chains. The nonconservative displacement of a dislocation with jogs is revealed by the tangential displacement of the interstices in the crowdion configuration along the Burgers-vector direction. As it moves, such a dislocation leaves behind it a parabolic loop of crowdions that move one-dimensionally in the close-packing direction. The shape of the crowdion loop and its decelerating action on the dislocation depend on the crowdion migration mobility. Assuming that the crowdion energy in the dislocation field is proportional to the hydrostatic component of the stress[13], we can show that a dislocation oriented at an angle ψ to the direction of the Burgers vector b and moving with velocity v is acted upon by a crowdion deceleration force equal to

$$F(T)=\frac{\mu(1+\nu)\Delta V\sin^2\psi}{3\pi(1-\nu)bd}\sum_{n=1}^{\infty}\left[n+\pi^2\frac{D_0}{av}\frac{\sin^2\psi}{\exp(E_m/kT)}\right]^{-2}.$$

Here μ is the shear modulus, ν the Poisson coefficient, ΔV the lattice volume deformation due to the crowdion, a the lattice parameter, D_0 the crowdion diffusion constant, and E_m the crowdion migration activation energy. This relation describes quasi-quantitatively the anomalous behavior of the flow limit of tungsten single crystals in the temperature interval $4°K \leq T \leq 120°K$[16]. The calculated crowdion strengthening coefficient $F^{-1}dF/dT$ coincides with the experimentally determined strengthening coefficient if one substitutes in the expression for F(T) the value $E_m = 0.08 \pm 0.02$ eV, which is equal, within the limits of experimental error, to the activation energy of the low-temperature interstice migration[12]. The latter confirms indirectly the assumption that interstices (crowdions) can be produced during the course of low-temperature dislocation displacement.

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