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Optimal deformation and ion irradiation modes for production of a uniform submicrograin structure in molybdenum

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Transmission and scanning electron microscopy have been used to investigate the structure of molybdenum after complex processing: deformation and irradiation with accelerated argon ion beams. The analysis of the effect of deformation and radiation parameters on the structural transformations and mechanical properties of molybdenum allowed us to find the optimal mode of processing to form a submicrograin uniform-in-size structure.

Keywords: high pressure torsion; irradiation; molybdenum; structure

1. Introduction

Nanocrystalline and submicrograin (SMG) materials are known to possess improved physical, chemical, mechanical, and other properties.[1] To produce such materials, complex processing that combines severe plastic deformation (high pressure torsion (HPT), molding, or equal-channel angular extrusion) and subsequent annealing is often used.[2,3] Annealing solves the problem of transition of non-equilibrium deformation-induced nano- and submicrocrystalline (SMC) structures into stable recrystallized SMG structures, but with a possible grain growth. For example, the SMC structure in a polycrystalline Mo has been formed by HPT at both room temperature and 400°C; the structural element sizes in these cases were strongly different and were 60 nm [4] and 450–220 nm,[5] respectively. However, despite the larger size of microcrystallites, the structure formed during deformation at 400° [5] was found to be more stable during subsequent heating than the structure after room-temperature deformation. The grain size of the SMC structure formed during room-temperature deformation increased by the order after recrystallization at 900°C in contrast to the structure formed during 400° deformation, the size of which changed slightly.

It is known [6] that the rates of nucleation and grain boundary migration are responsible for grain size in a recrystallized structure. For structure refinement, it is necessary to maximally increase the density of recrystallization nuclei and provide a low rate of migration of grain

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boundaries. The SMC structure formed during HPT deformation consists of microcrystallites, which present ready recrystallization nuclei (dislocation-free regions surrounded by high-angle boundaries). In addition to the microcrystallites, new nuclei of recrystallization can appear during heating. To increase the number of deformation-induced centers (microcrystallites) we may reduce the deformation temperature. It is known that a decrease in the deformation temperature facilitates structure refinement, for example, in nickel (average grain size of 80 nm after cryodeformation and 140 nm after room-temperature deformation).[7] However, a decrease in the temperature deformation can activate twinning, which retards the formation of a uniform-in-size SMC structure, which was observed in two last works [7,8] dedicated to Ni and Fe. We have also supposed that the deformation of Mo at a temperature of liquid nitrogen will not be accompanied by twinning, as compared with Ni and Fe, because of the high stacking fault energy ~ 300 mJ m^{-2},[9] so we will obtain a homogeneous structure composed of microcrystallites, and the low temperature of deformation will result in maximum structural refinement down to the nanoscale range. The formation of a homogeneous SMC structure upon deformation is a necessary condition.

New recrystallization nuclei can be obtained during post-deformation processing not only by the thermally activated way using annealing, but also by radiation processing. In a number of papers,[10,11] it is shown that the structure in deformed materials undergoes recrystallization under irradiation with argon ions at a depth of a few millimeters. This is much greater than the penetration depth of ions. The authors explain this phenomenon by radiation-dynamic effects caused by the generation and propagation of post-cascade shock waves in the material.[12]

The most promising annealing method seems to be the radiation one, which is different from common heating by its dynamic component of action. Radiation annealing allows recrystallization processes to occur at lower temperatures and in record time,[10,11] as compared with isothermal furnace annealing. In addition, it should be noted that the furnace annealing may result in the formation of a non-uniform-in-size structure due to the preferential growth of individual grains.[4]

Our study is aimed at finding the optimal deformation and ion-beam-processing modes to obtain the most possible uniform-in-size and fine SMG structure in molybdenum. Deformation has been performed at two temperatures: 290 and 80 K to form an ultrafine structure.

2. Experimental

All the disc-shaped samples of monocrystalline Mo under study were 5 mm in diameter and 0.3 mm thick. The diametral plane of the samples was parallel to the (110) crystallographic plane. Deformation was performed by shear under pressure (12 GPa) in Bridgman anvils at temperatures of 80 and 290 K. The angle of the anvil rotation was varied from 15° to 15 revolutions. The thicknesses of the samples measured after deformation were within 0.09–0.11 mm. The true strain (e) was calculated taking into account both torsion and upsetting deformation:

$$e = \ln\left(1 + \left[\frac{\varphi R_i}{h_{iR}}\right]^2\right)^{1/2} + \ln\frac{h_0}{h_{iR}}, \tag{1}$$

where φ is the angle of the anvil rotation**;** h_0 and h_{iR} are the thickness of a sample before and after deformation at a given distance from the center of a sample R_{i}. This method of calculation was described in detail in [13]. The error in the determination of the true strain was $\Delta e = \pm 0.2$, because of the variability of the sample thickness at the same distance from the center. In the case of room-temperature deformation, the true strain was varied in the range from 0.2 (upsetting) to

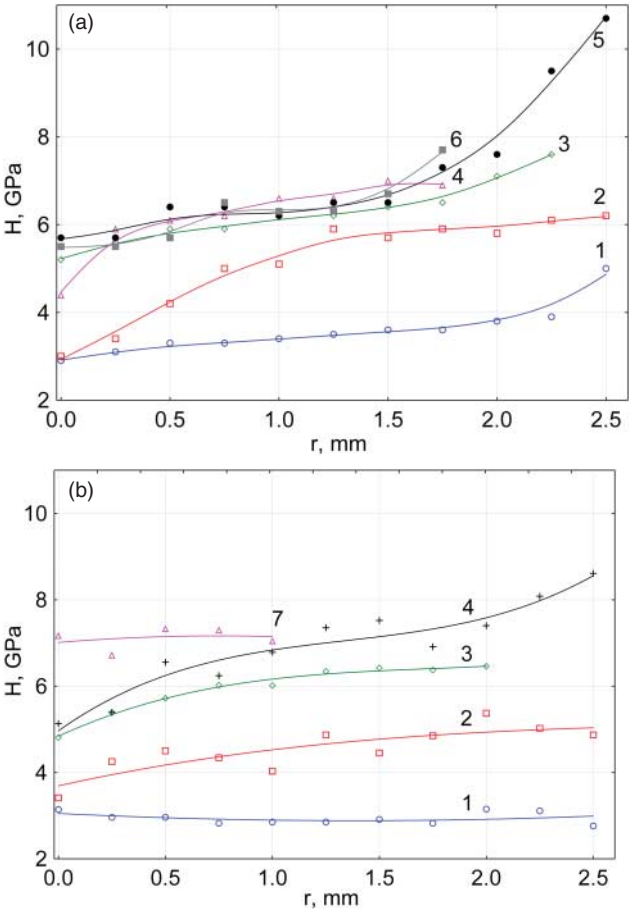

Figure 1. Microhardness of Mo vs. sample radius: (a) 290 and (b) 80 K. Rotation angle of the anvil: (1) 15°, (2) 180°, (3) 1 rev., (4) 3 rev., (5) 5 rev, (6) 10 rev, and (7) 1.5 rev.

9.6 (15 revolutions, $R = 2.5$ mm); in the case of cryodeformation, from 0.3 (upsetting) to 7.3 (3 revolutions, $R = 0.5$ mm).

The hardness of molybdenum after deformation was measured by the Vickers method along the radii of the samples (Figure 1) under a load of 0.5 N, using a PMT-3 hardness testing machine. The load was taken so that the diagonal of an indentation does not exceed one-third of the thickness of a sample after deformation and radiation annealing.[14] In constructing, the true strain dependence of hardness, its values taken from different samples were averaged over the intervals of the true strain $\Delta e = 0.4$.

Samples deformed to three revolutions ($e = 6.5$, the average grain size $d_{av} = 0.16$ μm) at 290 K were irradiated in a continuous mode using an ILM-1 ion implanter with a glow-discharge source with a cold hollow cathode PULSAR-1M.[15] The implanter emitted an accelerated Ar^+ ion beam which was ~ 100 cm^2 in its cross section. Irradiation parameters were as follows: ion energy $E = 20$ keV, ion current density $j = 300$–400 μA cm^{-2}, irradiation dose $D = 8 \times 10^{16} - 2.4 \times 10^{18}$ cm^{-2}. The sample temperature (Figure 2) was monitored with the help of a thin chromel-alumel thermocouple welded to an identical test sample. The error of temperature measurement was ± 5 K. The following four modes of irradiation were used: (1) $j = 400$ μA cm^{-2}, $D = 8 \times 10^{16}$ cm^{-2}, ion-beam heating to $T = 1070$ K for 30 s (without holding at this temperature); (2) $j = 300$ μA cm^{-2}; $D = 2.4 \times 10^{18}$ cm^{-2}, ion-beam heating

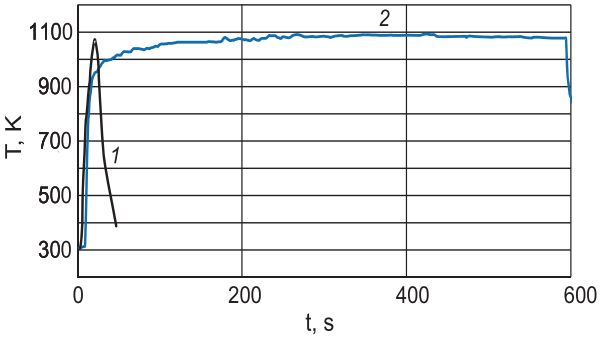

Figure 2. Temperature–time curves of Mo-deformed samples irradiated with Ar^+ ions: (1) $j = 400\ \mu A\ cm^{-2}$ without holding and (2) $j = 300\ \mu A\ cm^{-2}$ with holding.

to $T = 1070$ K for 2 min and holding at this temperature for 18 min; *(3)* $j = 400\ \mu A\ cm^{-2}$; $D = 1.7 \times 10^{18}\ cm^{-2}$, ion-beam heating to $T = 1170$ K for 100 s and holding at this temperature for 9 min and (4) $j = 300\ \mu A\ cm^{-2}$, $D = 1.3 \times 10^{18}\ cm^{-2}$, ion-beam heating to $T = 1070$ K for 2 min and holding at this temperature for 9 min.

Transmission electron microscopy (TEM) was performed on a JEM-200CX microscope; scanning electron microscopy (SEM) was performed on a PHILIPS SEM-515 microscope at the Center of Electron Microscopy of the Testing Center for Nanotechnologies and Advanced Materials of the Institute of Metal Physics, Ural Branch, Russian Academy of Sciences. The structure was investigated at a distance of 1.5 mm from the center of a sample. TEM examination was carried out in a section that was parallel to the irradiated surface and at a distance of 50 µm from it. SEM examination was performed in a cross section that was parallel to the irradiated surface and at a distance of 50 µm from it. The sizes of such structural elements as dislocation cells and microcrystallites were determined from bright- and dark-field images using at least four hundred measurements, the error being no more than 10%. Statistical analysis was performed using a STATISTICA 10 software.

3. Results and discussion

In the beginning of the work, we selected the deformation conditions (temperature and true strain), which allow us to obtain a quite homogeneous SMC structure.

Measuring the hardness of molybdenum deformed at different temperatures (Figure 3), electron microscopic examination showed stages in structure development. The hardness curve was constructed as a function of the square root of the true strain, since such a method described in [16] allowed stages of strain hardening to be distinguished. Two stages of deformation with different hardness-growth factors (which correspond to the inclination of the straight lines in Figure 3) can be identified from the dependence of the hardness on the square root of the true strain at both 80 and 290 K: $k_1^{290} = 0.7$ GPa, $k_2^{290} = 2.9$ GPa at 290 and $k_1^{80} = 0.4$ GPa, $k_2^{80} = 1.1$ GPa at 80 K. It is seen from the figure that molybdenum at 80 K is hardened less than at 290 K up to $e = 4$; the opposite is true at higher values of true strain. Since hardening is accompanied by structural refinement, we expected that the structure formed after deformation with $e > 4$ at 80 K would be finer than that formed after room-temperature deformation. Indeed, within the range $e = 3$–6, the average size of structural elements, which formed after 80-K deformation, is smaller ($d_{av} = 0.12$ µm) than that formed after room-temperature deformation ($d_{av} = 0.18$ µm). Further deformation at room temperature up to $e = 9$ results in a gradual decrease in the average

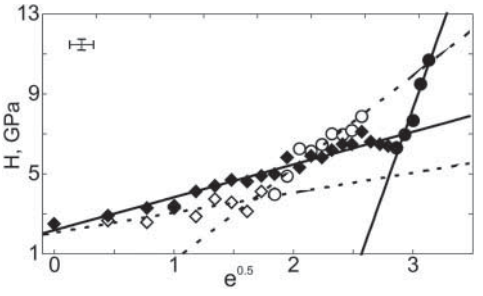

Figure 3. Microhardness of Mo deformed at two different temperatures as a function of $e^{0.5}$: (♦) first stage, 290 K; (●) second stage, 290 K; (◊) first stage, 80 K and (○) second stage, 80 K.

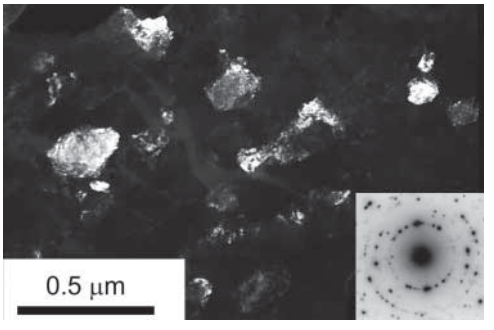

Figure 4. Structure of Mo after deformation to three revolutions ($e = 6.5$) at 290 K. Dark-field image in reflection (110) and electron diffraction patterns.

size of structural elements down to 0.13 μm. Unfortunately, deformation and respective possible structural refinement were limited by brittleness of molybdenum, especially at 80 K.[17,18] At this temperature, we managed to deform the samples to a maximum of three revolutions of the anvil. Further deformation led only to the destruction of samples. This can be explained by a strong temperature dependence of the yield stress of refractory bcc metals.[19] Moreover, even after the maximum true strain achieved under cryogenic deformation, the structure of Mo remained nonhomogeneous, namely, elements with low-angle misorientations existed along with microcrystallites with high-angle misorientations. In the case of room-temperature deformation, samples deformed for three revolutions of the anvil at 290 K ($e = 6.5$) were good for further processing and investigation (without cracks that tended to grow during subsequent grinding and electropolishing). The structure in this case is mainly composed of microcrystallites with an average size of 0.14 μm (Figure 4). The microcrystallites are ready recrystallization nuclei and the growth of which can be stimulated by further processing. Therefore, samples having such a structure would be appropriate for subsequent irradiation.

Accelerated-Ar^+-ion irradiation of the samples with SMC structures in different modes led to a decrease in microhardness (Figure 5). Short-term heating to 1070 K (mode 1) caused the smallest decrease in hardness by 1 GPa, but it was not sufficient to complete recrystallization. An increase in the dose of irradiation reduced the hardness by 2–2.5 GPa. The hardness values of the irradiated samples (see Figure 5, curves *3–5*) are within the error. The greatest decrease in hardness was observed after the following irradiation mode 4. As a result of this ion-beam irradiation, primary recrystallization was fully completed at stationary temperature $T = 1070$ K for ~ 9 min, whereas the period of time (heating time) during which the temperature was growing to the above level was 2 min. An average grain size of the formed uniform-in-size SMG structure was $d_{av} = 0.45$ μm (Figure 6).

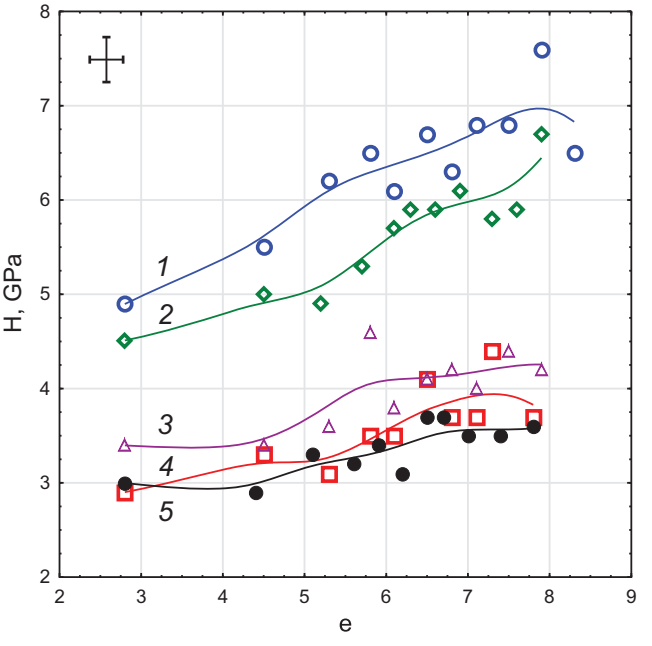

Figure 5. Microhardness of Mo after various modes of processing (deformation plus subsequent irradiation with Ar^+ ions with energies of 20 keV): (1) after deformation at 290 K; (2) mode 1; (3) mode 2; (4) mode 3 and (5) mode 4.

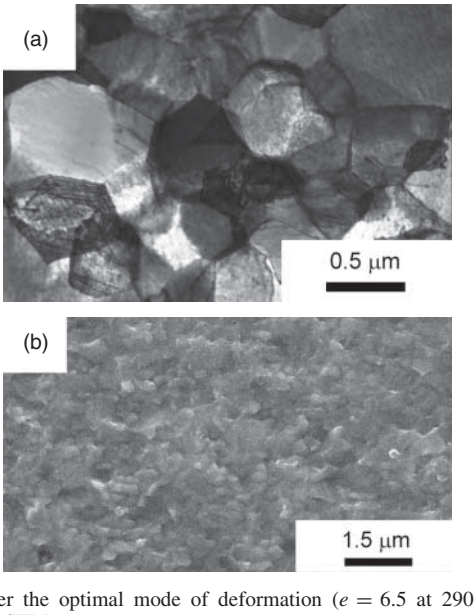

Figure 6. Structure of Mo after the optimal mode of deformation ($e = 6.5$ at 290 K) and irradiation (mode 4): $d_{av} = 0.45$ μm; (a) TEM and (b) SEM.

Uniformness of the structure, from our point of view, is one of the criteria for selecting the optimum mode for molybdenum radiation processing. It is characterized by the coefficient K of variation of linear grain sizes. The coefficient of variation is determined from the size distribution

of structural elements according to the following formula:

$$K = \frac{\sigma}{d_{\text{av}}},$$

where σ is the standard deviation of the average structural element size and d_{av} is the average size of structural elements. If the value of K is close to 0.5, this means that recrystallization during annealing proceeds on the normal growth kinetics and the structure in this case is considered to be quite uniform, as it does not contain nuclei of the secondary recrystallization. If the value of K is close to 1, the abnormal grain growth is observed in the material.[20] The smaller the variation coefficient, the more uniform the structure in size. In our case $K_{\text{def}} = 0.75$ after room-temperature deformation, that is, the structure does not contain secondary recrystallization nuclei and is not perfectly uniform. Irradiation at 1070 K with additional holding at this temperature for 9 min (mode 4) yields the optimal value of the coefficient $K_{\text{opt}} = 0.38$. Irradiation for 18 min at the same temperature (mode 2), despite the fact that the structure retains to be uniform-in-size $d_{\text{av}} = 0.42$ μm ($K_{1073} = 0.43$) and is of the required type, results in pulling out the material from the surface of the sample and material degradation. Irradiation of the deformed samples at a higher ion current density $j = 400$ μA cm^{-2} and a dose $D = 1.7 \times 10^{18}$ cm^{-2}, which provide heating to a stationary temperature of 1170 K for 100 s and holding at this temperature for 9 min (mode 3), resulted in structural non-uniformity due to an abnormal growth of individual grains. The average grain size increases to 1.5 μm, wherein individual grains achieve 10–12 μm in size, this corresponds to coefficient of variation of the linear dimensions $K_{1170} = 1.3$.

As a result, the optimum was established to be room-temperature deformation with true strain $e = 6.5$ and subsequent ion-beam irradiation on mode 4. Figure 7 shows the size distribution of the structural elements (a) after deformation and (b) the optimal mode of processing. It can be seen that the average grain size increases not only due to the growth of the coarse grains, but mainly due to a size increase in the finest structural elements (the value corresponding to the minimum grain size is shifted to the right). As a result, uniformity of the structure reaches

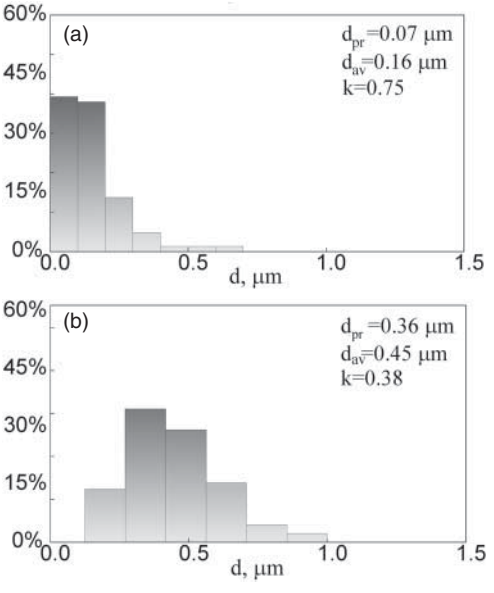

Figure 7. Size distribution diagrams of structural elements: (a) after deformation ($e = 6.5$) and (b) after subsequent irradiation mode 4.

a higher possible level than at common recrystallization,[4] which takes place during furnace annealing.

It should be noted that isothermal annealing in a furnace at 1070 K for 2 h did not cause the beginning of recrystallization in molybdenum, whereas a completely recrystallized structure with an average grain size of 0.8 μm in molybdenum was obtained at 1323 K after 1 h.[4] Such a repeatedly accelerated recrystallization process upon irradiation and a decrease in the temperature at which it occurs in molybdenum can be explained on the basis of the above-mentioned concepts of the radiation-dynamic effect of corpuscular radiation on metastable medium.[12] There is a number of papers [21–27] showing that ion bombardment decreases the temperature and increases the rate at which structural and phase transformations occur at a depth many times greater than the projected range of ions in metastable media. It should be noted that the effect of an ion beam, which is accompanied by heating of a sample, is not equivalent to common thermal heating. Indeed, it is shown in [25] that despite a good agreement between temperature–time curves $T(t)$ of Fe–15 at %Cr alloy samples (30 μm thick) heated either by a homogeneous light beam or by homogeneous Ar^+ ion beam for an irradiation time of 40 s to a stationary temperature of 450°C, the rate of short-range order formation under ion-beam exposure is much greater than that under light beam exposure.

4. Conclusions

The deformation of Mo at 80 K did not result in the formation of a uniform-in-size SMC structure, because of the molybdenum brittleness. Therefore, 290 K is the optimal temperature of deformation. At this temperature, we managed to obtain unbroken samples after deformation to tree revolutions of the anvil. In this case the sufficiently uniform SMC structure was formed at $e = 6.5$ with an average microcrystallite size of 0.14 μm and the coefficient of variation of linear sizes $K_{\text{def}} = 0.75$.

It was established that radiation annealing of the samples deformed at 290 K to $e = 6.5$ caused structure recrystallization at lower temperature and in record time as compared with furnace annealing. A decrement in the recrystallization temperature was 300 K.

The analysis of the dependencies of structural transformations occurring in molybdenum on the true strain and radiation parameters allowed us to find the optimal mode of processing: HPT deformation at room temperature to $e = 6.5$ and subsequent ion-beam irradiation (mode 4) that provides heating to 1070 K for 2 min and includes holding at this temperature for 9 min. This optimal mode of processing ensures the formation of an SMG uniform-in-size structure with an average grain size less than 0.5 μm.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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