EFFECT OF CYCLIC DEFORMATION ON THE DISLOCATION STRUCTURE AND MECHANICAL PROPERTIES OF MOLYBDENUM, CHROMIUM, AND TUNGSTEN

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The electron-microscopic study of some fcc metals and alloys in fatigue tests showed that under certain conditions after alternating loading a mosaic structure arises [1]. The formation of such a structure improves the strength and plasticity of metals, and consequently has a large practical value.

The purpose of the present work is to investigate the effect of low-frequency elastic vibrations on the dislocation structure and mechanical properties of molybdenum, chromium, and tungsten.

For the generation of low-frequency vibrations of 50 cps we devised a method which allows us to obtain maximum amplitude of the alternating stress (up to 100 kg/mm^2) in a consol-bending configuration. Samples of the investigated materials, cut out of 1-1.5 mm thick rolled sheets, had the dimensions 1 × 5 × 55 mm. Under uninterrupted cyclic loading the fatigue fracture of the samples occurred after 10^3-10^4 cycles. The number of cycles prior to fracture depended both on the stress amplitude and on the testing temperature. The cyclic loading tests were done in the 20-800°C temperature range. Because of this, the tests were carried out in vacuum (10^{-4} mm Hg). In the console configuration the maximum bending moment, and consequently the maximum normal stresses, act at the surface of the sample in a region of about 10 mm near the line separating the vibrating and fastened parts of the sample. The deformed region was easily observable on the polished surface thanks to the large number of slip lines.

The results of a microhardness study of molybdenum samples as a function of stress amplitude and duration and temperature (T_{cl}) of cyclic loading are shown in Fig. 1. The microhardness of the surface layer of molybdenum (after grinding to a depth of 20 μ) increases with increasing stress amplitude according to the curve of Fig. 1a. If the duration of cyclic loading increases while the stress amplitude is kept constant (Fig. 1b), a saturation takes place in the microhardness after exposure for 30 sec.

Let us consider the curve of the microhardness of a sample cyclically loaded at a stress amplitude of

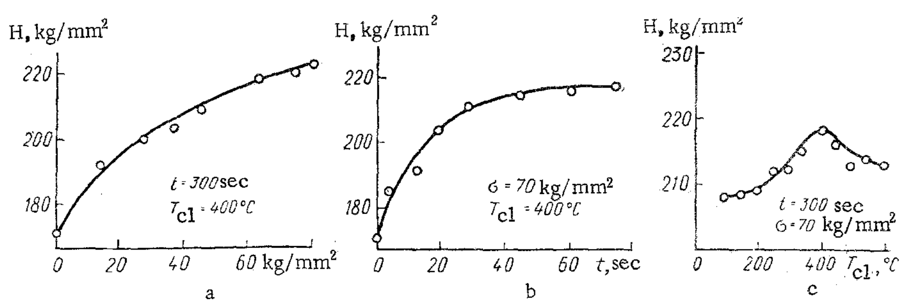

Fig. 1. Dependence of the microhardness on the amplitude (a), duration (b), and temperature (c) of cyclic loading.

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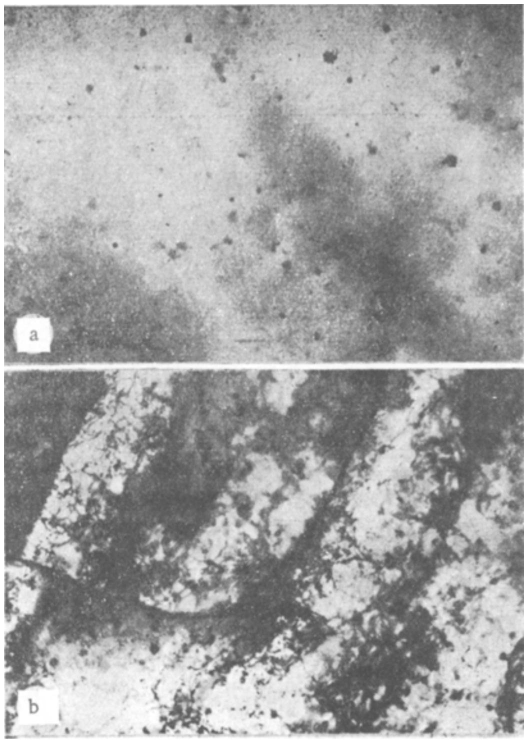

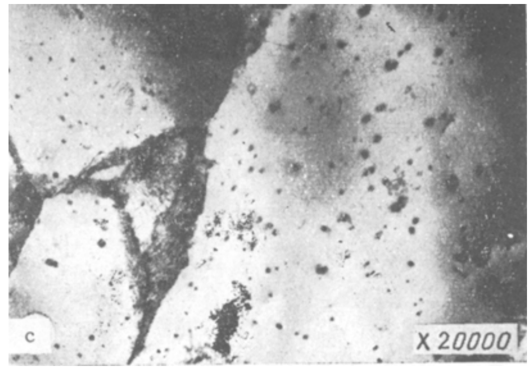

Fig. 2. Dislocation structure of MT grade molybdenum after recrystallizing anneal at 1400°C and cyclic loading at 400°C: a) initial state; b, c) after exposure for 15 and 200 sec, respectively.

70 kg/mm^2 for 30 sec at various temperatures (Fig. 1c). The highest microhardness is registered in the samples loaded at 400°C. For molybdenum this temperature range is characterized by the second stage of recovery with an activation energy of 1 eV [2]. The increase of microhardness in this range is probably due to a redistribution of point defects vacancies and interstitial atoms) and the formation of stable atmospheres around the dislocations.

Figure 2 shows the electron-microscopic image of MT grade molybdenum after recrystallizing anneal at 1400°C for 1 h and cyclic loading at 400°C with a stress amplitude of 70 kg/mm^2 for various durations. As is seen from Fig. 2b, c, there is a reorientation of the mosaic structure. With increasing time of exposure the misorientation of neighboring subgrains increases, and at the moment of fatigue

TABLE 1

Sample No.	Treatment		T_{cl}, °C	σ_u, kg /mm^2	$\sigma_{0.2}$, kg /mm^2	Bending deflection	Notes
	exposure, sec	stress amplitude, kg/mm^2					
1	Anneal at 1600°C	2 h (initial)	—	49,8	57,3	1,3	Fracture at T_x =80-100°C
2	1,2	70	600	47,6	56,8	1,0	The same
3	2,3	70	600	47,8	57,0	1,4	" "
4	4	70	600	51,5	58,5	0,9	" "
5	5	70	600	55,0	62,5	0,95	" "
6	8	70	600	52,5	61,4	0,7	" "
7	15	70	600	52,4	71,3	0,6	Fracture at T_x = 50°C
8	15	70	400	88,0	106,0	3,1	The same
	(Polished and annealed at 800°C, 1 h)						
9	(Polished and annealed at 800°C, 1 h)		400	82,4	96,0	2,0	" "
10	15	800°C, 1 h)	400	102,0	111,0	0,9	" "
	(Polished and annealed at 800°C, 1 h, twice)						
11	(Polished and annealed at 800°C, 1 h, twice)		400	95,5	108,0	2,0	" "
12	15		400	113,2	127,4	1,8	" "
	(Polished and annealed at 800°C, 1 h, thrice)						
13	Deformed by rolling at 950°C, 60%		400	92,0	94,0	8	Bent at T_x=80° C
14	Deformed by rolling at 950°C, 90%		400	112,0	125,0	8	T_x=120° C

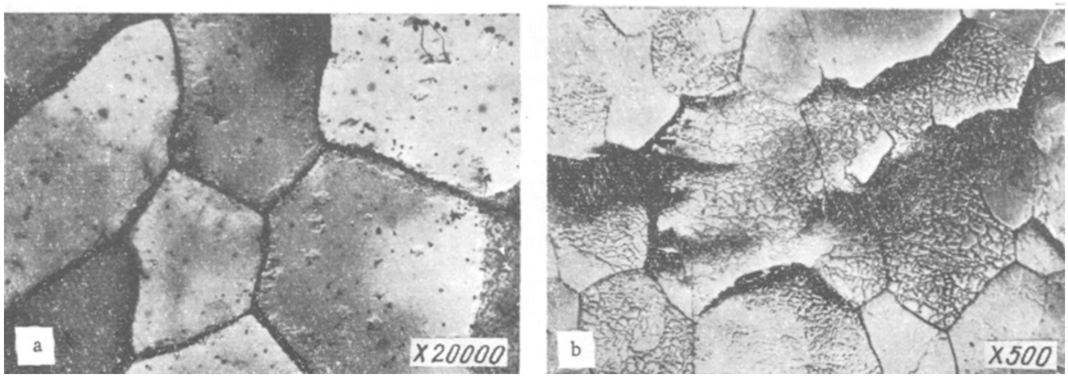

Fig. 3. Mosaic structure arising upon uninterrupted cyclic loading at 500°C: a) 90 sec exposure; b) microcracks in the surface layer of a sample after cyclic loading in a similar range.

fracture it reaches a considerable value. The azimuthal misorientation of neighboring subgrains, according to microdiffraction data, amounts to 1-3°. The same order of magnitude is found for the misorientation determined by the dark-field method in reflection perpendicular to the inclination axis of the goniometer objective stand. The subgrain size is rather nonuniform and fluctuates in the 1.5-5 μ range.

The investigation of the effect of temperature on the substructure formation showed that mosaic formation is observed in a wide temperature range — from 200 to 800°C. The most distinct subgrain boundaries were obtained after cyclic loading in the 400-500°C range. Above these temperatures the hardening processes evidently do not reach completion, and at the given deformation amplitude a distinct mosaic structure cannot always form.

The structural changes upon increasing the duration of cyclic loading are qualitatively similar to the ones taking place under "hot" plastic deformation [3, 4]. Unfortunately, the formation of misoriented mosaic structure under cyclic loading usually takes place at deformation amplitudes and exposures sufficient for the formation of microcracks (Fig. 3a). They are clearly visible in the surface layer on a previously polished surface (Fig. 3b). This circumstance imposes a limitation on the possibility of further effective hardening upon uninterrupted cyclic loading.

The results of static bending tests on the cyclically loaded samples showed that the hardening increases with increasing exposure (see Table 1, samples 2-7). For comparison, the data of mechanical tests on rolled polycrystalline MT grade molybdenum are given in Table 1.

As is seen, after alternating loading for 15 sec the values of the strength parameters σ_u and $\sigma_{0.2}$ are comparable with the hardening of molybdenum deformed to about 60% by rolling. Further treatment, including an intermediate anneal at 800°C for 1 h, electropolishing the surface layer in which microcracks

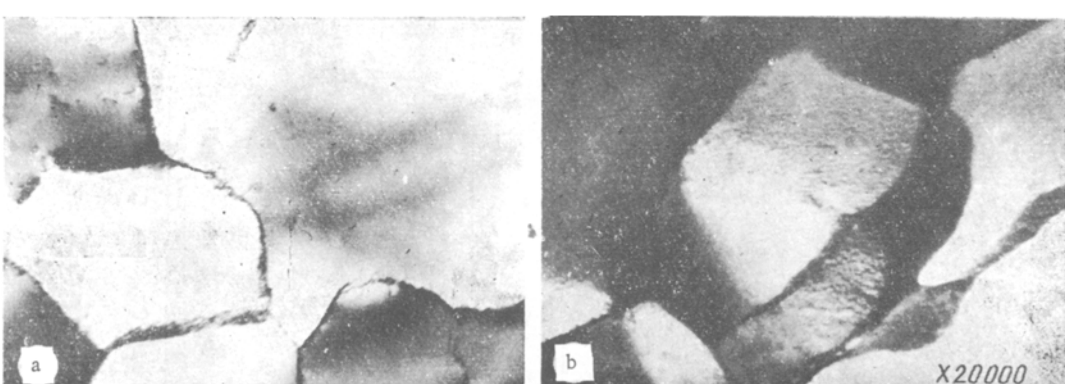

Fig. 4. Dislocation structure arising in molybdenum upon an intermediate anneal at 800°C after a single (a) and three-fold (b) cyclic loading.

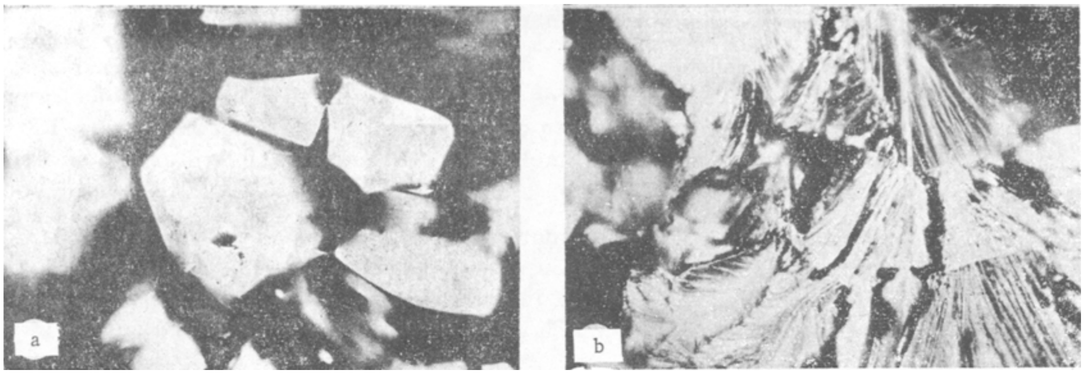

Fig. 5. Fractures on samples tested below the temperature T_X: a) initial state; b) after cyclic loading (×300).

are observed, and a second cyclic loading of the molybdenum sample resulted in a substantial hardening of the polycrystals. Similar hardening is observed only at deformation of $\varepsilon \approx 80-90\%$. At the same time the plasticity characteristics of the cyclically loaded molybdenum samples did not change substantially. This is seen from the values of the bending deflection given in Table 1. The temperature of cold brittleness meanwhile decreases only by 15-20°C compared to the initial state, and with the appearance of microcracks it even increases.

In the initial recrystallized state we observe a rather high density of carbide precipitates in the structure of polycrystalline molybdenum (see Fig. 2a). These are also observed after cyclic loading of the samples (see Figs. 2b and 3a). During the intermediate annealing up to 800°C the carbide phase is entirely dissolved (Figs. 4a, b). Thus, if the cyclic loading is accompanied by the formation of mosaic structure, and the annealing temperature is sufficiently high, the carbide phase is dissolved. The surplus carbon evidently segregates at the subgrain boundaries.

It is well known [5] that upon deformation of dilute molybdenum alloys one also observes the disappearance of the segregated surplus carbides when the mosaic structure is formed. Conversely, when the annealing temperature is raised, the segregation of carbides is observed from the moment of the disappearance of the mosaic structure.

In Fig. 5a, b typical fracture surfaces are shown for samples which were tested at room temperature, i.e., substantially below the temperature of cold brittleness in the initial state as well as after cyclic loading under conditions similar to those for sample 12 (see Table 1). In both cases the bending deflection before fracture is small (1.2-1.4 mm). While in the initial state the fracture was primarily intergranular (see Fig. 5a) after cyclic loading it took place across the bulk of the grain (see Fig. 5b).

The effect of elastic vibration on the dislocation structure was also studied on chromium and tungsten samples, which had undergone recrystallizing anneals at 1200°C and 1800°C, respectively. Thereafter

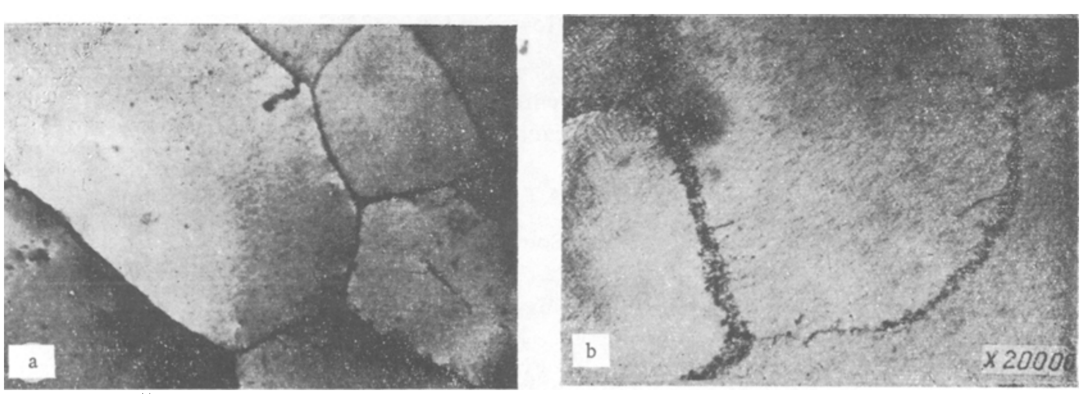

Fig. 6. Mosaic structure arising in chromium (a) and tungsten (b) sample upon uninterrupted cyclic loading for 15 sec.

they were cyclically loaded at a frequency of 50 cps in the console-bending configuration until the appearance of the first microcracks on the polished surface. The temperature of cyclic loading was 400°C for chromium and 600°C for tungsten.

As shown by the electron-microscopic studies (Figs. 6a, b), in this case too the deformation of the mosaic structure is observed. The microhardness of chromium samples increases from 130 to 160 kg/mm^2. The obtained data support the claim that mosaic formation is possible in principle in chromium and tungsten samples upon cyclic loading.

Thus, alternating deformation at 50 cps at high temperatures leads to the formation of mosaic structure in molybdenum, chromium, and tungsten. The misorientation of neighboring subgrains in samples tested up to the fatigue limit is comparable to that in deformed metals. The formation of strongly misoreinted mosaic structure leads to hardening which is comparable to what can be achieved by large plastic deformations.

However, no substantial improvement of the low-temperature plasticity was observed upon cyclic loading by low-frequency vibrations in the range tested in the present work. The temperature of cold brittleness is lowered by 15-20°C, and an insignificant increase of the bending deflection at room temperature is observed.

There may be several reasons for these phenomena. First of all, the formation of microcracks, which perhaps are not entirely removed by electropolishing and annealing, and worsen the plastic properties. It has been established for iron in [6] and in other works [7-9] that the mosaic structure arises for large amplitudes and long exposures, close to fatigue fracture. At the same time it was found that fatigue damage accumulates in the structure after a period amounting to 1-2% of the sample's lifetime for the given stress amplitude. Secondly, the most substantial structural changes in alternating bending are observed in a 0.1-0.2 mm thick surface layer, while the structure of the bulk of the sample remains practically unchanged. Thirdly, during plastic deformation there is a significant change in the shape of the initial grain [5]. For our method of cyclic loading and at stresses of about 70 kg/mm^2 the size and shape of the initial metallographic grain does not change significantly.

CONCLUSIONS

1. We have established that under the effect of elastic vibrations in molybdenum a mosaic structure arises with large angles of misorientation (3-5°). The mean subgrain size is about 2 μ.

2. We determined that the formation of misoriented mosaic structure in molybdenum, chromium, and tungsten takes place upon cyclic loading with amplitudes and exposure times at which cracks due to fatigue appear.

3. It was shown that the application of intermediate anneals up to 800°C and electrolytic polishing in order to remove the surface layer can substantially decrease the number of microcracks observed in the surface layer.

4. It was shown that the hardening achieved in molybdenum by low-frequency cyclic loading is comparable to the hardening that can be achieved by 90% plastic deformation in rolling. The yield stress of cyclically loaded samples amounts to 110-120 kg/mm^2 as compared to 50-60 kg/mm^2 in the initial state. The transition temperature to cold brittleness is lowered by 20-30°C.

5. We have established that cyclic loading of molybdenum samples leads to a change of the character of brittle fracture. While in the initial, recrystallized state the fracture is primarily intergranular, after cyclic loading we observed transgranular fracture.

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