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Recrystallization Diagram of Molybdenum

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As is known, the mechanical properties of metals, along with other factors, are affected by grain size. Molybdenum belongs to the metals in which this is manifested especially strongly; brittle coarse-grained molybdenum ingots, with properly selected mechanical and thermal treatment to obtain a fine-grained uniform structure, become much more ductile and can be subjected to cold deformation.

Consequently, the construction of a recrystallization diagram for molybdenum, relating the grain size of the metal to the degree of its deformation and the annealing temperature, is of particularly great interest.

The literature contains data on the dependence of the recrystallization temperature of molybdenum of various grades on mechanical and thermal treatment ([1]-[4]), but no systematic study of the recrystallization diagram has been carried out.

We have constructed a recrystallization diagram of first-grade molybdenum, cast in an arc furnace in vacuum with carbon deoxidation. Chemical and gas analyses showed that the molybdenum contained 0.2–0.1% carbon, hundredths of a percent of iron, and traces of silicon. The oxygen content was 0.002%.

To obtain a uniform fine initial structure, the molybdenum ingots were forged in several passes. The forging-start temperature was 1600°, and the finishing temperature 1200°. The specimens were heated before forging in a hydrogen furnace. The total degree of deformation was 96%. As a result of such treatment at high temperatures, the very coarse, nonuniform cast structure was destroyed; forging at a lower temperature (below the recrystallization temperature of molybdenum) led to the formation of texture. After annealing the deformed specimens at 1300° in vacuum for 1 hour (the annealing regime was selected as a result of experiments), the specimens had a polyhedral fine-grained structure with an average grain diameter of about 22–25 μ.

For construction of the recrystallization diagram, the initial specimens obtained by the method described above were subjected to cold deformation with degrees of reduction of 2.5; 5; 7.5; 10; 20; 30; 40; 50; 60; 70% by compression on a 35-ton hydraulic press. To obtain the specified dimensions, compression was carried out in limiting rings whose height corresponded to the required final dimensions.

Fig. 1

Figure 1: Fig. 1

Fig. 2

Figure 2: Fig. 2

No destruction of the specimens was observed in this process, and only when the degree of deformation was increased above 20% did small cracks appear along the edges, whereas attempts to deform cast molybdenum led to severe cracking even at small degrees of deformation. Investigation of the microstructure of the initial specimens after cold deformation showed that, at small degrees of deformation (2.5; 5; 7.5%), the appearance of oriented grain blocks was detected; at higher degrees, grain refinement was observed, and compression above 50% led to the formation of elongated parallel fibers in the direction of compression (Fig. 1 a, b, c).

Fig. 1. Microstructures of the initial molybdenum after cold deformation and annealing. 100×
a –7.5% compression; *b* –30% compression; *c* –70% compression; *d* –the same as *a* after annealing at 1530°; *e* –2.5% compression and annealing at 1730°; *f* –the same as *c*, after annealing at 1730°.

Fig. 2. X-ray diffraction patterns of molybdenum: *a* –initial; *b* –deformed by 70%; *c* –deformed by 70% and annealed at 935°.

The deformed specimens were annealed for 1 hour in vacuum (10^{-3}) at 1100, 1200, 1300, 1400, 1530, and 1730°. The annealed specimens were subjected to microstructural analysis, and the microhardness and hardness of the deformed and annealed specimens were also measured.

Determination of the temperature at which recrystallization begins was carried out by the X-ray method; to refine it, additional anneals were performed at temperatures of 935, 950, 960, 970, 980, 985, 1000, 1010, 1025, 1030, 1040, 1050, 1070, and 1180°. To reveal the microstructure, a solution of nitric and sulfuric acids in water was used as the etchant (HNO_3 5 parts, H_2SO_4 3 parts, H_2O 3 parts). To evaluate the dependence of grain size on the degree of deformation and annealing temperature, its mean diameter was determined under the microscope by the intercept method and by the method of calculating the grain area on photomicrographs.

X-ray photographs were taken in molybdenum radiation with an exposure of 1.5–2 hours. On the basis of the investigations carried out, it was established that recrystallization begins at 935° in molybdenum specimens cold-deformed by 70% (Fig. 2) and at 1025° for 50% deformation. At smaller degrees of deformation (10%), recrystallization begins only at 1200°.

Above 1300°, recrystallization is observed in all deformed and annealed specimens. Grain growth at the critical degree of deformation occurs from 1400° (degree of deformation 10%). An increase in temperature leads to still more intensive grain growth; moreover, the maximum grain size is observed at 1530° with a degree of deformation of 7.5%, and at 1730° with 2.5%, whereas at large degrees of deformation and the same annealing temperature (1530°, 70%) the grain size is considerably smaller (Fig. 1, *c*, *d*, *e*). Hardness was measured under a load of 100 kg with a pobedit cone; microhardness was measured with a diamond pyramid under a load of 50 g. It is interesting to note that the above-mentioned formation of a block structure at small degrees of compression is always accompanied by some decrease in the hardness of the specimens. Then, with increasing degree of deformation, the hardness and microhardness of the specimens increase. A particularly strong increase in hardness is observed beginning with 10% deformation. Subsequent annealing considerably lowers the hardness of the specimens. Thus, in the specimen deformed by 70%, the hardness was about 230 kg/mm^2, and after annealing at 1100° it decreased to 187 kg/mm^2 as a result of the recrystallization process. Raising the annealing temperature to 1730° lowers the hardness of such a specimen still further (to 157 kg/mm^2), which is caused by the growth of the recrystallized grain (Table 1). The presence of "critical" degrees of deformation causes some decrease in hardness, which is especially noticeable at high annealing temperatures. The change in microhardness as a function of

Table 1

Hardness of deformed and annealed molybdenum specimens (kg/mm^2)

Annealing temperature, °C	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %
Annealing temperature, °C	0	2.5	5	7.5	10	20	30	40	50	60	70
Deformed, not annealed	171	166	170	172	178	182	189	192	201	205	229
1100	168	162	163	164	173	173	173	173	178	178	187
1200	166	163	166	170	175	175	175	171	177	177	183
1300	165	163	164	169	169	171	172	172	172	174	176
1400	163	160	163	161	168	167	167	167	167	168	165

Fig. 3. Recrystallization diagram of first-kind molybdenum

Figure 3: Fig. 3. Recrystallization diagram of first-kind molybdenum

Annealing temperature, °C	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %	Deformation, %
1530	162	158	162	156	164	160	160	156	158	158	160
1730	154	142	152	156	157	156	156	152	155	157	157

degree of deformation and annealing temperature, similarly to the change in the overall hardness of the specimen, with the only difference that in this case higher hardness values are observed.

On the basis of the investigations carried out, we constructed a recrystallization diagram for molybdenum (Fig. 3). The temperatures of the onset of recrystallization as a function of the degree of deformation are plotted on the diagram with a dashed line. From the data of the diagram it is evident that, when cold-deformed molybdenum specimens are annealed up to 1300°, no grain growth is observed, and the grain sizes at all degrees of deformation are comparable with the grain sizes of the initial specimens; the average grain diameter in the initial specimens, annealed up to 1300°, is about 26–28 μ, while in deformed specimens annealed at the same temperature it does not exceed 30–32 μ, and at high degrees of deformation the average grain size decreases to 20 μ.

Fig. 3. Recrystallization diagram of first-kind molybdenum

Annealing at 1400° leads to noticeable grain growth at a deformation degree of 10%.

Above 1400°, coarsening of the grains is observed at all degrees of deformation; the “critical”degree of deformation shifts to 7.5%, and the average grain diameter increases at 1530° to 223 μ. The grain grows much less at high degrees of deformation (beginning with 40%), and at 70% its size is only 40 μ.

Annealing at 1730° leads to still greater grain growth. The “critical” degree of deformation at this annealing temperature shifts to 2.5%, and the grain size increases to 300 μ. The average grain diameter in specimens deformed by more than 20% is 80–90 μ and does not change with increasing degree of deformation.

On the basis of the totality of the data obtained in the investigation of molybdenum recrystallization, it may be concluded that hot-rolled molybdenum after cold working at deformation degrees above 20% may be annealed up to 1400°. Under these conditions the grain remains fine.

Higher annealing temperatures lead to the production of a coarse-grained structure after annealing.

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