# IMPROVEMENT OF THE HEAT-RESISTANT PROPERTIES OF MOLYBDENUM AFTER TREATMENT FOR POLYGONIZATION

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UDC 669.28:621.785.34.37

The most promising methods of strengthening metals and alloys not having polymorphous transformations but in which the solubility of secondary components changes sharply are evidently those which provide for stable alterations of the fine structure, leading to an increase in the extent of the mosaic structure and misorientation of subgrains and to the inhibition of elementary acts of plastic flow. Therefore it is expedient to use a combination of cold working and subsequent heating, which leads to the development of polygonization processes and "recrystallization in situ."

In [1] we determined the treatment conditions for molybdenum (0.008% $SiO_2$; 0.012% $Fe_2O_3$; 0.003% $Al_2O_3$; 0.002% CaO; 0.001% MgO, Ni, Mn; 0.01% Zn; 0.014% C; 0.0032% $O_2$; 0.25% $N_2$) which ensure polygonization. It was shown that deformation of molybdenum with 5, 9, and 13% reduction and subsequent annealing leads to a polygonal structure. With deformation at an elevated temperature (1150°C) polygonization begins during the deformation process itself and is completed during subsequent annealing. In the present work we made tests of the long-term strength and creep resistance in a vacuum apparatus under a pressure of $10^{-3}$ mm Hg. Samples 1 mm in diameter and 160 mm long were fastened in clamps made of a heat-resistant alloy and were heated at the rate of 100-150°/sec by passing an electric current through them. The elongation of the samples was measured in a base length of 15 ± 0.2 mm with the KM-6 cathetrometer through a window in the vacuum chamber. The time before fracture was measured with a stopwatch from the moment the sample reached the specified temperature.

Comparison of the long-term strength curves at 900°C for molybdenum previously treated under different conditions showed that polygonization increases the time before fracture (Fig. 1). However, the curve for the sample in which one observes a well developed substructure (Fig. 2) lies at an angle to the curve for the cold-worked sample, which indicates that in this case strengthening does not persist and in time disappears.

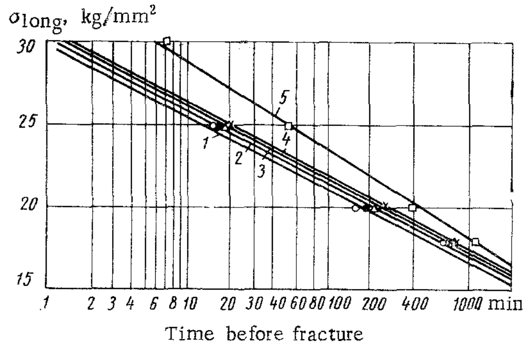

Fig. 1

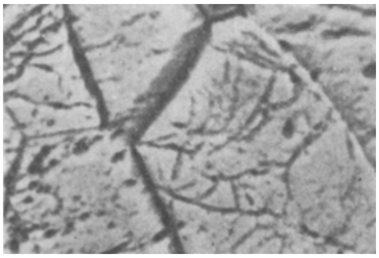

Fig. 2

Fig. 1. Long-term strength at 900°C of molybdenum samples deformed 9%. 1) Deformed at 300°C; 2) deformed at 1150°C; 3) deformed at 300°C, annealed 1 h; 4) deformed at 1150°C, annealed 1 h; 5) deformed at 1150°C, annealed 100 h.

Fig. 2. Microstructure of molybdenum deformed 9% at 1150°C and annealed 100 h at 1150°C. ×1000.

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Moscow Institute of Steel and Alloys. Translated from Metallovedenie i Termicheskaya Obrabotka Metallov, No. 8, pp. 60-63, August, 1967.

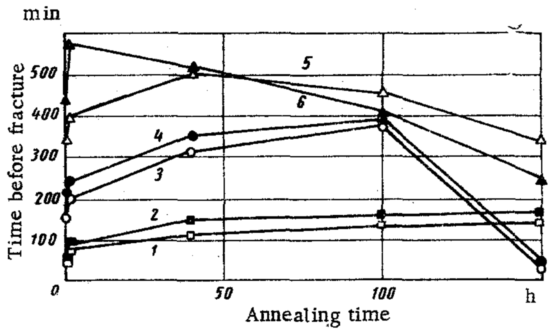

Fig. 3

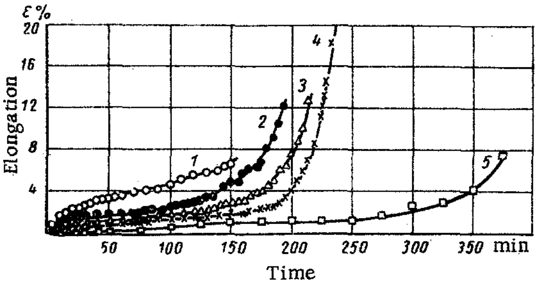

Fig. 4

Fig. 3. Long-term strength of molybdenum at 900°C under a stress of 20 kg/mm$^2$ as a function of previous treatment. 1) Deformed 5% at 300°C; 2) deformed 5% at 1150°C; 3) deformed 9% at 300°C; 4) deformed 9% at 1150°C; 5) deformed 13% at 300°C; 6) deformed 13% at 1150°C.

Fig. 4. Creep of molybdenum at 900°C under stress of 20 kg/mm$^2$ after preliminary reduction of 9%. 1) Deformed at 300°C; 2) deformed at 300°C, annealed 1 h; 3) deformed at 1150°C; 4) deformed at 1150°C, annealed 1 h; 5) deformed at 300°C, annealed 100 h.

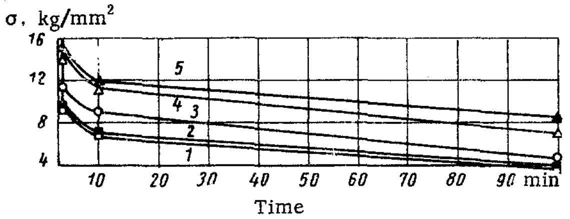

Fig. 5. Stress relaxation at 900°C of molybdenum subjected to various treatments. 1) Deformed 5% at 300°C, annealed 150 h; 2) deformed 5% at 1150°C, annealed 150 h; 3) deformed 9% at 1150°C, annealed 40 h; 4) deformed 13% at 300°C, annealed 40 h; 5) deformed 13% at 1150°C, annealed 40 h.

It can be seen in Fig. 3 that under all annealing conditions the time before fracture of molybdenum deformed 5 and 9% at 1150°C is somewhat higher than for the samples deformed at 300°C. Evidently this is due to the fact that deformation at a high temperature and subsequent annealing promote more complete polygonization in most of the metal grains.

After 5% deformation, annealing 1 h and 40 h at 1150°C increases the time before fracture slightly, while further increase of the annealing time to 100 and 150 h has almost no effect. After 9% deformation the longest time before fracture was noted in the samples annealed 100 h. After 13% deformation at 1150°C, annealing 1 h substantially increases the time before fracture; the time before fracture decreases with further increase of the annealing time.

The results show that preliminary polygonization of molybdenum notably increases the long-term strength. The influence of the substructure on the heat-resisting properties is due to the fact that "sharp" dislocation boundaries constitute effective barriers to moving dislocations. In addition, during annealing after deformation the dislocations interact with impurity atoms, which form elastic Cottrell "atmospheres," pinning the dislocations. The pinning effect of the atmospheres is also an important factor in increasing the resistance to prolonged heating at elevated temperatures.

In tests of molybdenum at 1000°C the time before fracture usually begins to decrease at shorter annealing times than in tests at 900°C. This is due to the acceleration of recrystallization processes under these conditions.

Figure 4 shows the creep curves for molybdenum deformed 9% under a stress of 20 kg/mm$^2$ at 900°C. Under these conditions it was impossible to determine the influence of previous polygonization in the nonsteady creep stage, where the basic variations of the form of the creep curve have been linked with preliminary treatment [2-4], because this stage is completed in a very short time.

The substructure produced before the creep tests has a strong influence on the steady creep rate. This stage is characterized by the equilibrium of two processes: increased resistance to plastic flow, induced by work hardening, and weakened resistance to plastic flow as the result of recovery. The substantial increase in the time of steady creep for molybdenum samples with a well developed polygonal structure in comparison with samples not polygonized indicates that weakening processes are inhibited in the presence of stable dislocation arrays (polygons).

It is interesting to note that molybdenum samples deformed at 1150°C, and also samples deformed at 1150°C and then annealed 1 h at 1150°C, have increased plasticity (by comparison with samples cold worked as well as cold worked and annealed 1 h). However, increasing the annealing time after deformation, i.e., conducting the optimum treatment for development of the polygonal structure, reduces the elongation of the samples in long-term strength and creep tests. While the observed substantial increase of the steady creep stage indicates the favorable influence of the changes in the substructure caused by polygonization, the decreased plasticity of the samples subjected to prolonged annealing indicates the influence of strain aging.

Stress relaxation tests of molybdenum subjected to various treatments were conducted at 900 and 1000°C and an initial stress of 24 kg/mm$^2$ on samples 1 mm in diameter and 60 mm long. As can be seen in Fig. 5, the highest stress relaxation was observed in samples reduced 5% at 300 and 1150°C. The greatest resistance to relaxation was observed in samples deformed 13% at 1150°C and then annealed 40 h.

From comparison of the relaxation data with the results of the creep tests it is evident that in all cases where plastic deformation is inhibited during creep tests at constant stress its sharp reduction under relaxation conditions is also inhibited.

## LITERATURE CITED

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