TIME DEPENDENCE OF MICROPLASTIC DEFORMATION OF MOLYBDENUM AT LOW TEMPERATURES

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The temperature dependence of the deformation processes in the stress region between the macroscopic flow stress and the elastic limit was investigated for pure recrystallized Mo in the temperature range between 298 and 443 K. Two short-time creep-like tests (temperature-step tests and stress-step tests) were employed. These tests have been found suited to differentiate between characteristic plastic deformation regions. An intrinsic flow stress limit at small strain rates could be identified, related to characteristic dislocation mechanisms.

INTRODUCTION

It is well recognized that the flow stress of bcc metals is drastically dependent on strain rate, temperature and impurity content, in particular below a characteristic transition temperature considered to correspond to approximately 0.2 T_m (T_m: melting point in K) [1,2]. This dependence has been attributed to the characteristic behavior of screw dislocations [3,4]. Only few investigations on the deformation behavior of bcc-metals at stresses below the flow stress have been published [5]. This stress region has been termed the region of "microplasticity". Detailed studies were published on the microplastic behavior of polycrystalline Fe in the temperature range of 5-290 K [5,6]. Some information is available on the temperature dependence of the microplasticity of high melting refractory metals, such as Nb, Ta, W and Mo [7-11].

From these investigations stress-temperature diagrams may be deduced which show characteristic regions of microplastic behavior. Lines separating areas of elastic strain, anelastic strain and macroscopic flow behavior can be identified [12]. In contrast to the strong temperature and strain-rate dependence of the macroscopic flow stress the microplastic behavior is characterized by a weak temperature and strain-rate dependence. The elastic stress limit was related to the bowing-out of pinned edge dislocations. The anelastic stress limit was defined by some authors as that stress at which edge dislocations become unpinned. The anelastic strain was attributed to dislocation interaction or multiplication. Other investigators related the anelastic strain to the cross slip of screw dislocations [5,6]. In the stress interval between the elastic and anelastic stress limits it was assumed that the dislocations always returned to their initial position after unloading while above the latter the dislocation may not completely regain their prior position [5,6]. To obtain detailed information of the

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deformation processes in the stress region between the flow stress and elastic limit short-time creep-like tests at temperatures between 298 K and 443 K were performed on specimens of pure recrystallized Mo.

EXPERIMENTAL INFORMATION

Specimens were prepared from powder metallurgical pure Mo cold-rolled sheet metal of 2 mm in thickness, with a parallel gauge section of 20 mm in length and 6 mm in width. After machining the specimens were recrystallized by annealing at 1200°C for 1h in hydrogen, resulting in a grain size of ASTM 8-9 [13]. The main impurities consisted of 190 ppm W, 30 ppm Fe, 20 ppm O_2 and 15 ppm C. The creep-like experiments were performed with a commercial tensile test equipment. The test temperature was varied between 298 K and 443 K by using a circulating-air heating chamber with a temperature control of better than ±0.5 K. The strain was measured with strain gauges attached by a high temperature adhesive to both sides of the specimens in the center of the gauge length.

Two types of creep-like tests were performed. The experimental procedure for one type (temperature-step test) consisted of constant stress tests for 10 minutes at a selected temperature in the range between 298 K and 443 K. For changing the temperature the specimen was unloaded to a low prestress level (~17 MPa) until the desired new test temperature was reached and then reloaded to the former stress value. The second test procedure (stress-step test) consisted of a stepped loading procedure, starting at 50 MPa up to approximately 300 MPa, for step increments of 25 MPa and holding time of 30 minutes. For each test sequence the temperature was kept constant to within ±0.5 K.

RESULTS AND DISCUSSION

Results of a temperature-step test for a stress level of 130 MPa is shown in Fig.1, similar tests were carried out at stresses of 150 and 200 MPa. The starting points of all test sequences in the figure were set to zero neglecting the cumulative strain of the previous steps. This appears justified in view of the small amount of pre-strain involved ($\varepsilon_{pl} < 10^{-3}$). The creep curves show characteristic changes depending on test temperature. Below a critical temperature the slope of the curves decreases with test time, while above this temperature an acceleration in creep rate can be noticed. The observed critical temperatures are closely related to the applied stress.

The results of a stress-step test are illustrated in Fig.2, with the starting strain set to zero for each curve. Clearly, in this type of tests a critical stress characterizing the change in the creep rate can be identified for the given test temperature. From these measurements the creep rates were calculated as a function of the creep strain following a procedure proposed by Stone and Conrad [7]. As an example, the results for a test temperature of 298 K are plotted in Fig.3. In this plot the small cumulative strain from the previous loading sequences is also neglected. From this presentation the critical stress at which the strain-dependence of the creep rate exhibits a slope change (from negative to positive) can easily be deduced.

A summary of the critical stress-temperature-time condition obtained from both types of creep-like tests is presented in Fig.4. Typical border lines can be identified. The lowest curve indicates the elastic limit, which appears weakly temperature dependent [12]. The top curve represents the macroscopic flow stress, defined by an offset strain of approximately 10^{-3} (i.e., the upper yield stress), determined by tensile tests at a total strain rate of $4x10^{-5}$/sec, corresponding to a loading rate of 14 MPa/sec. In-between these two lines the results deduced from the creep-like experiments (temperature-step tests with holding time of 10 min, stress-step tests with holding time of 30 min) are plotted, termed in the following as the intrinsic flow stress. This stress-step test was carried out with an equivalent loading rate of 0.014 MPa/sec, which corresponds to an average strain rate of approximately $4x10^{-8}$/sec, 1000 times slower than that applied in the tensile tests. For the temperature-step test an equivalent heating rate of 0.033 K/sec was selected. It can be seen that these flow stress lines are strongly dependent on the strain rate or holding time. A further support was obtained from long-time creep tests for which critical combinations of temperature and stress have been found to lead to comparable creep results [12]. Preliminary experiments have shown that the intrinsic flow stress may be affected by small amounts of pre-deformation.

The microplastic region is situated below the intrinsic flow stress line and above the elastic limit, as indicated in Fig.4. The microplastic behavior is characterized by a decelerating creep rate (Fig.1 and Fig.2). It may be speculated that the microplasticity is associated with the mobility of edge dislocations [14]. Above the intrinsic flow stress lines the plasticity may be related to an increasing contribution of mobile screw dislocations, since the screw dislocations have been assumed to govern the flow behavior [2,3]. This point will need support by detailed TEM investigations.

The intrinsic flow stress line observed from the present creep-like tests would represent a lower limit at a strain rate approaching zero, say, of the order of 10^{-8}/sec. The creep-like tests and slow constant strain rate tests should thus yield the same results. It is expected that the exact position of an actual intrinsic flow stress line should correspond to a long holding time in the creep-like tests.

CONCLUSIONS

1. Creep-like tests have been found suited to differentiate between characteristic regions of the micro- and macroplastic deformation behavior of recrystallized molybdenum.

2. An intrinsic flow stress is identified in-between the lines of elastic limit and the macroscopic flow stress. This border line is deduced from the change in the shape of the creep curves and can be explained by the temperature and strain rate dependence of the mobility of edge and screw dislocations.

3. The proposed intrinsic flow stress represents a lower limit of the flow stress determined in the tensile tests at very small strain rates.

ACKNOWLEDGMENTS

The authors thank the Plansee Aktiengesellschaft for active support and supply of the specimen material, and Dr. W.Pichl, Institute for Solid State Physics, University of

Vienna, for his interests and helpful discussion. The investigations were financed in part by the Austrian National Science Foundation (Project P10160-PHY). The work was carried out as part of a collaboration with the National Natural Science Foundation of PRC (Project A13).

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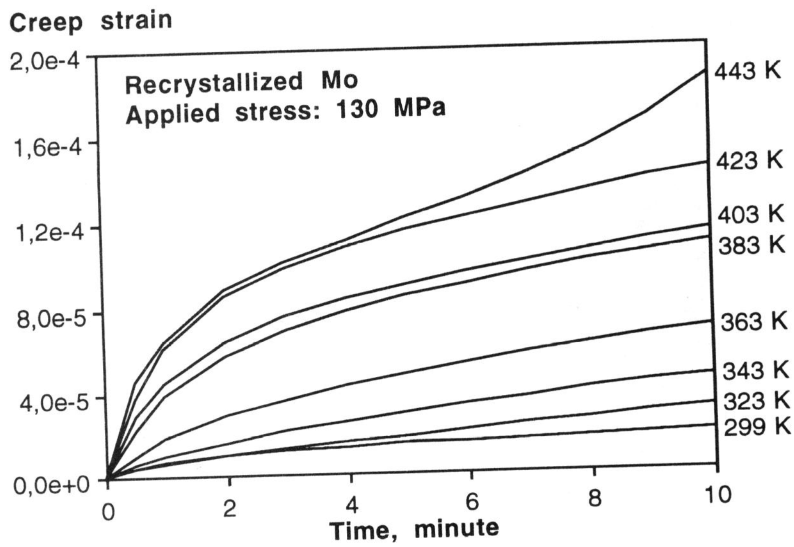

Fig.1 Results of a temperature-step test of recrystallized Mo at a stress of 130 MPa.

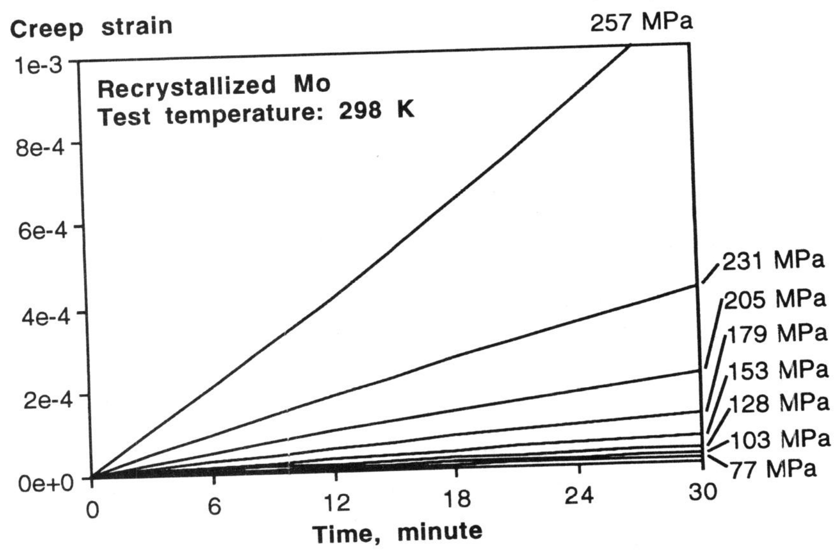

Fig.2 Results of a stress-step test of recrystallized Mo at 298 K.

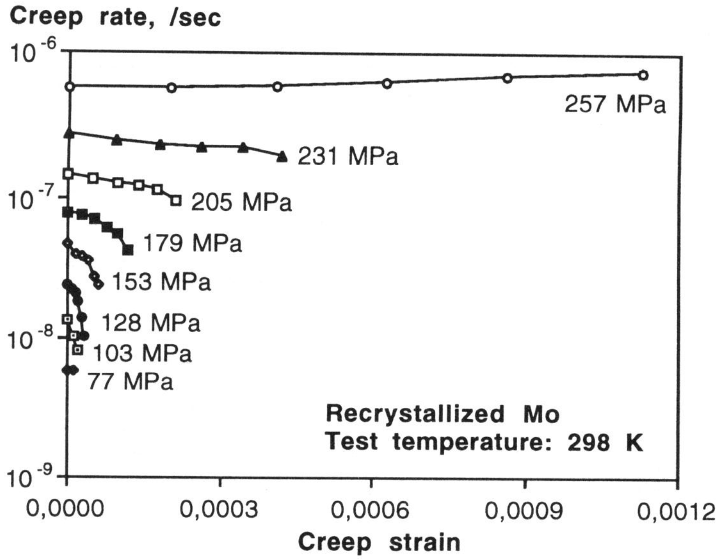

Fig.3 Change of the creep rate with the applied stress level as a function of the creep strain at 298 K.

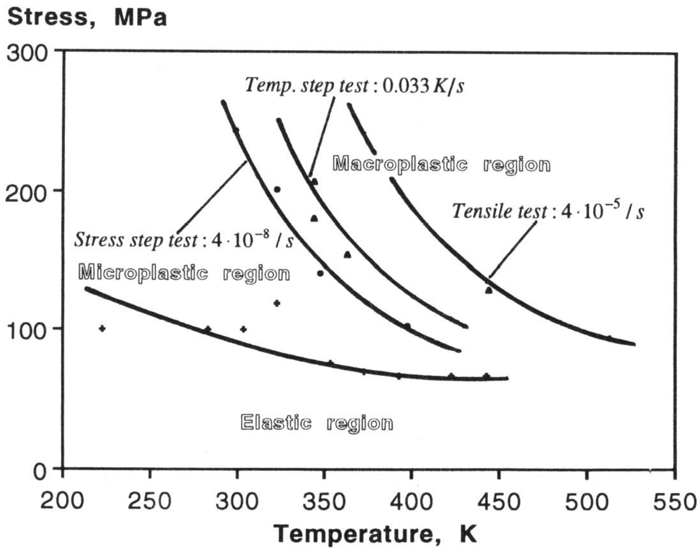

Fig.4 Summary of the test results in a stress-temperature diagram, indicating regions of microplasticity and macroscopic flow stress.