Int. Journal of Refractory Metals and Hard Materials 41 (2013) 35–40

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

In-situ SEM study of temperature dependent tensile behavior of wrought molybdenum

F. Fang [a],*, Y.Y. Zhou [a], W. Yang [b],[a]

[a] *School of Aerospace, Tsinghua University, Beijing 100084, China*
[b] *The University Office, Zhejiang University, Hangzhou 310058, China*

ARTICLE INFO

Article history:
Received 12 September 2012
Accepted 24 January 2013

Keywords:
Molybdenum
Refractory alloys
Uniaxial tension
Fracture
Elevate temperature

ABSTRACT

Molybdenum and its alloys are widely used in electronic industries, metallurgies, aerospace, and nuclear engineering, etc. The applications frequently involve environmental conditions where both the loading and the temperature change. Uniaxial tension tests are carried out for wrought molybdenum under a scanning electron microscope (SEM) at a temperature range from 300 °C to 750 °C. The results indicate that both the tensile strength and the fracture strain for wrought molybdenum are peaked at 300 °C within the tested temperature range. With further increases of temperature, the fracture strain decreases monotonically. In-situ SEM observations reveal the slip band evolution, the deformation of grains, as well as the crack formation. At elevate temperature, cross-slip occurs at a lower strain, and the entanglement and intersection of slip bands lead to the formation of cell structures. Combining the SEM surface morphologies and the fractographs, one concludes that the fracture type is mainly of cleavage when uni-axially tensioned at 300 °C, albeit extensive plastic deformation occurs prior to fracture. The fracture type becomes mixed cleavage and plastic shear when uni-axially tensioned at 450 °C, 600 °C, and 750 °C.

1. Introduction

Molybdenum is a refractory metal with high melting point (2610 °C), low thermal-expansion coefficient, and high thermal/electrical conductivity. Molybdenum suffers from a poor ductility at room temperature, while exhibits high levels of ductility and toughness at a homologous temperature (T/T_m (Kelvin)) greater than 0.3 [1]. Molybdenum is typically with a body-centered cubic (bcc) lattice structure. In 1958, Cottrell [2] proposed a theory of the deformation and fracture of bcc transition metals. Later on, a series of experiments on polycrystalline molybdenum are carried out which were designed to provide a quantitative test of Cottrell's theory [3]. It has been shown that the room temperature brittleness and the plastic behavior at higher temperatures are closely related to the dislocation pattern of $1/2<111>$ screw dislocations [2–4].

To improve the room-temperature ductility and high-temperature strength, alloying to molybdenum has always been employed. Examples of molybdenum-based alloys include molybdenum–0.5% titanium–0.1% zirconium (TZM), and oxide dispersion-strengthened (ODS) molybdenum. TZM is a micro-alloyed molybdenum with small amounts of titanium, zirconium, and carbon for precipitation strengthening by titanium and zirconium carbides and solid solution hardening [1,5–9]. ODS molybdenum (or Mo–La), is a fine grained molybdenum doped with lanthanum oxide particles [5–8]. Both TZM and ODS molybdenum possess excellent room temperature ductility as well as good high-temperature strength.

Molybdenum and its alloys are widely used in electronic industries, metallurgies, and nuclear engineering, etc. [8–12]. Such applications typically encounter severe hostile environmental conditions in terms of loading and temperature. It is then essential to characterize the temperature dependence of their mechanical behavior. Cockeram [15] systematically studied the temperature dependent tensile behavior and fracture mechanisms of wrought low carbon arc cast unalloyed molybdenum, ODS and TZM molybdenum at a temperature range of −100–1000 °C. It was observed that all the molybdenum alloys exhibited the same trend of tensile strength reduction with the rising of temperature. For both wrought Mo and TZM, the largest fracture strain was observed at room temperature while it was at 100 °C for ODS. The tensile strength and fracture strain are 653 MPa and 9% respectively for wrought unalloyed Mo at room temperature. Due to the finer grain size and second phase particles presented, the tensile strength and fracture strain for TZM and ODS are slightly higher than those observed for wrought unalloyed Mo [15]. Optical microscope observations indicated that the alloys exhibited a ductile laminate failure mode. Fracture initiated at grain boundaries, left ligaments of sheet-like grains, which were further pulled to failure with various degrees of necking in each grain [15].

Although there are numerous studies on the mechanical behavior and microstructure of Mo and its alloys [1–22], in-situ investigations on the microstructure, such as the slip band evolution under mechanical loadings are rare. In the present work, pure wrought molybdenum is used to preclude the effects of second phase on the mechanical behavior. Uniaxial tension tests on wrought molybdenum samples were conducted in-situ with SEM observations at a temperature range of 300–750 °C. The study aims to reveal the uniaxial tension behavior

---

* Corresponding author. Tel.: +86 10 62773331.
E-mail address: fangf@mail.tsinghua.edu.cn (F. Fang).

0263-4368/$ – see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.ijrmhm.2013.01.014

and the microstructure evolution for wrought molybdenum at different temperatures. The results would be of importance for better understanding of the structure–property relationship, as well as for practical application of the wrought molybdenum.

2. Experimental procedures

Plates of the wrought molybdenum are provided by General Research Institute of Iron and Steel, Beijing, China. In order to adapt to the vacuum chamber of Shimadzu SS550 Scanning Electron Microscope, flat and dog-bone shaped specimens are used, as shown in Fig. 1. In order to reveal the prevailing microstructure of the specimens under SEM, the specimens were first polished with diamond paste (down to 0.25 μm) until a near-mirror finish was obtained. They were later etched in an aqueous solution of potassium ferricyanide and sodium hydroxide for about 50 s at room temperature.

Shimadzu SEM Servopulser (High Temperature Fatigue Testing Machine with a SEM Microscope) was used to perform the uniaxial tests. The machine provides a maximum load of 1 kN and a displacement range of 25 mm at a temperature range up to 800 °C. The SEM was operated at an accelerating voltage of 15 kV. The tensile tests are conducted under displacement control mode. Since the stress–strain behavior depends on the strain rate at high temperatures, all the tests are conducted at a strain rate of 3×10^{-5} s^{-1}. 2–4 specimens were averaged to collect the tensile property of the samples. During the tests, the tensile loads (F_N) and displacement (ΔL) were recorded by the Shimadzu testing machine. The corresponding stress is obtained by F_N divided by the narrowest cross section area ($(0.8\text{–}1.0)\times2.5$ mm^2). The strains are obtained by recording the low-magnification SEM morphologies where there are two marks across the center of the sample. These pictures were later processed using software specifically written to obtain the strains as a function of the tensile stress.

3. Experimental results

The representative engineering stress–strain curves are shown in Fig. 2 for the wrought molybdenum under uniaxial testing at 300–750 °C. The stress–strain curve measured at 25 °C is also shown for comparison. Table 1 shows the tensile data of Young's modulus (Y), the proportional limit (σ_p), the yield strength (σ_y), the ultimate tensile strength (σ_{UTS}) or the maximum tensile stress (σ_{max}), and the fracture strain (ε_f) for the wrought molybdenum at different temperatures. The fracture strain ε_f and tensile strength show the same trend of reduction as the temperature rises from 300 °C to 750 °C. After yielding, all the curves show very low strain hardening exponent, indicating the low resistance to further plastic deformation.

In-situ SEM observations reveal that the surface morphology changes upon tensile loadings at different temperatures. The low magnification SEM micrographs show the overall morphology change during uniaxial tension. Fig. 3 shows low magnification SEM morphology change for wrought molybdenum under uniaxial tension at 300 °C. As the applied stress increases, the sample initially undergoes elastic deformation until the flow stress is reached. Then, elongation and rotation of grains are observed after yielding, and the original smooth and flat surface become undulated. Cracks are found at grain boundaries though they do not cause the final fracture. Necking occurs prior to fracture (Fig. 3(c)), and the fracture is of a trans-granular mode (Fig. 3(d)). The fracture surface is almost perpendicular to the external tensile load for testing at 300 °C. Similar morphology evolution takes place when the uniaxial testing is performed at 450 °C, 600 °C, or 750 °C. With the temperature increases, the plastic deformation is restricted to a narrower region, and the reduction in area (or in width) decreases. Fig. 4 shows the SEM morphology changes during uniaxial tension at 750 °C. The fracture is also of a trans-granular mode. However, the extent of necking is reduced, and the fracture surface is noticeably inclined to the applied tensile loading direction (Fig. 4(d)), which is similar to those tested at 450 °C and 600 °C.

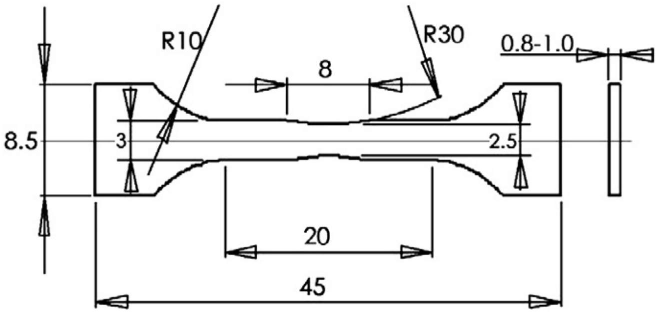

Fig. 1. Schematics of the flat and dog-bone shaped specimen (the sizes are in “mm”).

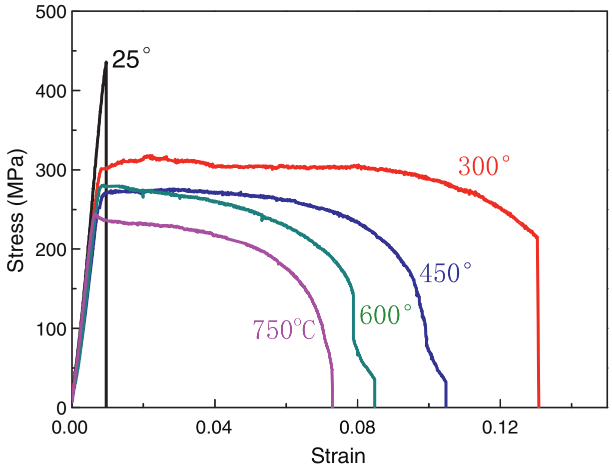

Fig. 2. Stress–strain curves for the wrought molybdenum under uniaxial tension at 300–750 °C. The stress–strain curve measured at 25 °C is also shown for comparison.

SEM micrographs with higher magnification reveal the slip-band structure evolution and crack formation for the wrought molybdenum at different temperatures. Fig. 5 shows the higher magnification SEM micrographs for wrought molybdenum under uniaxial tension at 300 °C. It can be seen that after yielding, at a tensile strain of 1.95%, parallel slip-bands, mostly in the 45° direction with respect to the tensile direction initiated and propagated (Fig. 5(b)). As the strain increases, slip-bands occur in other grains and the bands increase in width and depth so that they become more evidently. Fig. 5(c) and (d) shows the SEM morphologies at tensile strains of 5.76% and 8.13%. Grain elongation and rotation can be readily observed, by comparing grains “A” and “B” at different tensile strains. Inter-granular cracks are found and the crack opening increases with the strain.

As the uniaxial testing temperature increases, slip-bands occur at a lower stress. Those slip bands tend to be curved at a lower strain, indicating an early appearance of cross-slip. The density of slip bands increase rapidly, and the slip bands tangle and intersect with each other. Fig. 6 shows higher magnification SEM micrographs for the

Table 1
Tensile data for wrought molybdenum at different temperatures (Y, $\sigma_{p,}$ σ_y).

Temperature (°C)	Y (GPa)	σ_p (MPa)	σ_y (MPa)	$\sigma_{UTS}/\sigma_{max}$ (MPa)	ε_f (%)
25	46.0	428.8		435	0.96
300	38.4	294.8	301.7	317.4	13.0
450	33.3	251.9	271	275.8	10.5
600	35.1	278.2	279.1	280.4	8.5
750	41.4	229.2	238.7	243.6	7.3