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The effect of texture and microstructure on the properties of Mo bars

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ABSTRACT

The properties and microstructure of pure Mo bars were investigated during upset-forged deformation. The results show that transverse elongation and microhardness of the draw-forged molybdenum samples are improved by upset deformation. After the reduction increases to 80%, the transverse elongation and microhardness of Mo bars are up to 5% and 235HV. The texture of ⟨110⟩ fiber in draw-forged Mo bars is changed to ⟨100⟩ fiber and ⟨111⟩ fiber during upsetting process. Meanwhile, equiaxed dislocation cells are formed in ⟨100⟩ oriented grains, and microbands are formed in ⟨111⟩ oriented grains. Many sessile dislocations are usually formed in ⟨100⟩ oriented grains and glissile dislocations are usually formed in ⟨111⟩ oriented grains. The fracture mechanism of these different grains is quite different.

1. Introduction

The refractory metal molybdenum bars are widely used in industry for their excellent combined properties, such as high strength at elevated temperature, low thermal expansion coefficient, low sputtering yields and high Young's modulus [1–5]. However, the natural brittleness at room temperature limits its further applications [6–10]. Thus, many researches have been focused on improving ductility of Mo [11–14]. So far, the ductility of Mo bars in the longitudinal direction (LD) has been highly improved [15]. However, extremely poor transverse direction (TD) elongation is always observed, which is nearly zero [16]. The low transverse ductility of Mo bars is a key issue to limit their application in many high tech fields, including sleeves, nuts, flanges and pistons [17,18]. Even worse, this problem has attracted little attentions so far.

The ductility of Mo is influenced by process method and heat-treatment procedure. Normally, the plastic deformation can change both the deformation texture and the structural state of materials, which can determine the final properties of materials [19]. Thus, in order to improve the ductility of Mo bars in transverse direction, a special forge process should be applied. However, plastic deformation of materials is usually non-homogeneous, and deformation bands and shear bands are formed [20]. These bands can play an important role in texture control and properties of materials [21–23]. Thus, it is necessary to study the relationship between the orientation and microstructure. In this paper, the orientation dependence of microstructure of the Mo bars during upsetting treatments was investigated. Meanwhile, the fracture mechanism was carefully studied.

2. Experiments

The Mo bars were fabricated as follows: the $(NH_4)_2MoO_4$ solution firstly received an evaporation and concentration, and then calcined at 723–773 K for 4 h; the calcined product was reduced by H_2 at 673–1173 K to obtain the pure Mo powders. After isostatic pressing at 1573 K and 100 MPa for 2 h, ⌀45 mm Mo compacts were obtained. Then the Mo bars were sintered at 2173 K for 4 h. The bars were upset-forged to 50% and then draw-forged back to ⌀45 mm at 1573–1373 K with the forge reduction of every pass of about 10%, followed by annealing treatment at 1573 K for 5 min. This upsetting-drawing-annealing process was carried out for three times. The products were then drawn forged directly to ⌀18 mm and cut into short bar samples with a length of 50 mm. The samples were finally upset to 40%, 70% and 80% at 1373–1073 K.

The transverse elongation of Mo bars was measured at the room temperature by the self-made stainless bend mold composed of upper die and lower die, the schematic plan is shown in Fig. 1a. The specimens were cut from the different Mo bars with dimension of 15.0 mm (L) × 4.0 mm (W) × 2.0 mm (d). The orientation of the sample is shown in Fig. 1b. Then, a series of die with different radius of curvature, such as 1000.0 mm, 200.0 mm, 100.0 mm, 66.7 mm, 50 mm, 33.3 mm, 20.0 mm, 16.6 mm, 13.3 mm, 10.0 mm, 8.0 mm and 5.0 mm were used to bend the sample until the failure of the sample. Then the elongation of the

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sample can be evaluated by the formulation (1):

$$\delta = \frac{2\pi(R+d/2) - 2\pi R}{2\pi R} = \frac{d}{2R} \quad (1)$$

where δ is the elongation of the sample, d is thickness of the sample, and R is the radius of curvature of the half thickness of samples after it is bent.

The microhardness of the sample was measured at a load of 30 N with a holding time of 15 s on a HVA-10A type Vickers hardness testing machine. Texture measurements were carried out on a Bruker D8 Discover machine. The central part of the bars in TD–TD section was always prepared to investigate the texture. Incomplete pole figures (maximum tilt angle 75°) of the planes {200}, {220}, and {222} have been measured. Defocusing correction was carried out by using a random specimen of pressed and sintered high purity Mo powder. The optical microscopic (OM) observation was carried out on NEOPHOT-21metalloscope. The etchant of optical microscopic investigation is a mixing solution of HNO_3, H_2SO_4 and H_2O with a volume ratio of 5:3:4. Electron backscatter diffraction (EBSD) analysis was observed using FEI-Sirion 200 field emission scanning electron

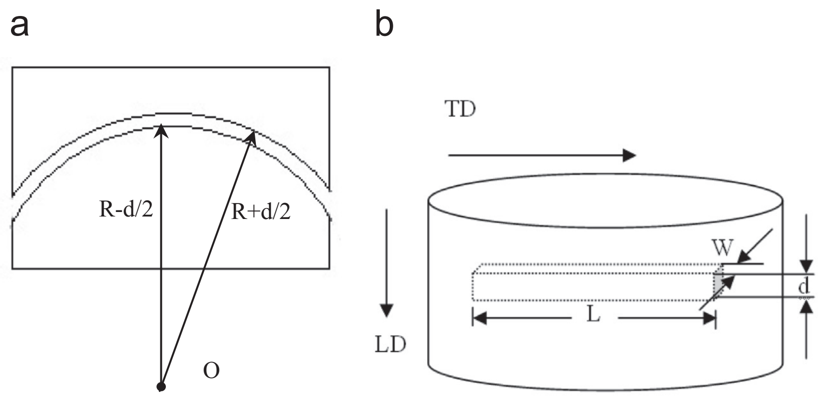

Fig. 1. Schematic diagrams of preparing the specimen and measuring bending property. LD means the longitudinal direction of Mo bars, TD means the transverse direction of Mo bars.

Table 1
The transverse elongation of pure Mo bars under different condition.

The amount of upsetting (%)	Elongation δ/%	Microhardness/HV
0	1.0	195 ± 6
40	1.5	217 ± 10
70	4.0	225 ± 15
80	5.0	235 ± 15

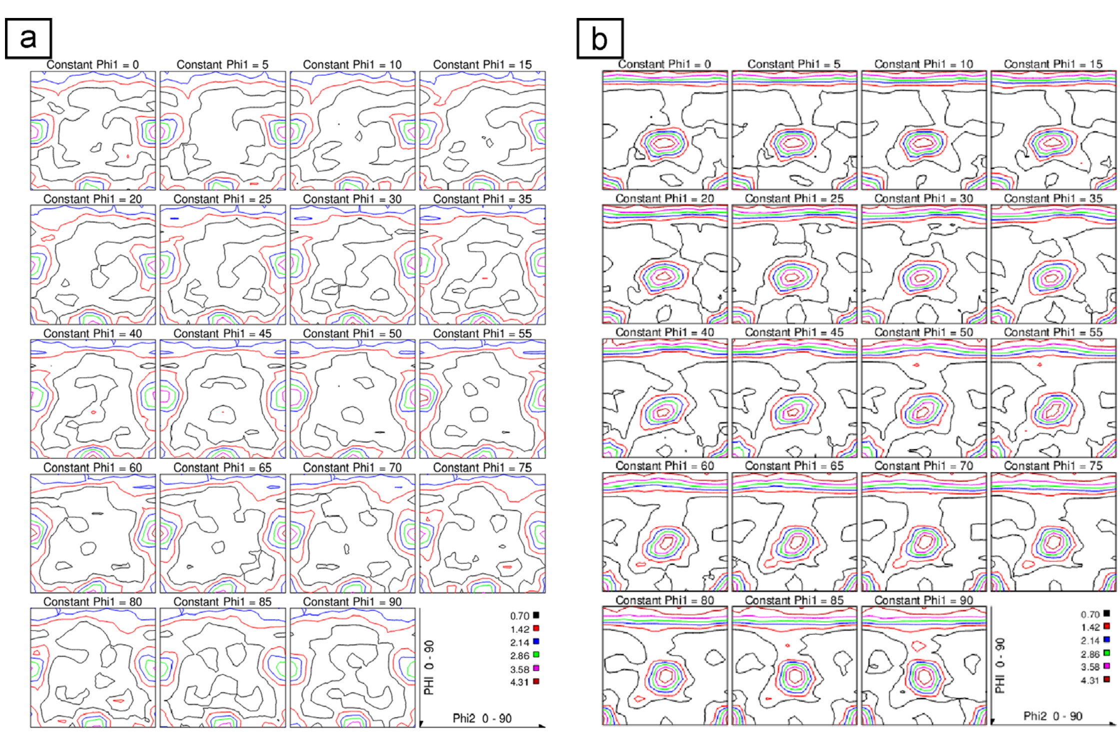

Fig. 2. Orientation distribution function (ODF) of pure Mo bars. (a) Draw-forged; (b) upset by 80%.