International Journal of Refractory Metals & Hard Materials 72 (2018) 367–372

Contents lists available at ScienceDirect

International Journal of Refractory Metals & Hard Materials

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

Recrystallization behavior of pure molybdenum powder processed by high-pressure torsion

Ping Li[a], Quan Lin[a], Xue Wang[a,b], Ye Tian[a], Ke-Min Xue[a,*]

[a] School of Materials Science and Engineering, Hefei University of Technology, Hefei 230009, China
[b] School of Mechanical Engineering, Hefei University of Technology, Hefei 230009, China

ARTICLE INFO

Keywords:
High-pressure torsion (HPT)
Molybdenum powder
Microstructure
Recrystallization behavior

ABSTRACT

High-pressure torsion (HPT) was conducted on pure molybdenum (Mo) powder (99.95 wt%) under the applied pressure of 3 GPa with different revolutions at the temperature of 623 K followed by heating to 1673 K with a rate of 20 K per minute at the pure argon atmosphere. The microstrain and dislocation density for the HPT-processed Mo were calculated by X-ray diffraction (XRD). The microstructure after HPT and the followed heating process was characterized by metallurgical microscopy, electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). The results show that the homogeneous ultrafine grains with non-equilibrium high angle grain boundaries (HAGBs) were formed in the Mo sample during HPT by continuous dynamic recrystallization (cDRX). The increasing HPT revolution results in the increase of dislocation density and the decrease of grain size, which tends to be stable beyond 5 turns due to the dynamic recovery accompanied by slight grain coarsing. Continuous static recrystallization (cSRX) occurred in the HPT-processed Mo sample with ultrafine grains during the followed heating process. The microstructure after heating is still with fine grains and homogeneous distribution even though the heating temperature up to 1673 K, which indicates the enhancement of the microstructure thermal stability.

1. Introduction

Molybdenum (Mo) has extensive applications in extremely challenging operation conditions, such as aerospace, electronics, medical diagnosis and military industries, owing to its high temperature hardness and strength, excellent wear corrosion resistance and thermal conductivity [1]. As a hard-to-deform refractory metal with body-centered cubic (BCC) crystal structure, Mo products are usually fabricated by the method of powder metallurgy (PM), which has the disadvantages of coarse grains and high contents of porosities. The conventional plastic deformation techniques, such as hot-rolling or forging, usually be used in order to close the residual pores of the sintered Mo. However, there are limits in the grain size refinement and the micro-orientation transformation of dislocations for the conventional plastic deformation techniques. Also, the applications of Mo are heavily limited by the low-temperature brittleness (ductile-to-brittle transition temperature, DBTT = 673 K–773 K) and recrystallization brittleness (recrystallization temperature, CRT = 1073 K–1373 K) [2].

The Mo with ultrafine grains (UFG, 100 nm $< d <$ 1000 nm) or nanocrystalline (NC, $d <$ 100 nm) has several advantages, such as low DBTT, high strength and toughness, good high temperature mechanical properties and enhanced thermal shock resistance [3]. Severe plastic deformation (SPD) techniques provide effective methods to produce ultrafine-grained or nanocrystalline bulk material with high density [4–7]. High pressure torsion (HPT) is a typical SPD technique. During HPT, the sample is subjected to a high applied pressure along the axial direction and a large concurrent torque along the torsional direction, which can refine grains and consolidate powder at relatively low temperatures [8,9]. In recent years, it has attained widespread attentions to consolidate powders into bulk materials with full density directly through HPT processing below the recrystallization temperature [10–18].

The objectives of this study are to produce bulk Mo with ultrafine grains from powder directly by HPT below the recrystallization temperature, and then to investigate the microstructure evolution and recrystallization behavior during HPT and the followed heating process.

2. Experimental materials and methods

Mo powder with a purity level of 99.95% was used and the morphology observed by JSM6490/LV scanning electron microscope (SEM) is shown in Fig. 1. The particle size distribution was determined by a

* Corresponding author.
E-mail addresses: li_ping@hfut.edu.cn (P. Li), xuekm@hfut.edu.cn (K.-M. Xue).

https://doi.org/10.1016/j.ijrmhm.2018.01.002
Received 10 October 2017; Accepted 5 January 2018
Available online 06 January 2018

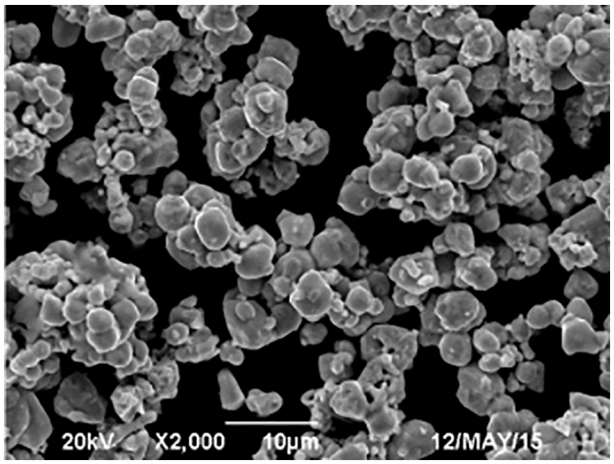

Fig. 1. The morphology of the initial Mo powder.

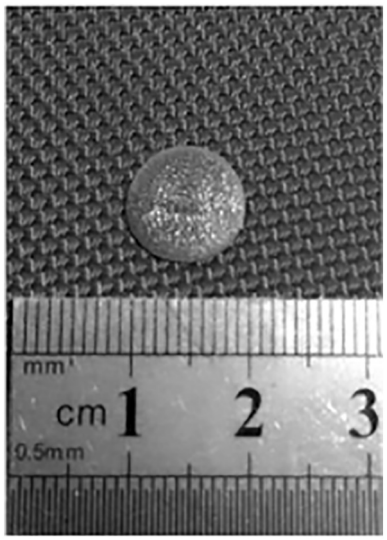

Fig. 2. Mo sample processed by HPT.

laser diffraction analyzer and the mean size of the particles is about 3 μm. Quasi-constrained HPT experiments with various revolution numbers of 5, 10 and 15 were carried out on a RZU2000HF pressing and torsion machine with an applied pressure of 3 GPa and a rotation speed of 0.67 rpm at the temperature of 623 K. Prior to HPT, the powder compact with the initial relative density of 0.6 and the size of 10 mm in diameter and 2 mm–2.5 mm in thickness was prepared and wrapped in a 304 stainless steel tube. The disk-shaped Mo sample with the relative density of above 0.99, the diameter of 10 mm and the thickness of 1 mm was obtained after HPT, as shown in Fig. 2.

For microstructure characterization, X-ray diffraction (XRD) measurement was performed on the bottom of the HPT-processed Mo sample using a D/MAX2500VL/PC XRD analyzer with Cu K_α radiation at a scanning step of 0.01°. Prior to the XRD analyses, the instrumental peak broadening of the radiation of XRD analyzer was removed and the exact peak positions of the Mo samples were calibrated by the standard Si powder. The parameters of XRD patterns for the samples were calculated through the software of Jade 6.5. Dislocation density was calculated from peak broadening analysis of XRD profiles.

The microstructure at the region with 2 mm–3 mm away from the center of the disk was studied by electron backscattered diffraction (EBSD) using a JSM-7001F scanning electron microscope. The accelerating voltage and beam current were 20 kV and 13 nA, respectively. The scanning step size was set as 0.1 μm and the analysis of the data

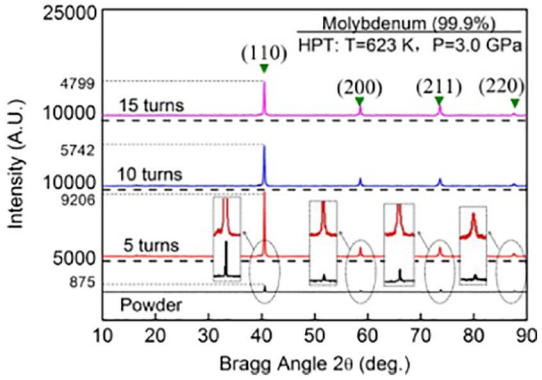

Fig. 3. XRD patterns of Mo samples with and without HPT processing.

was carried out using the EDAX-OIM software. The microstructure was characterized further by a FEI Tecnai G2 F20 S-TWIN transmission electron microscope (TEM). The TEM foils were thinned using twin-jet electro-polishing at − 243 K with an electrolyte solution of H_2SO_4:CH_3OH = 1:9 (in volume).

The HPT-processed Mo was heated to 1673 K with a constant heating rate of 20 K per minute and then cooled in the furnace to room temperature under the protection of pure argon atmosphere. The static recrystallization behavior was investigated by observing the microstructure 2 mm–3 mm away from the center of the disk using a MR2000 metallurgical microscope.

3. Results and discussion

3.1. X-ray diffraction analyses

Fig. 3 shows the XRD patterns of the Mo samples with and without HPT processing. There is an apparent peak breadths broadening for the HPT-processed Mo compared with the initial Mo powder. Generally, the broadening of peak breadths for polycrystalline metals is resulted from small crystallite size, high level of lattice distortion and their interactions, which can be depicted by the Cauchy-Gaussian function based on the assumption that the size broadening and strain broadening are Cauchy and Gaussian components, respectively [19,20]:

$$\frac{(\Delta 2\theta)^2}{(\tan\theta_0)^2} = \frac{K\lambda}{L}\left(\frac{\Delta 2\theta}{\tan\theta_0 \cdot \sin\theta_0}\right) + 16e^2 \quad (1)$$

where $\Delta 2\theta$ and θ_0 is the integral breadths (IB) and half of Bragg angle on different crystallographic planes, respectively. K is Scherrer constant and the value is 0.94 when $\Delta 2\theta$ is selected as the integral breadths for the metals having cubic crystal lattice, λ is the wavelength of the radiation of XRD analyzer, λ = 0.15406 nm, and L is crystallite size with the misorientation angle of 1°–2°. e is lattice strain related to the microstrain $< \varepsilon^2 >^{1/2}$, $e = 1.25 < \varepsilon^2 >^{1/2}$.

The multipeak linear fitting results are shown in Fig. 4. The values of crystallite size (L) and microstrain ($< \varepsilon^2 >^{1/2}$) can be obtained from the slope and the ordinate intersection from the fitting lines, respectively. The average dislocation density (ρ) due to crystallite size and microstrain can be calculated by the following equation [21,22]:

$$\rho = \frac{2\sqrt{3} < \varepsilon^2 >^{1/2}}{\|b\| L} \quad (2)$$

where b is Burgers vector with the value related to lattice parameter a in the terms of $\|b\| = \frac{\sqrt{3}}{2}a$ for bcc metals.

For the initial Mo powder, the line fitting result shows the average crystallite size of ~64.0 nm as well as a low level of microstrain and dislocation density about 0.039% and 7.76×10^{13} m^{-2}, respectively. For the sample processed by HPT with 5 revolutions, the crystallite size has an obvious decrease to ~42.8 nm and the microstrain and