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Microstructure and recrystallization behavior of pure W powder processed by high-pressure torsion

Ping Li *, Xue Wang, Ke-Min Xue, Ye Tian, Yu-Cheng Wu

School of Materials Science and Engineering, Hefei University of Technology, Hefei 230009, China

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ABSTRACT

High-pressure torsion (HPT) was conducted on pure W powder (99.9 wt.%) at the temperature of 713 K and the Vickers microhardness along the disk radius was measured. The microstructure and thermal stability after HPT were characterized by X-ray diffraction (XRD) and differential scanning calorimetry (DSC), respectively. The results show that the increasing equivalent strain resulted in grain refinement, microhardness improvement and thermal stability enhancement. The nanostructured W samples with crystallite size of 32.5 nm, dislocation density of 9.19×10^{14} m^{-2} and average microhardness of 1020 Hv were obtained through 4 GPa and 10 revolutions of HPT. For HPT samples at 2 GPa and 5 turns, uniformly distributed nucleation sites around grain boundaries led to the recrystallization temperature increasing. A complex recrystallization mechanism occurred in the sample after 4 GPa and 10 turns, including continuous dynamic recrystallization (cDRX) during HPT and continuous static recrystallization (cSRX) during DSC heating.

1. Introduction

W has extensive applications in extremely challenging operation conditions, such as in the aerospace, electronics, chemical and nuclear industries owing to its characteristic physical properties, nobler chemical stability and excellent high temperature strength [1]. In addition, W has been considered as one of the most promising candidates for plasma facing materials (PFM) applied in the controlled thermonuclear reactors (CTR) due to the outstanding comprehensive advantages [2]. However, the applications of pure W metal are heavily limited by the low-temperature brittleness (DBTT = 473 K–673 K) and the brittleness caused by recrystallization (CRT = 1173 K–1673 K) [3,4].

One of the methods for ductilization and decreasing the DBTT is grain size refinement. The ultrafine-grained or nanocrystalline W material has characteristics of low ductile-to-brittle transition temperature, high strength and toughness, good elevated temperature mechanical performance and enhanced thermal shock resistance [5]. Plastic deformation is an effective technique for grain refinement, microstructure homogenization and mechanical properties improvement to enhance the performance of W-base materials without changing their chemical composition. However, there are limits in the grain size refinement and the micro-orientation transformation of dislocations for the conventional plastic deformation techniques, such as rolling [6], swaging [7] and extrusion processing [8]. Accordingly, severe plastic deformation (SPD) techniques can achieve exceptional microstructure refinement with small grain size in the range of sub-micrometer and even nanometer and enhance the mechanical performance effectively [9]. Therefore, it provides a powerful method to produce ultrafine-grained or nanocrystalline W bulk material with high level of density [10–13] and give potential to enhance the grain boundary strength and improve the ductility.

For the common researches of ultrafine-grained or nanocrystalline W processed by SPD, the investigated materials were usually selected from the porous W materials fabricated through powder sintering processing followed by conventional thermoplastic deformation. However, a new procedure, consolidating powders into bulk materials with full density directly through SPD processing below the recrystallization temperature, has attained widespread attention in recent years, and researchers have focused on these investigations for light metal powders, such as Al and Ti powders [14–16]. In one SPD technique, high pressure torsion (HPT), the sample is subjected to a high applied pressure along the axial direction and a concurrent torque along the torsional direction, which can lead to the particles bonding and the powder consolidation. In addition, severe shear strains and extremely high hydrostatic pressure decrease the consolidation temperature and improve the comprehensive performance [17,18]. Some researches indicate that HPT could produce pure metals or composite materials from powders at relatively low temperature, such as Al powder [19,20], Mg powder [21] and W-Al mixed powders [22]. These results provide an essential foundation for fabricating pure bulk W materials directly from powders with HPT at relatively low temperature.

The objectives of this study are to consolidate W powder material with refined grains and nanostructure by using HPT processing, and

* Corresponding author.
E-mail addresses: li_ping@hfut.edu.cn (P. Li), wangxue6179@163.com (X. Wang), xuekm0721@sina.com (K.-M. Xue), clcx2009ty@163.com (Y. Tian), ycwu@hfut.edu.cn (Y.-C. Wu).

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then to investigate the microstructure evolution and the recrystallization behavior of HPT-processed W samples.

2. Experimental materials and methods

The W powder with a purity level of 99.9% was used and the morphology of the initial powder observed by JSM6490/LV scanning electron microscopy (SEM) is shown in Fig. 1. The particle size distribution was determined by a laser diffraction analyzer and the mean size of the particles is 2.5 μm. HPT experiments were carried out on a RZU2000HF pressing and torsion machine with a rotation speed of 0.67 rpm at the temperature of 713 K. Prior to HPT, the powder was prepared with the initial relative density of 0.75 and different shear strains were imposed on the powder through two series of parameters, either an applied pressure of $P = 2$ GPa concurrent with revolution number of $N = 5$ turns or a pressure of $P = 4$ GPa together with $N = 10$ turns. The disk-shaped W samples having the diameter of 10 mm and the thickness of 1 mm were obtained after HPT, as shown in Fig. 2.

For microstructure and microhardness characterization, the HPT-processed samples were first polished to a mirror-like surface on both sides and X-ray diffraction (XRD) measurements were performed at the middle part (3–4 mm away from the center) of the disk samples by using a D2500 XRD analyzer with Cu K_α radiation ($\lambda = 0.15406$ nm) at a scanning step of 0.01°. The parameters of XRD patterns, such as diffraction angle, peak breadths and lattice parameter for the HPT-processed W samples were calculated through the software of Jade 6. Second, the Vickers microhardness (Hv) along the radius of the samples at every 1.0 mm away from the disk center was measured by using a MH-3L Vickers microhardness tester under an applied load of 200 g for 15 s at four different radial directions. Third, an STA449F3 differential scanning calorimetry (DSC) was employed to test the thermal stability of HPT-processed W samples. DSC samples with the diameter of 2 mm and thickness of 0.75 mm were sliced from the middle part of HPT-processed W disks and the samples were measured by an electronic balance having the mass of 43.7 mg and 43.9 mg for 2 GPa with 5 turns and 4 GPa with 10 turns, respectively. Then the DSC sample was heated to the temperature of 1723 K with a heating rate of 20 K per minute under the protection of pure argon atmosphere. Fourth, the samples after DSC testing were polished to mirror-like surfaces with abrasive papers and an etching solution of the following composition: 25 vol.% of ammonia and 75 vol.% of hydrogen peroxide, and then a MR2000 metallurgical microscopy was used for microstructure observation.

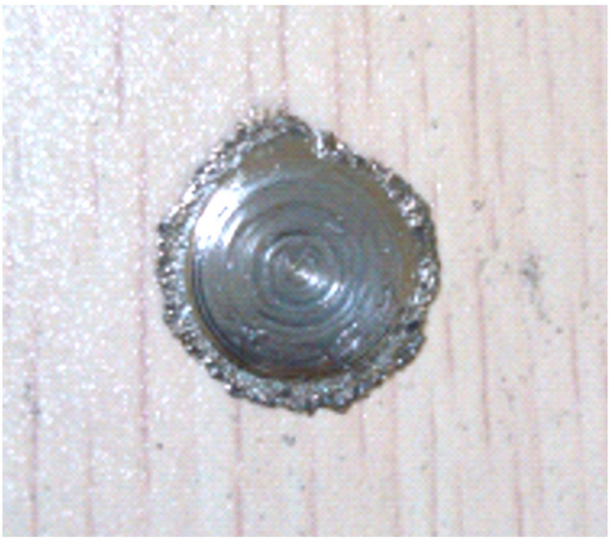

Fig. 2. W sample processed by HPT.

3. Results and discussions

3.1. X-ray diffraction analyses

Fig. 3 shows the XRD patterns for both the initial W powder and the samples processed by HPT. The statistical data, diffraction angles (Bragg Angle) and peak breadths (integral breadths, IB) on different crystallographic planes, as well as the lattice parameters, based on XRD analyses are summarized in Table 1. Prior to the XRD analyses, the instrumental peak broadening of the radiation of XRD analyzer was removed and the exact peak positions of the W samples were calibrated by using the standard Si powder. From Fig. 3, there are apparent intensity decreasing and peak breadths broadening for the HPT-processed W samples compared with the initial W powder, especially on (211) reflection, which indicate that grain refinement and plastic deformation were imposed on the W samples during HPT processing, and small crystallite size and lattice distortion have priority to occur on (211) crystallographic plane. These can be explained by the crystal structure and the plastic deformation behavior of W metal. As a refractory metal with body-centered cubic crystal structure (bcc), W has the slip system of {112}<111> under the conditions of low temperature and high strain rate according to the Peierls equation for single bcc crystal, and then the dislocations generation and slipping occurred along the direction of <111>

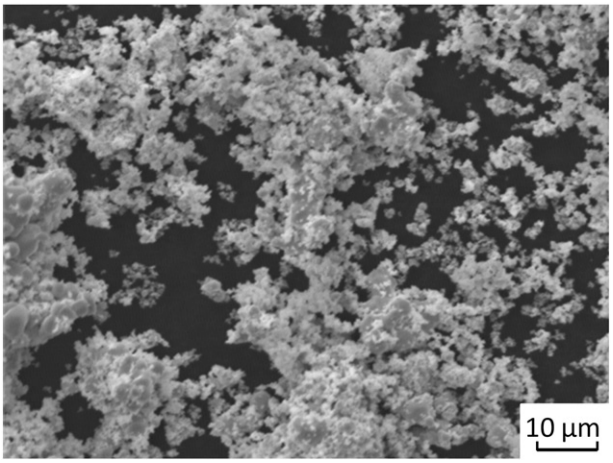

Fig. 1. Morphology of pure W powder.

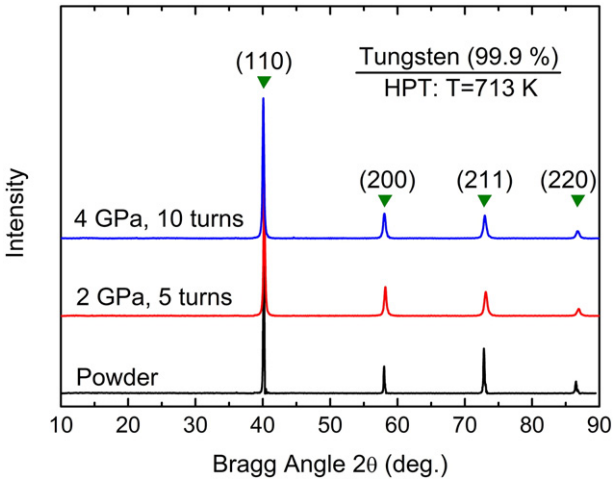

Fig. 3. XRD patterns of W samples processed by HPT, including the initial powder.