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ARTICLE

Observations on the Ductility and Thermostability of Tungsten Processed from Micropowder by Improved High-pressure Torsion

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Abstract: Tungsten was subjected to severe plastic deformation at 440 °C using high pressure torsion (HPT) with an improved die-set. Microhardness measurements suggest an appreciable ductility level in tungsten after HPT with Vickers microhardness HV as high as ~11 500 MPa. Differential scanning calorimetry (DSC) analyses show that samples with less strain have a higher recrystallisation temperature (greater than 1450 °C) than samples with more strain (~800 °C). X-ray diffraction analyses indicate increases in lattice strain up to 0.35%, lattice parameter up to 0.3177 nm and dislocation density up to 2.4×10^{15} m^{-2}. The current study introduces the improved HPT process as an effective route for the production of ultrahigh strength W with significant ductility and specified thermostability.

Key words: severe plastic deformation; consolidation; ductility; thermostability; dislocation

Tungsten is widely used in many fields such as filaments, fusion reactors and military purposes. It possesses a high melting point, high erosion resistance and good thermal conductivity. However, tungsten is a very brittle metal with a high ductile-to-brittle-transition temperature (DBTT), which is influenced by parameters such as cold work, grain size and impurities [1,2].

Recent studies on tungsten have shown that ultrafine grained (UFG) or nanocrystalline (NC) materials produced by severe plastic deformation (SPD) have properties that differ substantially from those of their coarse-grained (CG) counterparts [3-5]. UFG/NC W, compared with conventional CG W, exhibits a higher flow stress, enhanced ductility, reduced strain-hardening capacity, and reduced strain-rate sensitivity.

High pressure torsion (HPT) is a method of SPD used to produce bulk nanostructured materials with grain sizes sometimes smaller than 100 nm [6]. This procedure results in unique mechanical properties such as increased hardness and considerable ductility [7].

In the present study, W powder was subjected to HPT. The study had two main objectives: (i) to consolidate W powder directly using an improved HPT die-set and (ii) to produce tungsten with ultrahigh strength, considerable ductility and thermostability. Ductility was confirmed by observing the indentation impressions after the microhardness measurements using an optical microscope (OM) and scanning electron microscope (SEM). Thermostability was investigated using differential scanning calorimetry (DSC). The evolution of the microstructures during HPT was investigated through quantitative X-ray diffraction (XRD) analysis.

1 Experiment

The W powder had a purity level of 99.9% and an average particle size of less than 5 μm. The morphology of the W powder is shown in Fig.1. HPT was conducted at 440 °C to consolidate the powder discs of 10 mm diameter and 1 mm thickness under the nominal pressure of P = 2, 4 GPa. Shear strain, γ ($\gamma=2\pi rN/h$, where r is the distance from

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the disc centre, N is the number of turns, and h is the disc thickness), was introduced through rotations for either N = 5, 10 turns with a rotation speed of ω = 0.67 r/min.

A conventional HPT die-set is shown in Fig.2a. It is difficult to maintain the support in ideal coaxial rotation with reference to the plunger, so the clearance between the plunger/support and ring to consolidate W powder is difficult to design. If the clearance is too small, a tiny misalignment will cause interference between the plunger/support and the ring. The die will then be damaged after several turns. However, if the clearance is too large, the abrasion on the die from trimming will be considerable (because of the high hardness of W).

Therefore, the die-set has been improved as shown in the sketch in Fig.2b. In this structure, the ring has been replaced by a compressible gasket. This design provides two important advantages: (i) the off-axis fault tolerance of the support is increased significantly, thus decreasing the difficulty of HPT accordingly; and (ii) compression of the compressible gasket will exert extra pressure on the sample, and thus, ceteris paribus, the pressure on the sample will be greater than that in conventional HPT.

For development, disc samples were first polished to a mirror-like surface on both sides. Secondly, the Vickers microhardness was measured on the downward surface of a 4 GPa sample and on both surfaces of a 2 GPa sample every 1 mm from the centre using an applied load of 200 g for 20 s at four different radial directions. Thirdly, the ductility was investigated by observing the impression of the microhardness indentations after testing at a load of 200 g or 2 kg and a loading time of 20 s. Fourthly, DSC analysis was performed on the samples. The sample were heated under argon gas to 1450 °C at a rate of 20 °C/min. Finally, XRD analysis was performed on the samples using Cu Kα radiation at 40 kV and 40 mA with a scanning step of 0.02° and a scanning speed of 2°/min.

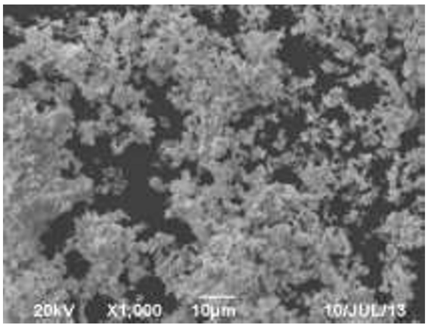

Fig.1 SEM morphology of W powder

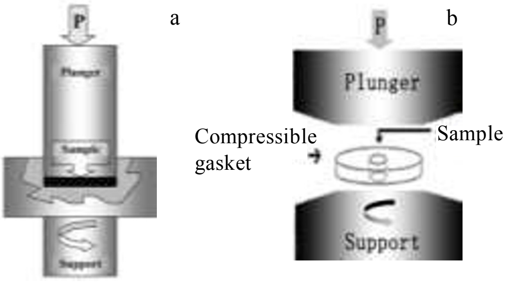

Fig.2 Sketches of conventional HPT (a) and improved HPT (b)

2 Results and Discussion

Fig.3 displays the hardness variation with distance from the disc centre after 2 GPa, 5 turns or 4 GPa, 10 turns. The microhardness increases with the increasing pressure, turns and distance from the disc centre. The saturation of the hardness appears in the 4 GPa, 10 turns disc sample at 3 mm from the disc centre. The microhardness on the bottom of the 2 GPa, 5 turns sample is higher than that on the top between the centre and 2 mm from the centre; however, the micro- hardness in the area from 2 mm to 5 mm is almost identical. The hardness HV level at saturation exceeds 12 000 MPa for the 4 GPa, 10 turns sample. Notably, these impressively high hardness levels are much higher than those of HPT-processed pure W[8,9] and intermetallic-based nanocomposites[10].

For a polycrystalline metal, microhardness can be quantitatively correlated with yield strength and grain size; i.e., a high microhardness value indicates a smaller grain size and higher yield strength. In turn, a high yield strength corresponds to a fine grain size, as determined by the conventional Hall-Petch relationship. The Hall-Petch relation for W in terms of Vickers hardness is [11]

$$H=H_0+K_H\cdot d^{-1/2} \qquad (1)$$

where, H_0 is 350 kg/mm^2, and K_H is 10 kg/mm^2; $H = H_V/0.102$. Using the data in Fig.3, we estimate the grain size of the 4 GPa sample to be approximately 140 nm near the disk edge. This result indicates that the increased level of strain induced in the W by the higher pressure and the greater number of turns increases the grain refinement.

The presence or absence of cracking around the

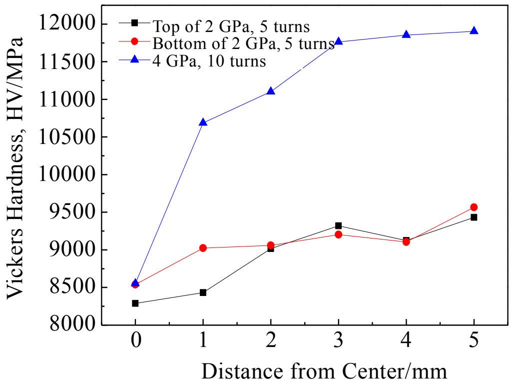

Fig.3 Variations of Vickers microhardness plotted along distance from disc centre for W