Journal of Nuclear Materials 386–388 (2009) 733–735

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

High heat load properties of ultra fine grained tungsten

Zhangjian Zhou [a,*], Jochen Linke [b], Gerald Pintsuk [b], Juan Du [a], Shuxiang Song [a], Changchun Ge [a]

[a] School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China
[b] Forschungszentrum Jülich GmbH, EURATOM Association, D-52425 Jülich, Germany

A B S T R A C T

Ultra fine grained tungsten samples with average grain size of 0.2, 1 and 3 μm were fabricated by resistance sintering under ultra-high pressure. The annealing experiments for the investigation of the material resistance against grain growth showed that recrystallization and grain growth occur obviously at heating temperature of 1500 °C. The finer the initial grain sizes of tungsten, the smaller its grain growth. The effects of transient high heat loads on tungsten surface morphology have been performed in an electron beam test facility with 4 ms pulses at different power density of 0.11, 0.16, 0.22, 0.27 and 0.44 GW/m^2, respectively. Particle erosions occurred for tungsten with 3 μm size at 0.16 GW/m^2, but for tungsten with 0.2 and 1 μm size at 0.27 GW/m^2. The weight losses of tungsten with 0.2, 1 and 3 μm size are 2, 0.1 and 0.6 mg, respectively, at 0.44 GW/m^2.

1. Introduction

Tungsten is increasingly considered as a promising candidate armour materials facing the plasma in tokamaks for medium to high heat flux components (EAST, ASDEX, ITER) [1–3]. This kind of materials will face severe problems such as thermal shock fracture, surface erosion due to sublimation, and very high heat load. Fabrication of tungsten with ultra fine grain size is considered as an effective way to ameliorate some disadvantages of tungsten, such as its brittleness at room temperature [4]. Due to the high melting point of refractory metals, it is not easy to fabricate these kinds of materials with very high density and retaining fine grain size. The research data on the performance, especially of high heat loading test, of ultra fine grained tungsten are very limit. A novel method, resistance sintering under ultra-high pressure (RSUHP), has been developed for fabrication of tungsten based materials with fine grain size [5]. The purpose of this study was at contributing to some elementary results of the high heat load performance of pure tungsten with a microstructure of fine grains size fabricated by RSUHP.

2. Experiments

For fabrication of tungsten with different grain size, W powders with average particle size of 0.2 μm and a purity of 99.2%, 1, 3 μm and a purity of 99.8% were used as the starting materials. A special experimental setup described in Ref. [5] was employed to fabricate tungsten with ultra fine grain size. The size of all samples was Φ 18 mm × 10 mm.

The density was measured by Archimedes' method. The microstructure was investigated using scanning electron microscopy (SEM). Vickers micro-hardness tests were performed at room temperature using a Vickers diamond pyramid and applying a load of 19.6 N for 20 s. Three-point-bending test at room temperature was performed on samples with an area of 15 mm × 3 mm and a thickness of 2 mm. Thermal conductivity was tested by Laser Flash Method at room temperature. The annealing experiment was done by annealing samples in a vacuum furnace at different temperature holding for 2 h, respectively. The effects of transient high thermal loads on tungsten surface morphology were performed in electron beam test facility JUDITH at Forschungszentrum Julich, Germany.

3. Results and discussion

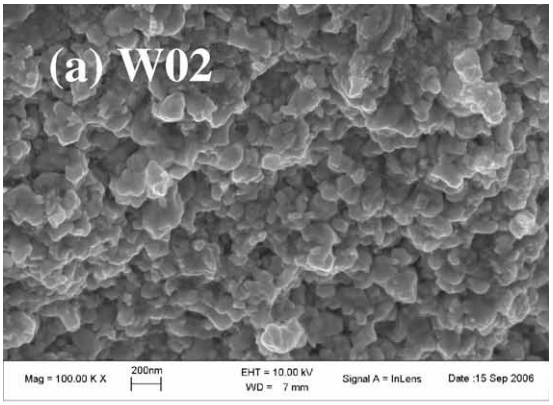
3.1. Microstructure and properties of tungsten with ultra fine grain size

One of the best advantages of RSUHP is that very short sintering time (about 1 min) will be enough, thus almost no obvious grain growth would be occurred during sintering. Fig. 1 shows the fracture morphology of sintered W named as W02, W10 and W30 with average starting powder size of 0.2, 1 and 3 μm, respectively. It was found that the grain size of the sintered pure tungsten samples were still nearly same to or even less than the particle size of the starting powders. These demonstrated that the RSUHP is a very effective method to fabricate ultra fine grained refractory metal.

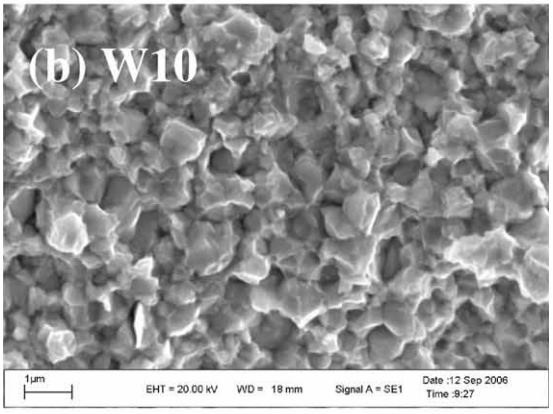
Table 1 shows the density, hardness, bending strength and thermal conductivity at room temperature of tungsten with different

* Corresponding author. Tel./fax: +86 10 62332472.
E-mail address: zhouzhj@mater.ustb.edu.cn (Z. Zhou).

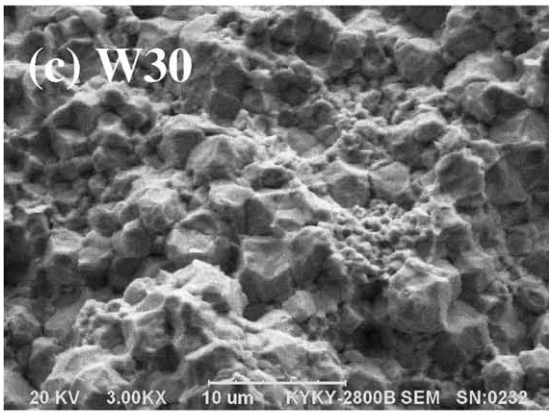
0022-3115/$ - see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2008.12.306



(a) W02



(b) W10



(c) W30

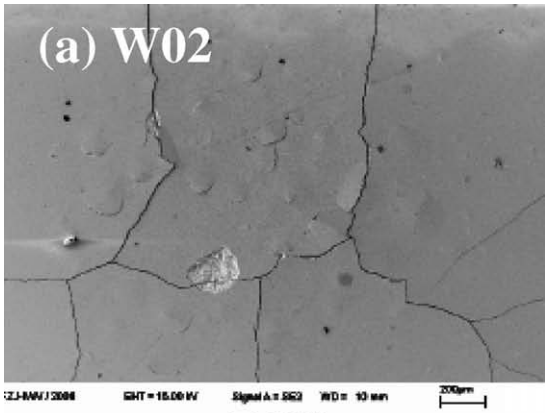
Fig. 1. Fracture morphology of sintered tungsten with starting powder size of 0.2, 1, 3 μm.

Table 1
Property of tungsten with different grain size.

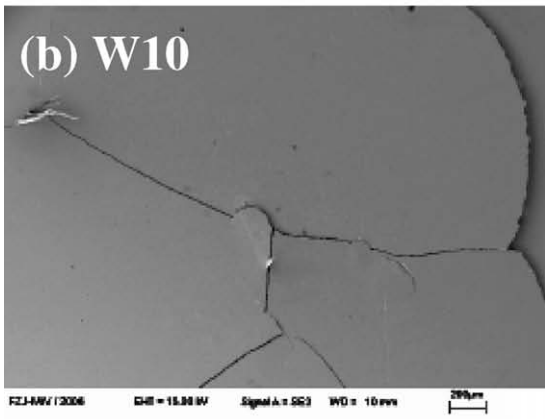
Property	Sample W02	Sample W10	Sample W30
W powder size, μm	0.2	1	3
Relative density, %	94.68	97.49	98.22
Micro-hardness, MPa	989.92	772.30	585.66
Bending strength, MPa	597.29	561.12	329.93
Thermal conductivity, W/mK	105.10	130.50	78.30

Table 2
Annealing test of tungsten with different grain size.

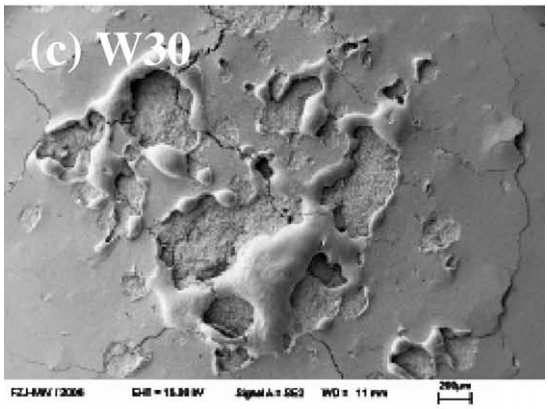
Annealing temperature, °C	Sample no.	Mean grain size, μm^2	Max. grain size, μm^2	Circle form factor
1500	W02	39	286	0.61
	W10	55	316	0.69
	W30	105	326	0.66
1750	W02	181	964	0.66
	W10	342	2123	0.68
	W30	1705	11748	0.68



(a) W02



(b) W10



(c) W30

Fig. 2. SEM micrograph of tungsten surface after high heat load at flux test 0.27 GW/m^2.

grain size. When decreasing the grain size of tungsten, although its relative density will decrease a little, its micro-hardness and bending strength will increase significantly. This result is accordance with the Hall–Petch relationship. It was a little strange for the results of thermal conductivity. Usually, for pure metal, the finer the grain size, the lower the thermal conductivity will be. But in our results, the sample of W30 showed the lowest thermal conductivity. From Fig. 1 we can see that, for W30, apart from particles nearly same to the initial powder size, there have lots of massed very fine particles which may due to powder crushing by ultra-high pressure loaded. This inhomogeneous microstructure may explain its low thermal conductivity.

3.2. Annealing test

The annealing experiments for the investigation of the material resistance against grain growth have been done by annealing samples in a vacuum furnace at 1250 °C, 1500 °C and 1750 °C holding for 2 h, respectively. It is found that recrystallization and grain growth occur at heating temperature of 1500 °C, as shown in Table 2. The finer the initial grain sizes of tungsten, the smaller its grain growth. Concerning grain growth small grains show advantages and from this point of view, W02 is preferred.

3.3. High heat load test

The effects of transient high thermal loads on tungsten surface morphology were performed in electron beam test facility JUDITH. The thermal loads tests were carried out with 4 ms pulses at different power density of 0.11, 0.16, 0.22, 0.27 and 0.44 GW/m^2, respectively. It was found that particle erosions occurred for sample W30 at 0.16 GW/m^2 and for sample W02 and W10 at 0.27 and 0.44 GW/m^2, respectively. Fig. 2 shows the surface morphology of different tungsten samples at heat load test of 0.27 GW/m^2. It can be seen that besides cracks occurred in the surface of all samples, W30 showed the heaviest particle erosion; while W10 had no particle erosion occurred. This may because that W10 had the highest thermal conductivity and thus the best heat transfer ability, while W30 had the lowest thermal conductivity and thus the worst heat transfer ability.

The weight loss of W02, W10 and W30 were 2, 0.1 and 0.6 mg, respectively, at power density of 0.44 GW/m^2. This was an interest result that although W10 has the smallest weight loss as expected, W02 shows the highest weight loss. This demonstrates that at this power density, tungsten with submicron grain size had worse performance than that of the other two micron grain sized samples. As we know that the phenomenon of agglomerations usually are easy to occur among submicron powders, these 'agglomerated particles' will be weak bonded after sintering, these may lead to a significant increase of the erosion rate under a certain high heat load.

Horizontal cracks formed for all tungsten samples at 0.22 GW/m^2, and aggravated at higher thermal power density, as shown in Fig. 2. These horizontal cracks will lead to surface roughening, material lifting and erosion when they were exposed to higher heat load. The occurred horizontal cracks indicated that there existed some weak bonding of tungsten particles in all tested samples, which may due to the special sintering method we used. Further processing amelioration should be studied to improve the grain to grain bonding and microstructure homogeneous of the ultra fine grain tungsten.

4. Conclusions

Tungsten with ultra fine grain size had been fabricated by resistance sintering under ultra-high pressure. The densities of the samples were higher than 94%. When decreasing the grain size of tungsten, its hardness and bending strength increase significantly.

The annealing experiments for the investigation of the material resistance against grain growth showed that recrystallization and grain growth occur at heating temperature of 1500 °C. The finer the initial grain sizes of tungsten, the smaller its grain growth.

The high thermal loads on tungsten surface morphology were performed by electron beam test facility with 4 ms pulses at different power density. Horizontal cracks formed for all tungsten samples at 0.22 GW/m^2. Particle erosions occurred for tungsten with size of 3 μm at 0.16 GW/m^2, but for tungsten with 0.2 and 1 μm size at 0.27 and 0.44 GW/m^2. The weight loss of tungsten with 0.2, 1 and 3 μm size are 2, 0.1, 0.6 mg, respectively, at 0.44 GW/m^2.

Acknowledgements

The authors would like to express their thanks for the financial support of National Natural Science Foundation of China under Grant No. 50634060 and National Basic Research Program of China under Grant No. 2007ID102.

References

[1] M. Roedig, W. Kuehnlein, J. Linke, M. Merola, et al., Fus. Eng. Des. 61&62 (2002) 135.
[2] H. Bolt, V. Barabash, W. Krauss, et al., J. Nucl. Mater. 329–333 (2004) 66.
[3] K. Krieger, H. Maier, R. Neu, J. Nucl. Mater. 266–269 (1999) 207.
[4] H. Kunrishita, Y. Amano, S. Kobayashi, et al., J. Nucl. Mater. 367–370 (2007) 1453.
[5] Z.J. Zhou, Y.S. Kwon, J. Mater. Process. Technol. 168 (2005) 107.