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Progress of research on plasma facing materials in University of Science and Technology Beijing

Chang-Chun Ge *, Zhang-Jian Zhou, Shu-Xiang Song, Juan Du, Zhi-Hong Zhong

Research Center on Fusion Materials (RCFM), School of Materials Science and Engineering, University of Science and Technology Beijing (USTB), Beijing 100083, China

Abstract

In this paper, we report some new progress on plasma facing materials in University of Science and Technology Beijing (USTB), China. They include fabrication of tungsten coating with ultra-fine grain size by atmosphere plasma spraying; fabrication of tungsten with ultra-fine grain size by a newly developed method named as resistance sintering under ultra-high pressure; using the concept of functionally graded materials to join tungsten to copper based heat sink; joining silicon doped carbon to copper by brazing using a Ti based amorphous filler and direct casting.

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Keywords: Graded materials; Tungsten; Graphite; Plasma facing materials

1. Introduction

Tungsten and Carbon based materials are the most promising candidates for plasma facing materials used in fusion reactor [1,2]. Significant progress has been obtained on tungsten and carbon based plasma facing materials. But there are still several difficult problems that needed to be solved. One of the problems is the brittleness and high DBTT of tungsten. Fabrication of tungsten with ultra-fine grain size is considered as one of the best solution way. The other problem is the joining technology of tungsten or carbon based materials to copper based heat sink. The concept of functionally graded materials (FGM) is efficient to solve this problem.

On ICFRM-9 (1999), we have made a presentation titled 'Development of Functionally Graded Plasma facing materials' [3]. Since then, we have made some progress in research on plasma facing materials, especially on fabrication of tungsten with ultra-fine grain size, and graded joining of W–Cu and C–Cu with Ti based amorphous filler.

2. Tungsten with ultra-fine grain size

Fabrication of tungsten with ultra-fine grain size is very important to increase the strength and decrease the DBTT of tungsten. But it is not easy to fabricate fine grain sized tungsten, because of its high melting point and high sintering temperature

* Corresponding author. Fax: +86 10 62332472.
E-mail address: ccge@mater.ustb.edu.cn (C.-C. Ge).

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needed. In our laboratory, plasma spraying is developed to fabricate tungsten coating with ultra-fine grain size, and a new technology named as resistance sintering under ultra-high pressure has been developed to fabricate bulk tungsten with ultra-fine grain size without any sintering additive added.

2.1. Tungsten coating with ultra-fine grain size

Tungsten coating with ultra-fine grain size has been fabricated by atmosphere plasma spraying. The initial powder size of tungsten is 0.2 μm, as shown in Fig. 1. It is granulated to particle size of about 30–50 μm for increasing its fluidity that is necessary for plasma spraying. The substrate is oxygen free copper with size of $\varnothing 50 \times 10$ mm. Fig. 2 shows the micro-graph of the coating. The thickness of the coating is about 600 μm. The grain size of tungsten is less than 1 μm. The primary high heat flux test shows that this kind of W coating has higher thermal shock resistance than the normal W coating by plasma spraying.

2.2. Bulk tungsten with ultra-fine grain size

Bulk tungsten with ultra-fine grain size has been fabricated by a newly developed technology named as resistance sintering under ultra-high pressure (RSUHP). The starting powder size of tungsten is about 1 μm. The sample size is $\varnothing 20$ mm $\times$ 18 mm. Fig. 3 shows the micro-graph of the tungsten powder and the fracture surface after sintering. Table 1 shows the property of pure tungsten with different grain size. It is found that as the grain size of tungsten decreases, its micro-hardness and bending strength will increase significantly.

3. W/Cu FGM

W/Cu FGM was fabricated by the technology of RSUHP. W powder with average particle size of 1 μm and purity of >99.5% and Cu powder with particle size of −200 mesh and purity of >99% are used. W powder and Cu powder are mixed and milled with different volume ratios. Experiments

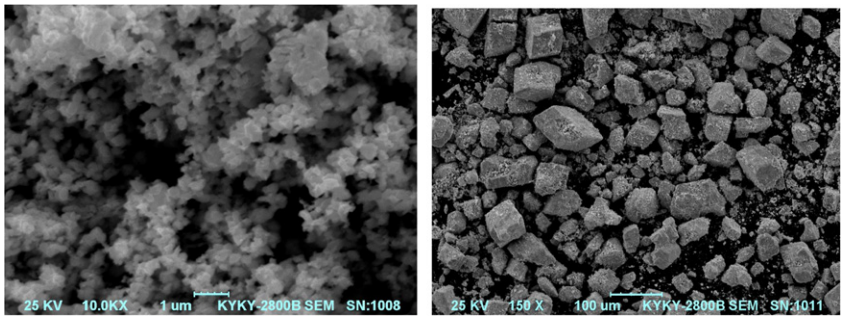

Fig. 1. The micro-graph of 0.2 μm W powder and its granulate.

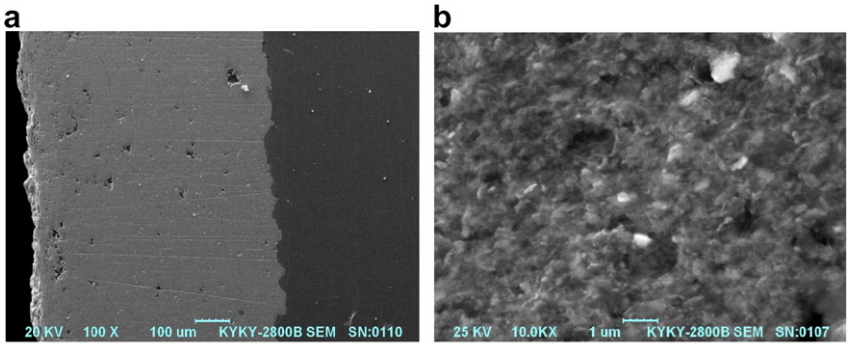

Fig. 2. The micro-graph of W coating.

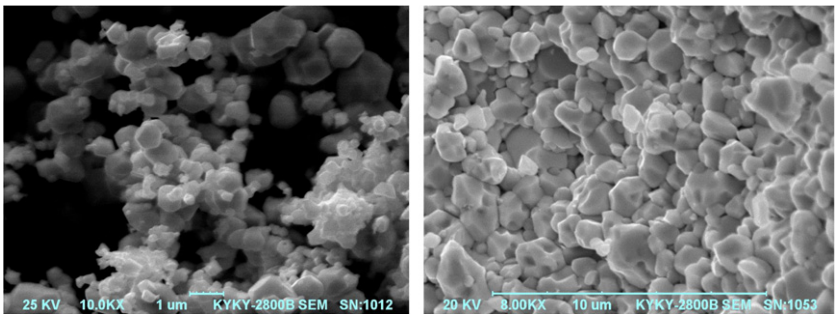

Fig. 3. Micro-graph of tungsten powder with 1 μm size and the fracture surface after sintering.

Table 1
Property of pure tungsten with different grain size

Property	Sample A	Sample B	Sample C
W powder size (μm)	0.2	1	7
Relative density (%)	95.91	97.49	98.40
Micro-hardness (MPa)	1169.80	772.30	592.20
Bending strength (MPa)	596.29	561.12	453.51

on the six-layered W/Cu FGM are conducted. The mixed powder with different compositions are stacked layer by layer in a steel mould to form a green compact with a diameter of 20 mm and height of 5–10 mm. Then the compact was sintered under ultra-high pressure of 5 GPa, loaded strong current for about 1 min. Fig. 4 is the backscattering image of the cross section of overall six-layered FGM, a good graded compositional transition is found. This reveals that there was no obvious composition migration during very short sintering time.

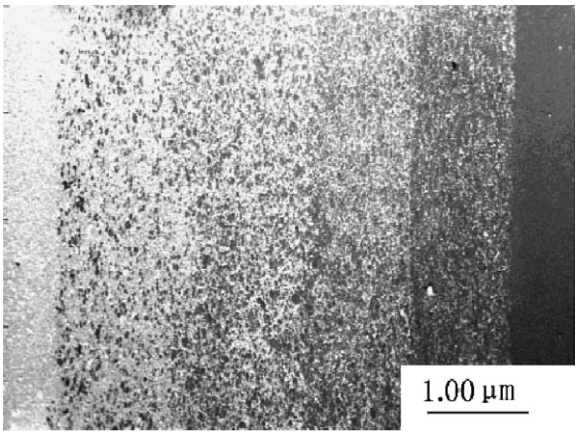

Fig. 4. SEM micro-graph of well-sintered W/Cu FGM.

4. Joining of carbon to copper

Two methods to join silicon doped carbon to oxygen free copper have been developed in USTB. One method concerns the use of a Ti-based amorphous filler metal. The other method is a new technique mixing pure copper powder with transition metal powder of VI B group first and then directly casting this mixed powder on the doped carbon.

4.1. Brazing technique

The brazing process was performed in a vacuum furnace. The 'sandwich like' samples (carbon-brazing alloy-interlayer-brazing-copper) were put in a special stainless steel sample-holder in order to avoid the misalignment during the brazing process. The brazing temperature was 900 °C and the holding time was 15 min, heating rate is 10 °C/min.

The Ti-based brazing alloy used in this work is an amorphous silver-free alloy with a liquidus temperature at 850 °C. The presence of titanium in the alloy improves the wettability and the adhesion to the carbon substrate.

Fig. 5 shows the cross section morphology of brazed joint. No cracks appear in the brazing alloy and in the copper. The C/brazing alloy and the brazing alloy/copper interfaces are homogeneous and pore-free. It can be seen that Ti was diffused into graphite about 5 μm and formed TiC on the interface of C/brazing alloy.

The mechanical shear strength was tested under different conditions. The highest shear strength was 25 MPa. Analysis of the fracture surfaces revealed that the cracks propagated through the graphite where near the C/brazing alloy interface. The C/brazing alloy interface and the brazing

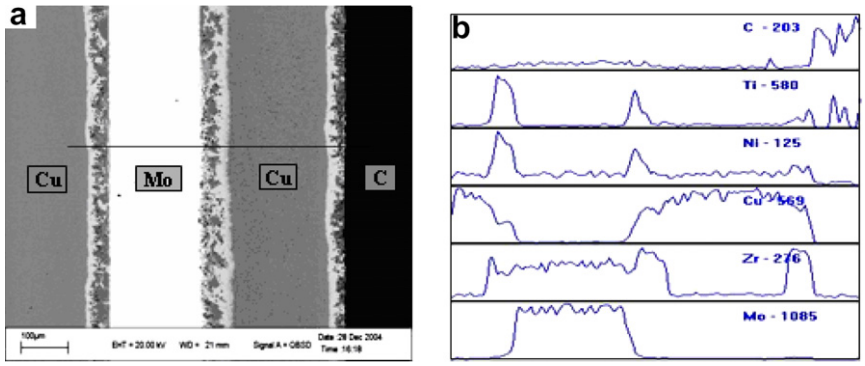

Fig. 5. Morphology and EDX of brazed joint with interlayer.

alloy/copper interfaces did not fail: this behavior indicates a strong adhesion of the brazing alloy to both the doped graphite and the copper.

4.2. Direct casting

The C–Cu joints were obtained by direct casting copper alloy at high temperature (>1100 °C) in a vacuum furnace for ~50 min. The carbon surface was tool machined before direct casting and the machining depth was about 200 μm.

The very high contact angle of molten copper on carbon ($\theta = 140°$) forbids the direct casting of pure copper on carbon based composites. In this work, the direct casting of copper on doped graphite was possible through mixing transition metal to decrease the contact angle between graphite and copper. The reaction between the transition metal and graphite determined the formation of a thin carbide layer on the graphite surface, as detected by X-ray diffraction analysis: this carbide was chosen because of its high wettability by molten copper. The wetting angle of copper alloy on this carbide was lower than 45° at 1130 °C by calculation. This low contact angle allowed the casting of copper alloy on the doped graphite.

The morphology of the C/Cu-alloy samples prepared at 1130 °C for 40 min was analysed by SEM. The clear C/Cu-alloy interface can be seen in Fig. 6(a), it is present also in the grooves of the doped graphite surface, formed during the mechanical machining. The molten copper alloy powder is infiltrated into all of these grooves, and no void is found at the interface. In Fig. 6(b), we can find that the thickness of the dense carbide layer is about 7 μm, the two interfaces (C/carbide and carbide/Cu-alloy) are continuous and no cracks or pores are detected in the samples. Furthermore, although the large thermal expansion mismatch between copper alloy and doped graphite, no cracks are present in the doped graphite or at the interfaces after cooling from high temperature (>1130 °C, cooling rate about 5 °C/min). However, there are some cupped pits in the carbide layer, studies on the appearance of these pits in the carbide layer should be carried out, because it may decrease the heat transfer through the layer. This joining technique did not limit the thickness of the copper alloy layer: up to 6 mm have been cast on doped graphite.

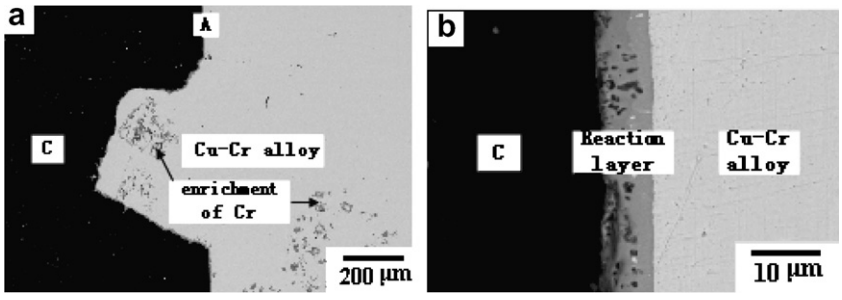

Fig. 6. The cross section of C–Cu joint by direct casting.

5. Conclusion

Tungsten coating with ultra-fine grain size can be fabricated by plasma spraying when using tungsten powder with sub-micro-size.

Tungsten with ultra-fine grain size can be fabricated by resistance sintering under ultra-high pressure. As the grain size of tungsten decreases, the micro-hardness and bending strength will increase significantly.

Ti based amorphous filler is very effective for brazing silicon doped carbon to copper. Direct casting is also a promising method to joining carbon to copper with low cost.

Acknowledgements

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