Mechanical properties of tantalum-tungsten interlayer between tungsten tile and thimble to prevent helium leak from He-cooled divertor

Pingping Zhang, Weiping Shen, Yanan Zhou, Qingling Zhang
Institute of Nuclear Materials, School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China

E-mail: shenwp@ustb.edu.cn

Abstract. The tungsten parts made of pure tungsten tile and dispersion strengthened tungsten thimble with 3 mm interlayer of tantalum-tungsten alloy are fabricated by Spark Plasmas Sintering (SPS). The process of SPS is that the temperature is raised to 1700 °C at a rate of 100 °C/min and kept for 3 min, under a constant pressure of 50MPa along the Z-axis. The mechanical properties of the interlayer with different percent of tantalum are measured. The results show that with increasing percent of tantalum, the hardness first increases and then decreases; and as the indentation on the sample is closer to dispersion strengthened tungsten, the value of Vickers hardness is much higher. The Vickers hardness of interlayer is the highest when the content of tantalum is 50% and the indentation is next to dispersion strengthened tungsten. Bending strength drops with increasing content of tantalum, when the content of tantalum is 100% the value of bending strength is the lowest. The fracture toughness is highest as the content of tantalum is 25%, the value is 9.89MPa•m$^{1/2}$. The toughening tungsten-tantalum interlayer between tungsten tile and thimble would better prevent helium leak from He-cooled divertor for DEMO.

1. Introduction

Different concepts of He-cooled divertor have been studied, which shapes are finger (HEMJ or HEMS), T-tube and plate [1]. The concept of HEMJ (He-cooled modular divertor with multiple-jet cooling) was developed at KIT several years ago. Selection of suitable materials and technologies to manufacture the HEMJ is the most difficult, because divertor design requires surviving at cyclic heat load of 10-15 MW/m$^2$. During operation, the temperature of the tile is raised to 1000 °C, while it remains 600-700 °C at thimble/steel joining area [2]. To satisfy this situation, many kinds of materials have been used, such as pure W, La$_2$O$_3$-W, brazing alloy, etc. The other difficulty is manufacture, many methods have been researched, such as PM (powder metallurgy), EDM (electro discharge machining) and conventional machining (turning, milling, grinding). Each of these methods has its own advantages and disadvantages.

To make use of He-cooled divertor, tungsten is regarded as the most suitable armor material, because it possesses a good thermal conductivity, high melting point, and high strength. And the PM is regarded as a suitable method to manufacture He-cooled divertor. But the micro-cracks between tile
and thimble should be took care, from the previous experiments, it was found micro-cracks at bottom of the tile, and the micro-crack will go through the tile and thimble because low thermal fatigue resistance of pure tungsten tile, very thin brazing filler alloy and dispersion-strengthened tungsten thimble in high-heat-flux (HHF) [3,4,5]. To solve the problem of cracking, a new design of He-cooled divertor such as adding a few millimeter interlayer should be studied.

In this paper, pure tungsten and dispersion-strengthened tungsten (W-2% TaC) were also used for tile and thimble respectively, Spark Plasma Sintering (SPS) for the fabrication of He-cooled divertor. The characteristic of this method are SPS could make the high densities and the interlayer was used to block crack propagation. Different interlayer will have different mechanical properties. This paper will discuss the mechanical properties of samples with different interlayer to find the optimizations.

2. Experimental procedure

Two kinds of powders were used for the interlayer, tungsten and tantalum. Tungsten was supplied by Xiamen Golden Egret Special Alloy Co. Ltd, the size of tungsten powders is 2μm and the content of oxygen is 0.045%. Tantalum was commercial powder, the nominal size of which is 48μm. Tantalum carbide (TaC) was synthesized by a liquid precursor route, the average size of particles is about 50nm with 5% free carbon. The progress of fabrication contains two steps, the first step was mixing the powders of tungsten and TaC by ball milling, the second step was sintering the samples with the interlayer by SPS.

Ball-milling process was performed at room temperature in a horizontal planetary ball mill [4] using tungsten rods of φ8mm×10mm as grinding media. The raw materials were mixed with the powers of tungsten and nano-TaC in a weight ratio of 10:1 (total mass of 100 g), followed by ball-milling at a rotating speed of 150 r/min under Ar atmosphere for 6 h. And then, the mixed powders were charged into an SPS graphite die of 20 mm in diameter. W-TaC powders were at bottom of graphite die, W-Ta powders were at the middle of graphite die, and pure tungsten powders were at top of graphite die. Graphite paper was filled between the graphite die and powder blend. Then put the die into the SPS furnace to sintering. After the SPS chamber was evacuated (about 10Pa), the temperature was raised to 1700 °C at a rate of 100 °C/min and kept for 3 min, under a constant pressure of 50MPa along the Z-axis.

The samples were cut in size of 13mm×3mm×4mm by EDM to test bending strength and fracture toughness with a notch in interlayer about 3mm. The microstructure of fractures was observed using a scanning electron microscope (SEM), the bending strength and fracture toughness was measured using three-point bending of 10mm span, and the Vickers hardness of samples was determined at a load of 100g and for 10s. The measured hardness value was calculated at an average of 5 different points on the same line of sample.

3. Results and discussion

3.1. Bending strength of the interlayer

The bending strengths of interlayer with different content of Ta are shown in Fig.1. It is apparent that the bending strengths decrease with increasing content of tantalum in the interlayer. When the content of tantalum is 0, the bending strength is the highest, reaches about 820MPa. When the content of tantalum is 100%, the bending strength is the lowest, decreases to about 140 MPa. The force impending dislocation motion decreases when the content of tantalum increases. There are two reasons to explain the value of bending strength is low.

First, with increasing content of tantalum, the size of grain becomes larger and the number of grain boundary becomes much fewer. Fig. 2 shows the microstructure of the interlayer with different content of tantalum. Due to grain boundaries become much fewer, the force impending dislocation motion will decrease.

And then, the other reason for the falling bending strengths is the lattice constant. Lattice constant increases linearly when the concentration of tantalum increases [6]. According to dislocation theory,
slip plane spacing increases with an increase of lattice constant. It means that the width of the dislocation will be wider, which reduces the resistance to lattice dislocation motion. So the bending strength shows a downward trend with increasing the content of tantalum (Fig.1).

![Fig.1. Bending strength of interlayer with different content of tantalum at room temperature](image)

![Fig.3. Vickers hardness of W-Ta interlayer with different content of Ta in SPS samples](image)

(a) Coarse granular fracture of 100% tantalum  
(b) Fine granular fracture of 100% tungsten

![Fig.2. The microstructure of different interlayer cross-section by SEM](image)

### 3.2. Vickers hardness of the interlayers

The Vickers hardness of different content of Ta in samples (Fig.3) clearly shows the Vickers hardness depending on the content of Ta and the distance to the dispersion strengthened tungsten. In Fig.3, the left presents pure tungsten and the right presents dispersion strengthened tungsten with 2% TaC. It shows that the Vickers hardness of dispersion strengthened tungsten is higher than the pure tungsten. The Vickers hardness of interlayer are around 500–1100HV0.1/10. As the content of tantalum increases, the Vickers hardness first increases and then decreases. So the value of hardness is highest when the content of Ta is 50%. And the Vickers hardness is much higher when the indentation is closer to the dispersion strengthened tungsten. For the samples, when the indentation is away from the center about 0.5 mm, the hardness is the highest. So when the content of tantalum is 50%, at the indentation away from center about 0.5 mm, the Vickers hardness is the highest, the value is about 1100 HV0.1/10. But as the content of tantalum is 100%, the Vickers hardness is highest at the indentation away from center at 1mm.
The Vickers hardness is always affected by the microstructure of samples, the hardness is mainly depending on the solid solution and grain boundary hardening. The contribution of grain boundaries can be described by Hall-Petch equation [7].

$$\sigma = \sigma_0 + kd^{0.5}$$

where $\sigma_0$ is the intrinsic hardness of the material, $d$ is the diameter of grain and $k$ is a material constant. When the content of Ta was a little, Ta will strengthen the materials as solid solution composition, so the hardness increases when the content of Ta increases.

For solid solution hardening of W in Ta, the hardening effect can be calculated using the linear hardening model based on the elastic size misfit interaction with screw dislocations [7, 8].

$$\sigma = 0.1274c$$

where $c$ is the atomic concentration of W. For the equation, Vickers hardness will decreases as the content of Ta increases. Because of these two mechanisms, the boundary hardening plays a major role when the content of tantalum is low; the solid solution plays a major role when the content of tantalum is high. So with increasing content of tantalum, the Vickers hardness first increases and then decreases. The Vickers hardness will increase as the indentation is close to dispersion strengthened tungsten. It indicates that free carbon in the nano-TaC powders diffuses into the W-Ta layer to form tungsten carbide or tantalum carbide, as the Vickers hardness of tungsten carbide and tantalum carbide is higher than tungsten and dispersion strengthen tungsten, so the hardness of interlayer is higher than tungsten and dispersion strengthened tungsten. The different content of carbon diffused into W-Ta and then forms different phases when the distance is different. The content of carbon diffused in W-Ta and dispersion strengthened tungsten shows in Fig.4. The left of sample is the interlayer of W-Ta, the right is dispersion strengthened tungsten. It might be well to point out that the content of carbon in the right is more than it in the left, and then the content of tungsten carbide and tantalum carbide are more, so the right Vickers hardness is higher.

Fig.4. Carbon diffused in a sample from W-Ta interlayer (left) to dispersion strengthened tungsten (right).

3.3. Microstructure and fracture toughness of the interlayer

The thermal cracking is regarded as the important problem to be solved in He-cooled divertor of DEMO, so
the new design of adding the interlayer of tungsten-tantalum is studied. The fracture surface of different content of Ta in samples (Fig.5) clearly shows the size of grain gets much larger and from brittle intergranular fracture to transgranular cleavage fracture appears in the fracture surface with increasing content of tantalum. It suggested that the bending force between grains is different with different content of tantalum. It may be that a solid solution has been formed between tungsten and tantalum. And then the cracking can’t go along the grain boundaries, it have to need much more energy to go through the grain. But the number of grain boundary gets fewer with increasing content of tantalum, the energy to go through the grain will get low. So it can be said the interlayer of tungsten-tantalum alloy can block crack propagation when the content of tantalum is proper.

Fig.5. Microstructure of fracture surface in interlayer with different content of tantalum by SEM:

(a) The content of tantalum is 0;
(b) The content of tantalum is 25%
(c) The content of tantalum is 50%;
(d) the content of tantalum is 75%;
(e) the content of tantalum is 100%

Fig.6. Fracture toughness of interlayer with content of the tantalum at room temperature
Fracture toughness is the most important mechanical properties of materials to describe the ability of blocking crack propagation. Fig 6 shows the fracture toughness of interlayer with different content of Ta. It was shown that the fracture toughness is rise and fall. The fracture toughness is highest when the content of tantalum is 25%, the value is 10.08MPa·m\(^{1/2}\). There are three categories of possible mechanisms affecting the fracture toughness. The first one is the effects of flaws, the second one is the effects of interfaces, and the last one is related to the effect of interfaces involving the deformation mechanisms [9].

For these reasons, the most important actor is the force impeding grain boundary sliding. With increasing content of tantalum, the forces of impeding grain boundary are different. When the content of tantalum is 25%, the flows and interfaces are much relatively, it means that the grain boundary strength and the force are large, so the value of fracture toughness is the highest.

Some phenomenons can be found through compare Fig.5 and Fig.6. The brittle intergranular fracture of coarse grains and pores in Fig.5 (a) and then the interlayer has lower fracture toughness. Transgranular cleavage and intergranular fracture of fine grains make more crack deflections in Fig.5 (b). And the highest fracture toughness, and fracture toughness reduce gradually with increasing grain size of transgranular cleavage fracture in Fig.5(c)-(e). By the same reason, content of about 25% Ta in W, proper grain size and moderate interlayer thickness have better He leak-proof between tungsten armor and thimble of HEMJ by thermal shock.

4. Conclusions

Pure tungsten and dispersion strengthened tungsten with interlayer of W-Ta alloys with Ta composition from 0 to 100% were fabricated by Spark Plasmas Sintering (SPS) and their Vickers hardness, bending strength and fracture toughness were measured. Conclusions derived from the tests are given as follows:

(1) With increasing content of tantalum, Vickers hardness first increase and then decrease. When the content of tantalum is 50%, the Vickers hardness is the highest. In the interlayer, the Vickers hardness is much higher when the indentation is closer to the dispersion strengthened tungsten. So when the content of tantalum is 50% the Vickers hardness at the indentation between W-Ta and dispersion strength is the highest, the value is 1100 HV0.1/10.

(2) The plasticity of tantalum is higher than tungsten, and the hardness is lower than tungsten, so with increasing content of tantalum from 0 to 100%, the bending strength dropped precipitously from 820MPa to 120MPa.

(3) The transgranular cleavage fracture appears in surface when the content of tantalum increases, it means that cracking need more energy to go through the materials with increasing content of about 25% tantalum.

(4) The fracture toughness is first increasing and then decreasing, with increasing content of tantalum. When the content of tantalum is 25%, the fracture toughness is the highest, the value is 9.89 MPa·m\(^{1/2}\). When the content of tantalum is 100%, the fracture toughness is the lowest, the value is 7.57 MPa·m\(^{1/2}\). Presumably the tungsten-tantalum alloy interlayer with proper tantalum content between tungsten tile and thimble of mock-up can preferably prevent helium leak from He-cooled divertor.

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6. References

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