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Improvement of mechanical property of air plasma sprayed tungsten film using pulsed electric current treatment

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ABSTRACT

The air plasma spraying method has a problem in the formation of many splat boundaries throughout the plasma sprayed layer, which seriously degrades mechanical properties of the metal layer. In order to improve the mechanical properties of air plasma sprayed tungsten (APS-W) by reducing the splat boundaries, a pulsed electric current is applied on as-sprayed specimens. Through microstructure observations and microhardness measurements, it was confirmed that the reduction of splat boundaries and the increase of hardness were obtained. The results are compared with those of APS-W annealed using induction heating under the same temperature, time and pressure.

1. Introduction

In the early development of plasma-facing components (PFCs) for fusion reactor, low atomic number (low-Z) carbon materials were investigated as a suitable material. Low central radiation loss, high melting point, light weight and good thermal conductivity made low-Z carbon materials attractive. However, the application of low-Z carbon materials is limited due to high tritium retention, degradation of thermal conductivity from neutron damage and a high erosion rate at elevated temperatures [1–3]. As an alternative, tungsten has been considered as a strong candidate material for PFCs, since tungsten has good properties to endure the harsh environment of a fusion reactor: high melting point, high thermal stability, low sputtering rate, low tritium retention, low erosion rate and high thermal conductivity [4–9]. However, the actual application of bulk tungsten materials is limited due to its embrittlement at low temperature and high atomic number (high-Z). The embrittlement of tungsten in low temperature limits formability and high-Z of tungsten causes high central radiation loss and imposes high stress to the reactor structure [2,9]. To overcome the weak points of bulk tungsten, the deposition of tungsten on a substrate composed of structural materials such as reduced activation ferritic martensitic (RAFM) steel or graphite has been investigated [9–21]. In lightweight tungsten-coated PFCs, the tungsten layer faces the plasma and the substrate supports the first wall. It is possible to fabricate a complex-shaped structure through the depositing process.

Several deposition methods have been suggested for fabrication of a tungsten layer on a substrate such as physical vapor deposition, chemical vapor deposition and plasma spraying. In this study, the air plasma spraying (APS) method, which carries advantages over aforementioned deposition methods due to its large coating areas, *in-situ* repair-ability, low deposition cost and fast deposition rate, was utilized [16–18]. However, the APS method results in the formation of many splat boundaries containing voids and oxides which lead to poor mechanical properties and limit actual application [18–21]. Microhardness of the air plasma sprayed tungsten (APS-W) is reported to have <30% of that of bulk tungsten [18]. Various studies to improve the mechanical properties and microstructure of the APS-W through post-treatments have been reported. However, no remarkable results have been reported [18,19].

In this study, a pulsed electric current treatment (PECT) with high current density was investigated to improve the mechanical properties of the APS-W. It is known that the application of an electric current through a metal can change the microstructure and increase the plastic deformation (often called 'electroplasticity') [22,23]. Various probabilities have been suggested on the electric current effect such as thermal effects due to Joule heating effect [24,25], electron wind effect [26,27] and electrostatic field effect [28,29]. Recently, one of authors had obviously demonstrated that the electric current enhances the atomic diffusion in metals which may accelerate a microstructural change [30–34]. Therefore, the post-treatment for APS-W using an electric current was introduced to reduce the splat boundaries and to evolve the sound

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microstructure without a severely elevated temperature, which may critically damage the substrate. For example, RAFM steels have lower melting points than the treatment temperature of tungsten. To confirm the effect of PECT, the microstructure and microhardness after the treatment were compared with those annealed using induction heating under the same temperature, time and pressure. The microstructural observations for the specimens were performed by a field emission scanning electron microscope (FE-SEM) with electron backscatter diffraction (EBSD). In addition, the microhardness values of the specimens were measured to evaluate the improvement of the mechanical properties.

2. Materials and methods

Commercially available tungsten powders with a diameter of approximately 44 μm were sprayed on a substrate composed of graphite (Φ5 × 3 mm) with the APS method using lab-made equipment. Deposition conditions were optimized to produce tungsten coating with low porosity and oxide contents [35] (Table 1). During the spraying process, argon was used as the primary and carrier gas and hydrogen was used as a secondary gas to reach a higher temperature than would be possible using argon alone. The thickness of the sprayed tungsten layer was 200 μm.

After deposition, one of the specimens was annealed using a vacuum furnace (Lindberg/Blue M, Thermo Electron Corporation) in order to improve the mechanical properties. Under a pressure of 0.1 Torr, the tungsten layer was first annealed in a vacuum furnace at 800 °C for 1 h and then at 950 °C for 2 h for the reduction of tungsten oxides on splat boundaries [18,36,37].

Two different treatments, press-annealing (PA) and PECT, were conducted for the as-sprayed specimens. PA is a post-treatment method using induction heating with pressure. In both treatments, the pressure of 25 MPa and 38 MPa were applied at a normal direction to the specimen surface. Temperatures of 900 °C, 1000 °C and 1100 °C were set below the recrystallization temperature of tungsten (1300–1500 °C) [15,38]. The heating rate was 100 °C/min and the duration time at the target temperature was 10 min. After heating, the specimens were cooled to room temperature in the furnace. PA in an argon atmosphere was carried out with a ThermecMaster equipped with an induction heating system. While heating, two graphite blocks gave uniform pressure on the top and bottom surfaces of the specimens normal to the sprayed area. Fig. 1 shows a schematic diagram of the treatment machine used for PECT. The as-sprayed specimen was positioned between two graphite electrodes in order to apply pressure and a pulsed electric current that passes through the specimen. Constant current densities were applied to the specimen to maintain object temperature: 10 A/mm^2 for 900 °C, 13 A/mm^2 for 1000 °C and 15 A/mm^2 for 1100 °C.

Every specimen including an as-sprayed specimen was cut using an Accustom-5 (Struess) for the cross-section observation. Specimens were observed using FE-SEM (Hitachi, SU70) and EBSD (EDAX, Hikari). Both SEM and EBSD images were taken at 1200 times magnification with a step size of 0.15 μm. The critical misorientation angle for grain definition was set at 15°. 'TSL OIM Analysis 6' was used for the EBSD analysis. Microhardness values of the specimens were measured by a micro-indenter (Nanovea) with a diamond Berkovich tip. The loading/unloading rate of the indenter was 1 N/min and the maximum load was 0.5 N. The duration time at a maximum load was 10 s. A minimum of 30 microhardness measurements were conducted for each specimen.

Table 1
Specific conditions of specimen fabrication.

Parameter	Condition
Arc current	700 A
Temperature control	Unsprayed bottom side: 10 °C
Blowing gas	45lmp (Ar), 5lmp (H_2)
Type of torch	Non-transferred DC plasma torch
Speed of provided W powder	5 lpm (40 g/min)
Substrate size	Graphite. Φ5 × 3 mm
Distance of spraying	110 mm
Thickness of the deposition	200 μm

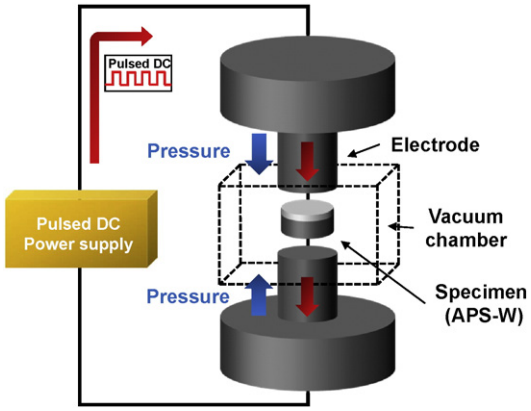

Fig. 1. Schematic diagram of the pulsed electric current treatment (PECT).

3. Results and discussion

Figs. 2(a) and (b) show cross sectional images of the as-sprayed specimen taken by the SEM and EBSD inverse pole figure (IPF) mapping in a sprayed direction (SD). In the SEM image, the accumulation of splats and their boundaries could be observed. Splats are lamellar layers formed by melted tungsten particles and the boundaries between each splat are called splat boundaries. Splat boundaries are made up of voids and oxides, which result in the degraded mechanical properties of APS-W. The EBSD IPF map provides the orientation information of each grain consisting of splats. Inside each splat, multiple grains were observed, which were irregularly distributed without preferred orientation. The black areas in the EBSD image indicate unindexed areas, which are mainly related with splat boundaries. Due to the drastic difference of temperature between the substrate and the molten splats, the grains were formed as a columnar structure that follows the temperature gradient.

As-sprayed specimens were post-treated by the PA and PECT in order to improve the mechanical properties of the tungsten layer by reducing the splat boundaries. Figs. 2(c) and (d) show the cross-sectional images of the PAed specimen and Figs. 2(e) and (f) show the cross-sectional images of the PECTed specimen taken by the SEM and EBSD IPF mapping in SD. Both images were under the condition of 25 MPa at 1100 °C. Through the SEM images of the PAed and PECTed specimens, it can be seen that the amount of splat boundaries decreased after the post-treatments. However the PECTed specimen showed a decreased amount of splat boundaries than the PAed specimen. The EBSD IPF map of the PAed and PECTed specimens show that there were no noticeable changes of texture orientation in the tungsten layer after the post-treatments. In Figs. 2(d) and (f), the amount of the unindexed area significantly decreased in the PECTed specimen in comparison to that in the as-sprayed and PAed specimens, which indicates a greater decrease of splat boundaries in PECTed specimen. The decrease of splat boundaries in each specimen was quantified by means of image processing of the SEM images. For the as-sprayed specimen, the area fraction of splat boundaries was about 10.0%. In the PECTed specimen, the fraction was decreased to about 2.8%, which was 3.0% lower than that in the PAed one, 5.8%. The SEM and EBSD observations demonstrate that atomic diffusion could be enhanced by an electric current, which agrees well with the reported results for aluminum and steel alloys [29–34].

Figs. 3(a) and (b) show the misorientation angle profile of the PAed and PECTed specimens under the condition of 25 MPa at 1100 °C. In the