Manufacture of Vacuum Plasma Spraying Tungsten with Homogenous Texture on Reduced Activation Ferritic Steel at about 873 K

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The key to improving the heat load of vacuum plasma sprayed tungsten coatings on low activation ferritic steel maintained at low temperatures is elimination of stratified low-density layers with many large pores, in which thermal cracks propagate preferentially. The low-density layers are formed owing to the deposition of large solidified tungsten particles, which remain mainly at the periphery of the spray stream. In this study, by shading this periphery, partially homogeneous tungsten coatings without large pores were successfully obtained. The coatings are expected to show good heat load, which is feasible for nuclear fusion applications.

Keywords: first wall, vacuum plasma spray, tungsten, F82H, EBSD, columnar grain

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1. Introduction

Recently, considerable attention has been paid to the use of tungsten coatings to protect plasma-facing surfaces of experimental fusion devices from very high flux heat loads. Among the various coating methods, the low pressure plasma spray (LPPS) technique is a potential protection method because it can rather easily coat thick films onto large areas. LPPS is often called the vacuum plasma spray (VPS) technique [1]; in this technique, powdered feedstock materials melted in a plasma jet are sprayed onto substrates in a controlled atmosphere at low pressure by scanning the plasma spray gun in two-dimensions.

Tungsten coatings for fusion devices require superior heat load and mechanical properties. It is well known that the quality of plasma-sprayed coatings depends strongly on many manufacturing conditions such as heating power, particle size of the powder, and substrate conditions. Tungsten coating on carbon materials such as graphite and CFC acts as an armor for divertors; for example, it was shown that VPS-tungsten coated at temperatures higher than 1273 K could endure a cyclic heat load of 10 MW/m^2 to 160 cycles [2]. Tungsten-coated isotropic graphite tiles have been tested in the Large Helical Device (LHD) as divertors, and no significant damage has been reported so far [3].

VPS-tungsten coatings have been trialed for improving the heat load of reduced activation ferritic steel (RAFS) [4–7], which is proposed as a structural material for a future demo fusion reactor. For this application, the substrate temperature is the most critical factor, as it is required to be kept lower than the ferritic-austenitic transformation temperature (about 973 K) during the VPS process; this is because its superior mechanical properties may be deteriorated by the phase transition. The current limit of the cyclic heat load of tungsten-coated RAFS under the serious temperature condition is 4.8 MW/m^2 to 100 cycles [8]. Although this material will be able to endure the heat load at the first wall, a higher performance is necessary in order to use it with confidence under unsteady high heat loads in the case of abnormal events. The present performance is too low for it to be utilized in the divertor region, where the heat flux is higher.

We have reported that alternate layers of high- and low-density stacks are present in the VPS-tungsten coat-

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Table 1 Conditions of VPS.

Conditions	Spray-1 (0 mm pitch)	Spray-2 (single pass)	Spray-3 (shading)
Substrate	F82H steel (Fe-8Cr-2W)	W	SS430
Surface treatment	blast treatment	blast treatment	blast treatment
Dimension	30 × 100 × 5 mm³	Φ30 × 5 mm³	30 × 100 × 5 mm³
Substrate temperature	895 K	857 K	894 K
Average size of W powders	37 μm	37 μm	37 μm (cut smaller than 25μm)
Number of pass	30 (one-dimension)	1 (two-dimensions)	20 (two-dimensions)
Thickness of VPS-W	0.45 mm (at *y*= +15 mm)	1.18 mm	0.59 mm
Spray distance	250 mm	250 mm	250 mm
Gun type	F4VB	F4VB	F4VB
Input power	42 kW	42 kW	42 kW

ing on RAFS around 873 K [8, 9]. The high-density stack consists of columnar crystal grains of several micrometers in length grown toward the surface and randomly oriented fine crystals, while the low-density stack is a mixture of large roundish grains having a size of ∼15 μm and fine crystal grains having a size of < 1 μm with larger pores. Thermal cracks parallel to the surface, which limit the cyclic heat load, propagate in the low-density layers under a strong heat load of more than 5.5 MW/m^2 [8, 9]. Moreover, the results of an impact test showed that the texture of columnar grains have good mechanical properties [10]. These results suggest that the heat load of VPS-tungsten coatings on RAFS at low temperatures can be improved by excluding the large grains and pores stacked in layers.

The present study focuses on clarifying the formation mechanism of the layer structure and optimizing the VPS to form homogeneous tungsten coatings of high density on the substrates at temperatures lower than 973 K.

2. Experimental Procedures

2.1 Vacuum plasma spraying of tungsten

Vacuum plasma spraying was performed at Tocalo Co. using an A-2000V low-pressure plasma spray system equipped with a plasma spray gun F4VB produced by Sulzer Metco Co. Powdered tungsten was injected together with a carrier gas into the plasma jet at a very high temperature through the feeding nozzle. The metal is melted in the plasma jet and propelled onto the workpiece surface. In the case of the F4VB type plasma spray gun used in the present study, the feeding nozzle is perpendicular to the plasma jet. Therefore, some injected particles have a chance to cross the plasma jet without sufficient melting and deposit as unmelted or resolidified particles. Many of them are probably spherical, and their sizes are close to those of the original powder particles. In practice, it was difficult to melt all tungsten feed particles completely because the melting point of tungsten is extremely high.

To understand the influence of the unmelted and resolidified particles on the texture of the coatings, two types of spray experiments (Spray-1 and Spray-2) were conducted at first. Then, to achieve homogeneous coatings of high density, a third experiment (Spray-3) was conducted under optimized conditions using the results of Spray-1 and Spray-2.

In this study, although three kinds of substrate materials with different material properties were used, it is believed that these differences do not significantly affect the ratio of the columnar and resolidified grains. The substrate material used in Spray-1 was F82H (Fe-9Cr-2W, 30 mm × 100 mm × 5 mm), which is an RAFS developed by JAEA as a structural material for a demo fusion reactor [11]. In Spray-2, since the control of the substrate temperature was difficult, powder metallurgy tungsten (ϕ = 30 mm × 5 mm) was used as the substrate. SS430 (Fe-18Cr, 50 mm × 50 mm × 3 mm), which is similar in structure to F82H was used as the substrate in Spray-3. In all cases, the substrate temperature was adjusted to around 873 K before the beginning of plasma spraying. The substrate temperatures during the VPS process were also maintained at around 873 K (Spray-1: 895 K, Spray-2: 857 K, Spray-3: 894 K). Tungsten powders having average sizes of 37 μm were used in Spray-1 and Spray-2, and powders smaller than 25 μm were removed from those used in Spray-3. Details of the spray conditions are listed in Table 1.

2.1.1 Spray-1 (zero-pitch scanning)

In conventional plasma spraying, the spray gun is repeatedly scanned in two dimensions, as shown in Fig. 1 (a). However, in Spray-1, the gun was scanned repeatedly only along the x-axis without shifting along the y-axis (zero-pitch scanning in y-direction) (see Fig. 1 (b)). Here the direction of ejection of the powder particles having an average size of 37 μm through the feeding nozzle and the point directly under the plasma jet are defined as $+y$ direction

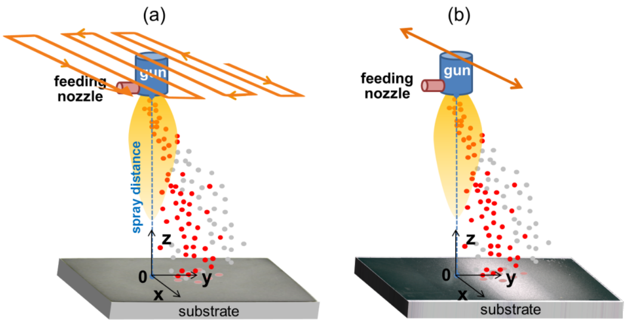

Fig. 1 (a) Conventional two-dimensional scanning (a) and (b) one-dimensional scanning along x-axis used in Spray-1.

and origin of the coordinate axes, respectively, as illustrated in the figure. Sprayed tungsten was stacked in the $+z$ direction. In this zero-pitch scanning spray, the same part in the spray stream always deposits on each position.

2.1.2 Spray-2 (single-pass scanning)

To acquire details on the internal structure formed in a single pass, a coating having a thickness of about 1.2 mm was formed by scanning the plasma spray gun only once in two-dimensions at an extremely low speed. Because the plasma gun was shifted from the $+y$ side to the $-y$ side, the periphery of the spray stream on the feeding-nozzle side (called near-side periphery, hereafter) was deposited first. Then, the central part and opposite periphery (far-side periphery) were deposited successively.

2.1.3 Spray-3 (shading)

To exclude tungsten particles at the periphery of the spray stream, especially the far-side, a shading plate was placed between the plasma gun and the substrate materials. Coatings were deposited onto SUS430 substrates at 894 K by the multi-scanning method. The plasma heating conditions were similar to those of the conventional plasma spraying method.

2.2 Evaluation of plasma-sprayed coatings

The surface morphology and cross-sectional morphologies of the coatings were examined using an optical microscope and a scanning electron microscope (SEM). In the case of SEM, electron backscatter diffraction was used to evaluate the crystal grain size and shapes in each grain. The aspect ratio of each crystal grain (ratio of large diameter (d_L) to small diameter (d_S), d_L/d_S) was evaluated using orientation imaging microscopy. In the preset study, grains with aspect ratios greater than 2.5 were defined as columnar grains for convenience.

3. Experimental Results and Discussions

3.1 Spray-1 (zero-pitch scanning)

In case of zero-pitch scanning, it was observed that tungsten was deposited on the area from $y = -5$ to $+50$ mm. This indicates that the spray stream was expanding toward the direction of powder feeding. Although the texture did not change significantly in the z-direction, as expected, it depends strongly on the positions along the y-axis, as shown in Fig. 2. As shown in Fig. 3, the thickness of the coating per x–y scan, fraction of columnar crystal grains, and that of very large solidified grains ($> 20\,\mu m^2$) are plotted as functions of distance. It is noticeable that the texture in the area between $y = 10$ and 20 mm, where the deposition rate is the highest, is uniform and dense. The fraction of columnar grains reaches 45% and that of large grains is only 0–2%. As shown in the magnified images at $y = +15$ mm (Fig. 4), the deposited particles become flat, and columnar grains grow through the deposited particles. The adhesion of newly deposited particles with the solid surface seems to be strong. The number of pores, which were mainly formed between the sprayed particles, is very small, and large pores exceeding a few micrometers were seldom observed. These results indicate that most of the tungsten particles deposited in this region have been melted very well. Judging their extremely homogeneous texture, it can be expected that the coatings in this region exhibit excellent heat load resistance and mechanical properties.

On the other hand, solidified large particles, such A and B in Fig. 2, become apparent at the near-side periphery around $y = 0$ mm and the far-side periphery beyond $y = 25$ mm. Owing to the large pores, which are often formed around large grains, the density of these particles decreases. The size and shape of the large particles suggest that they are unmelted and/or resolidified particles. Hereafter, they are called solidified particles (or grains) for convenience.

The experimental results mentioned previously indicate that tungsten particles at the center of the spray stream melted well, while solidified particles were deposited along with the melted ones at the periphery of the stream. Especially, a large number of solidified particles were distributed widely at the far-side periphery.

3.2 Spray-2 (single-pass scanning)

Figure 5 shows the typical morphology of the coating formed via spraying by single-pass scanning. The SEM micrograph (a) shows the cross section of the entire coating; (b), (c), and (d) are the magnified SEM micrographs of the top, middle, and bottom portions, respectively, while (e), (f), and (g) are the corresponding inverse pole figures (IPFs). Fractions of columnar grains, fine grains and large grains at the top (T), in the middle (M), and at the bottom (B), respectively, are plotted in Fig. 6. It is clear that the

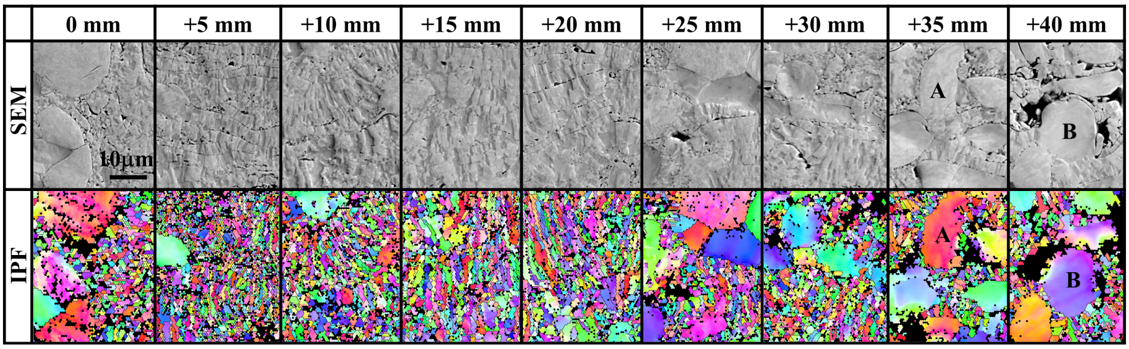

Fig. 2 Cross-sectional micrographs of SEM and IPF of Spray-1 taken at 7 positions from y = 0 to 40 mm.

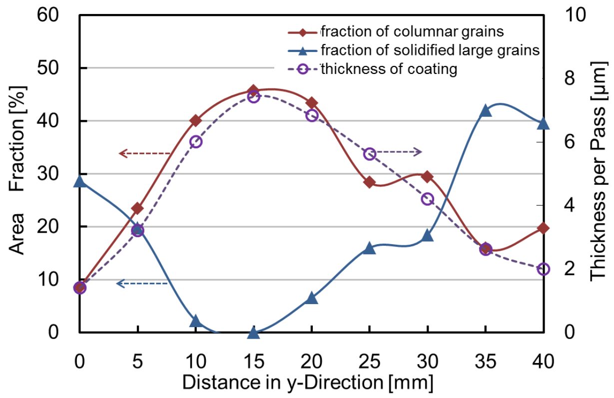

Fig. 3 Fractions of columnar and solidified large grains plotted against the distance in the y-direction measured from the point directly under the plasma gun with the thickness of the coating per scanning pass in Spray-1.

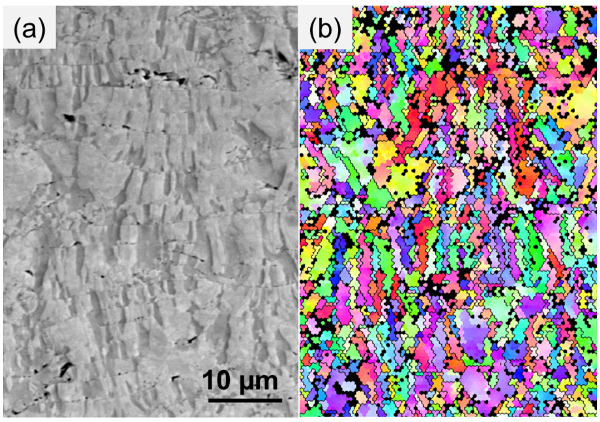

Fig. 4 (a) SEM micrograph and (b) IPF at y = +15 mm of Spray-1. The coating consists of columnar and very fine grains only.

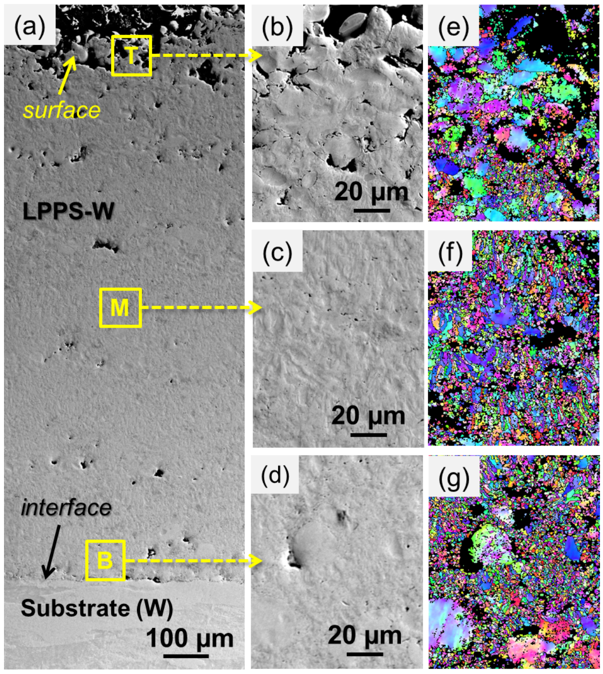

Fig. 5 (a) Texture of coating formed by the single-pass spraying (Spray-2); (b), (c), and (d) are magnified SEM micrographs at the top (T), middle (M), and bottom (B) positions, where (e), (f), and (g) are the corresponding IPFs.

morphology in the middle part, where well-melted particles were deposited, mainly shows fine grains (49.4%) and columnar grains (48.3%). The fraction of the large grains is only 2.3%. In contrast, the large grains and pores having sizes of more than 10 μm are observed at the top and bottom. Especially, the top surface is fully covered by large solidified particles and pores because the spray stream at the far-side periphery containing a large number of solidified particles was deposited last. These results imply that the density of the coating formed in a single pass of two-dimensional scanning continuously fluctuates. Therefore, in the case of spraying by multi-scanning, the density of the coating fluctuates periodically with pass repetitions.

3.3 Spray-3 (shading)

According to Spray-1 and Spray-2, low-density layers, which are unfavorable as plasma facing materials for protection against high-flux heat loads, are formed owing to the deposition of large solidified particles distributed

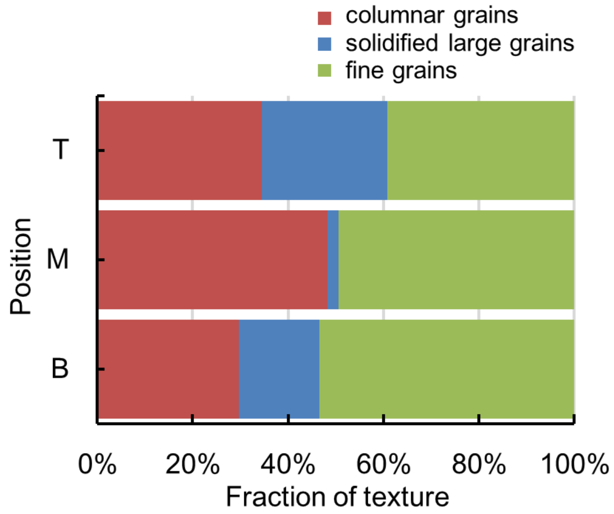

Fig. 6 Fraction of columnar, solidified, and fine grains at T, M, and B of the coating shown in Fig. 5.

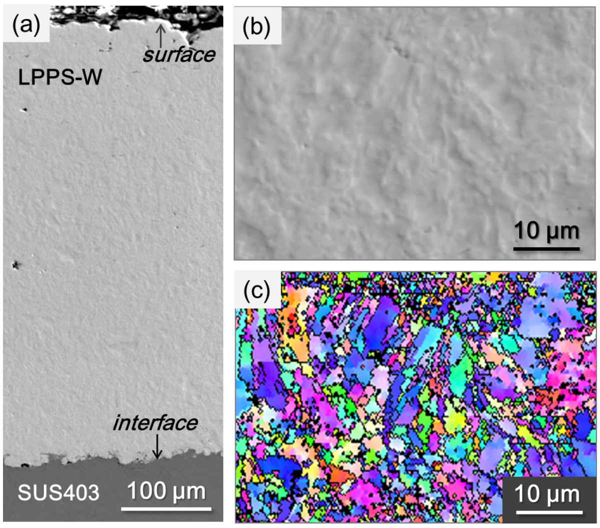

Fig. 7 SEM micrograph of the cross section of the coating in Spray-3 is shown in (a). Magnified SEM micrograph and IPF are shown in (b) and (c), respectively.

mainly at the periphery of the spray stream. Therefore, in Spray-3, multi-scan plasma spraying was performed with the part of the spray stream beyond $y = 20$ mm excluded by placing a shading plate between the gun and the substrate.

The low-magnification SEM micrograph in Fig. 7 (a) shows the cross section of the 0.59-mm-thick coating formed on the SUS430 substrate at 894 K. The texture is quite homogeneous from the interface to the top surface. A magnified SEM micrograph and the corresponding IPF from the middle region show that the texture consists of columnar and fine grains (Figs. 7 (b) and (c)). Large grains and pores were seldom observed, as expected. The density seems to be significantly high throughout the coating.

In Fig. 8, the surface morphology of the coating

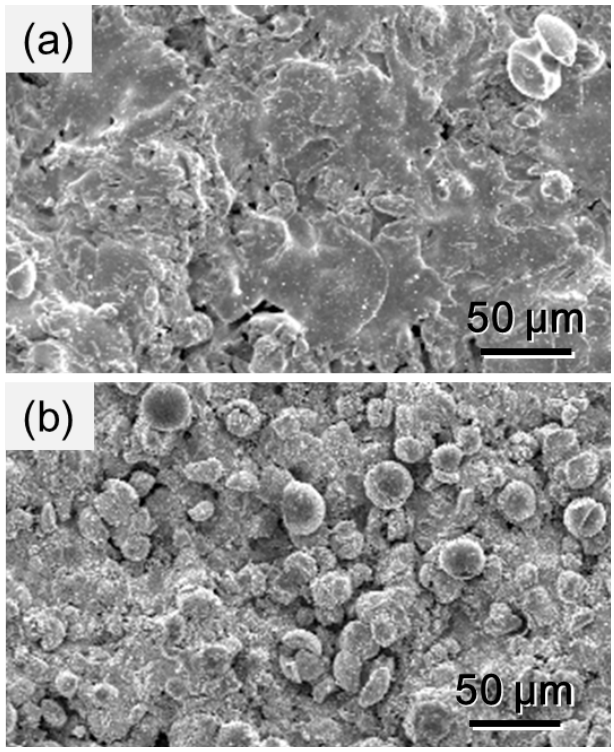

Fig. 8 Surface morphology of coatings formed by Spray-3 (shading) and that formed by conventional spraying.

formed by Spray-3 is compared with that of the coating formed by conventional spraying. It is clear that the surface of the former is mainly covered by flattened particles, which were resolidified after stacking on the substrate, while that of the latter is mainly covered by large spherical particles, which were deposited in the solidified state. This indicates that the melted particles were selected well, as expected.

It has been shown that a shading plate placed between the plasma gun and the substrate is very effective in forming a homogenous dense tungsten coating composed of columnar and fine grains on the substrate, even at 873 K. This tungsten coating is expected to show excellent performance under high heat load conditions. The estimation of heat load and fundamental properties such as thermal conductivity and density will be performed in future studies.

The shading method used in the present study is rather simple, but the efficiency of the coating technique decreases if one aims to prepare higher-quality coatings. A drastic measure could be improvement of the gun. If the tungsten particles are injected parallel to the plasma jet, the number of solidified particles is reduced considerably. The investigation of this large-scale conversion of the gun is necessary when mass production of coating material is required.

4. Summary

This paper reported that the heat load of VPS-tungsten formed on RAFS at low temperatures was low because the coating has a structure of alternating high- and low-density layers, in which thermal cracks propagate preferentially. To improve the cyclic heat load of VPS-W/RAFS to make it feasible as a plasma-facing material, the reasons for the weakness of the low-density layer have been investigated, and an improved spray method was developed to form homogeneous tungsten coatings with high densities.

Owing to the structure of the spray feeding nozzle for powdered tungsten and very high melting temperature of this metal, solidified tungsten particles remain at the periphery of the spray stream. The large solidified particles may prevent smooth stacking of the sprayed tungsten that follows, leading to the formation of large pores.

Therefore, the regions where the periphery part of the jet stream was deposited have an unfavorable structure with many large grains and pores. Because the plasma gun is scanned in two dimensions repeatedly, the unfavorable low-density layer and the favorable high-density layer (formed by well-melted tungsten particles in the center of the spray stream) are stacked alternately.

By shading the periphery part of the spray stream, homogeneous tungsten coatings without large grains and pores were successfully manufactured. It is expected that the VPS-W/RAFS manufactured using this method shows a high heat load, which is feasible for fusion reactors.

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