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Citation	Transactions of JWRI. 2008, 37(1), p. 63-67
Version Type	VoR
URL	https://doi.org/10.18910/4497
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Tungsten Coating for Thermal Fusion Material Produced by Gas Tunnel Type Plasma Spraying[†]

KOBAYASHI Akira*

Abstract

Tungsten (W) is a material which has the highest melting point of 3422 °C among metals. Therefore when deposited as a coating it can protect the substrate surface from high heat flux. The W composite will be a superior candidate for thermal fusion materials. In this study, tungsten sprayed coatings were produced on stainless steel substrates by gas tunnel type plasma spraying at a short spraying distance (40mm). The Vickers micro-hardness of the W coating was measured as the mechanical property. The tungsten coating had a hardness of Hv=260-300 along its cross section, which is a little lower than the hardness for bulk tungsten. Regarding the microstructure the coating contained some pores. The results of X ray Diffraction show that the coating consists of pure tungsten, without oxidation.

KEY WORDS: (Tungsten coatings) (Fusion materials) (Gas tunnel type plasma spraying) (Hardness test) (XRD)

1. Introduction

Tungsten (W) is a special material, which has a high melting point, whose melting temperature is the highest among the metals ($T_m = 3422^\circ\text{C}$). It is therefore desirable as a refractory material for high temperature applications. Furthermore, tungsten has the lowest sputtering yields among metals and is therefore being developed for fusion reactor applications such as the first wall¹⁾, which requires minimum sputtering of foreign elements into the plasma. The W composite will be a superior candidate of a material for thermal fusion materials.

For practical application of a tungsten target of an X-ray machine, using a coating would help solve the problem of heavy weight of W. Active research is ongoing to replace the W-target with a low weight silicon carbide (SiC) substrate coated with tungsten. Although chemical vapor deposition (CVD) has been used to deposit tungsten onto the SiC substrate, CVD is a slow and relatively costly method, and the resultant coating may contain contaminated species. Plasma spraying of tungsten is a more economical and convenient method for coating preparation. Another example application is the plasma spray tungsten coated SiC- fiber (foam) for high heat-resistance. It contributes to the enhancement of the SiC- fiber heat-resistance that tungsten has almost the same expansion coefficient as SiC, but exhibits higher melting point than SiC, whose melting point is

2500-2700°C. This composite material is expected to be a good material for space propulsion and/or thermal fusion material.

Regarding the plasma sprayed tungsten coating, the high melting point of tungsten requires the plasma spray equipment to run at high power. High-power gas tunnel type plasma spraying has been developed²⁾ and is used to achieve efficient melting and coating deposition of tungsten. Compared with conventional plasma spray equipment, the high-power gas tunnel type plasma spraying can produce denser coatings with 20-30% higher hardness, and, several ceramic coatings deposited using the gas tunnel type plasma spraying have been investigated and reported previously³⁻⁶⁾. For example, zirconia coatings produced at short spraying distances (L), have a surface layer with higher hardness than the inner layers, which indicates the graded functionality of the coatings^{8,9)}. The Vickers micro-hardness of the zirconia coatings was about $Hv=1200$ ⁷⁾ at a power of $P=33$ kW and spraying distance of $L=30$ mm. The mechanical properties and the bonding strength between the zirconia coatings and substrates have also shown that use of the gas tunnel type plasma spraying results in superior coatings¹⁰⁾.

Ceramic coatings such as zirconia coatings are used for high temperature protection of metallic structures because of their high temperature resistance. Zirconia

[†] Received on July 11, 2008

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Transactions of JWRI is published by Joining and Welding Research Institute, Osaka University, Ibaraki, Osaka 567-0047, Japan

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coating has been used as TBC of the hot sections of gas turbine engines and the high temperature parts of detonation furnaces. In order to enhance the quality of the TBC, plasma spraying has been contributing to combine the high heat capability of tungsten and the low thermal conductivity of ZrO_2 even for developing novel heat resistant TBCs.

In this study, tungsten sprayed coatings were formed on stainless steel substrates using the gas tunnel type plasma spraying at a short spraying distance of 40 mm, in order to develop a high performance material for thermal fusion. The Vickers micro-hardness of the tungsten coatings was measured by a micro hardness tester. Also the microstructure of the coatings was viewed by an Optical Microscope (OM) and a Scanning Electron Microscope (SEM). The crystalline structure of the coatings was investigated by X-ray Diffraction (XRD). Variations of tungsten coatings' properties were also examined with different coating thickness.

2. Experimental

The gas tunnel type plasma spray torch developed by the author²⁻³⁾ is shown schematically in **Fig. 1**. The experimental methods for production of the high hardness ceramic coatings by means of the gas tunnel type plasma spraying have been described in the previous publications⁴⁻⁶⁾. For the current studies, a gas divertor nozzle diameter of 20mm was chosen.

The overall experimental conditions for the plasma spraying of tungsten are shown in **Table 1**. The input

power to the plasma torch was about $P=13.5$ and 22.5 kW. The power input to the pilot plasma jet was supplied by the power supply PS-1 and was turned off after starting of the gas tunnel type plasma jet. A short spraying distance of $L=40$ mm was chosen for all tungsten plasma spraying deposition processes. Argon was used as the working gas, and its flow rate was $Q=170$ l/min.

The powder feed rate of tungsten was about $w=16-24$ g/min and the gas flow rate of carrier gas was 10 l/min. The substrate was traversed 0-12 times during the spraying.

The chemical composition and particle size of the tungsten powder used are also given in **Table 2**. The tungsten powder was 99.9% in purity and the average particle size was 12 μ m. **Figure 2** shows the microphotographs of tungsten powder. The profile of the powder was not spherical and the size is average 12 μ m. **Figure 3** shows the result of XRD measurement for the powder, which shows the powder was pure tungsten.

The AISI304 stainless steel ($3 \times 50 \times 50$ mm³) substrate used was sand-blasted before spraying deposition.

The Vickers micro-hardness of the sprayed tungsten coating was measured in cross sectional regions, where is free from porosity. The hardness test loading weight was 100 g and the loading time was 25 s. The Vickers micro-hardness was calculated as a mean value of 10 measurements.

The cross section of the tungsten coatings was observed for surface morphology at magnifications of 200 or 400 times with an optical microscope. Also, an SEM was used to observe clearly the structure. X-ray diffraction (XRD) with a θ -2 θ geometry was conducted for the crystal structure of the coatings utilizing a Co target and tube voltage of 30 kV and current of 14 mA.

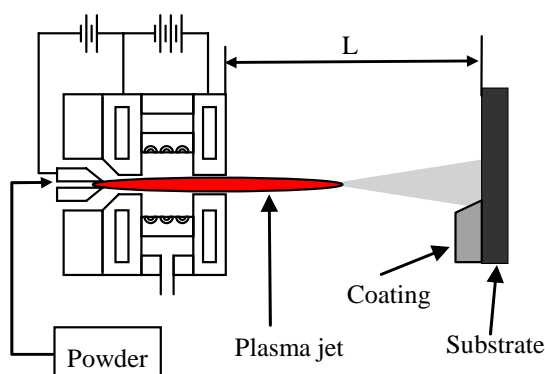


Fig.1 Gas tunnel type plasma spraying used in this study (L =spraying distance).

Table 1 Spraying Condition.

Arc current	450A
Voltage	-50V
Spraying distance	40 mm
Working gas Ar flow rate	170 l/min
Feed gas flow rate	10 l/min
Powder feed rate	16-24 g/min
Traverse number	0, 12 times
Spraying time	20 s, 24 s

Table 2 The chemical composition and particle size of the tungsten powder used.

Material:	Tungsten (W)
Melting point:	$T_m = 3422^\circ\text{C}$
Purity:	99.9%
Particle size:	12 μ m (average)

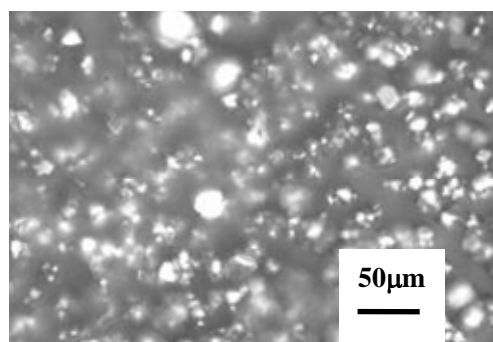


Fig.2 Microphoto of W powder used in this study.

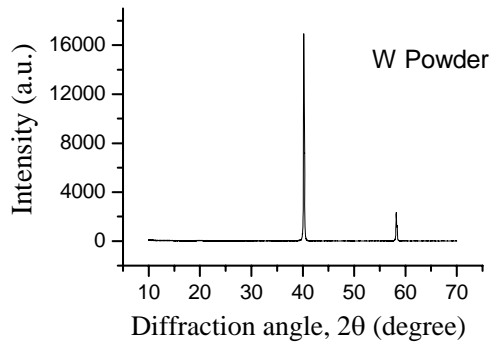


Fig.3 XRD pattern of the tungsten powder used.

3. Results

3.1 Microstructure of tungsten coating

Figure 4 shows the surface photographs of a tungsten thick ($\sim 180\mu\text{m}$) coating. The photographs were taken by an optical microscope from the central area of the deposition area, and the cutting line for coating cross-section observation is shown at their upper part. The color of the thicker sample was grey coating. Plasma sprayed pure tungsten coatings are black in color. But generally yellow appears, which indicates the presence of tungsten oxides.

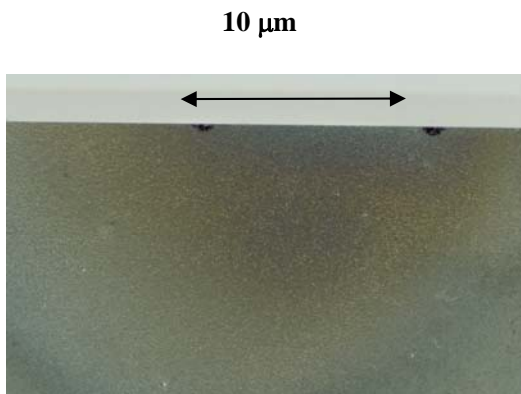


Fig.4 Surface photograph of the coating sprayed at $L=40\text{mm}$ with $P=13.5\text{ kW}$, for thick coating.

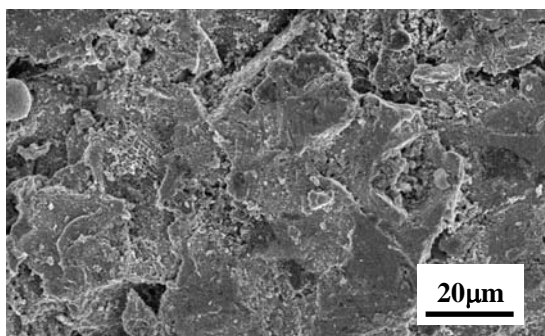


Fig.5 Surface photograph of the coating sprayed at $L=40\text{mm}$ with $P=25\text{ kW}$, taken by SEM.

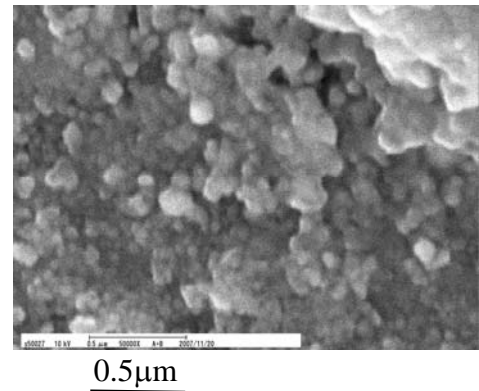


Fig.6 Surface of the coating appeared with nano particles taken by high magnification SEM. 27

The surface photograph of the tungsten coating sprayed at $L=40\text{mm}$ with $P=25\text{ kW}$, taken by SEM, is shown in **Fig.5**. This photograph shows that tungsten powder was sufficiently molten. The length of each splat is more than $20\mu\text{m}$ in this case. And there were nano size particles at some points on the coating surface (**Fig.6**). This means that there are some possibilities for the controllability of spraying condition in order to make nano size surfaces of W coatings in the future.

The cross section images of tungsten coating were taken by an optical microscope. **Figure 7** show the cross sectional image of the same tungsten coating samples as shown in Fig.4, but the microscope spans only within a space of 1cm in the center of the deposition area.

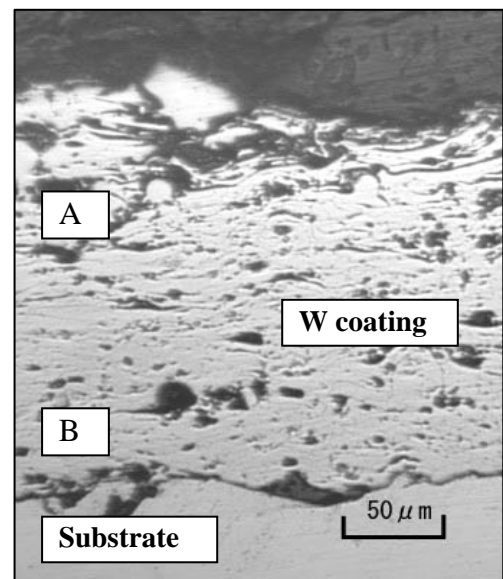


Fig.7 Cross-sectional optical microscope image of coating sprayed at $L=40\text{mm}$ with $P = 13.5\text{ kW}$, for thicker coating. A: Surface region, B: Bond interface.

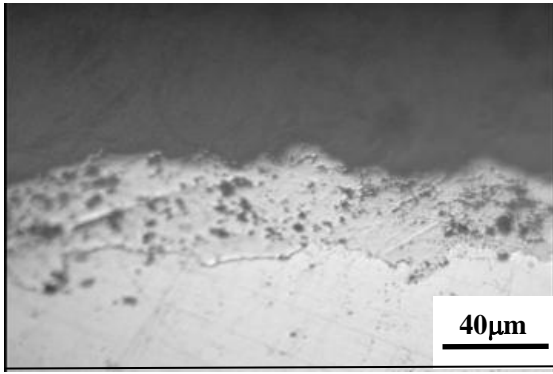


Fig.8 Cross-sectional optical microscope image of W coating sprayed at $L=40\text{mm}$ with $P = 25\text{ kW}$.

The region near to the the surface side of thick coating ($\sim 180\mu\text{m}$), i.e. [Region A], was re-melted and a dense tungsten layer was formed. Meanwhile, the cross section adjunct to the substrate [Region B] shows that the particles were deposited separately and were condensed together during the initial stage of deposition. It is the coating heat transfer features, changing gradually along with the undergoing deposition, which results in graded changes in the morphology and density. It is therefore believed that sufficient plasma torch heating occurred during the thinner layer deposition for re-melting to occur.

In the case of a traversed coating, a thinner ($\sim 40\mu\text{m}$) coating was obtained as shown in **Fig.8**. The spraying time for this thin coating was shorter than that for the thicker coating in Fig.3. It shows a more uniform highly dense or re-melted structure. For both the coatings, the number of pores is substantially lower than that of zirconia coatings deposited under given conditions

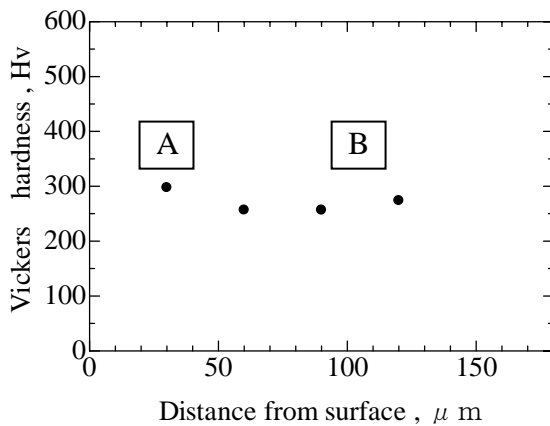


Fig.9 Distribution of Vickers micro-hardness along the cross section of the thicker coating, A: Surface region, B: Bond interface.

3.2 Vickers hardness of tungsten coating

The Vickers micro-hardness cross sectional distribution is measured from the coating surface to the substrate-coating interface. The result for the thicker coating was shown in **Fig.9**.

The hardness distribution gradually decreased from the surface to the interface. Near the surface [Region A], the Vickers micro-hardness was around $H_v = 300$, while near the substrate [Region B], it dropped to about $H_v = 270$. It is hinted that the hardness distribution is in coincidence with the variation of the coating micro structure, shown in the cross section of both [Region A] and [Region B] of Figure 7. The structure of [Region A] was much denser than that of [Region B], in which the powders were deposited separately.

The influence of thickness of the W coating on the Vickers micro-hardness was measured along cross section of each coating. The results of the Vickers micro-hardness tests are shown in **Fig.10**. The Vickers micro-hardness of the thicker coatings were $H_v = 280$, and thinner $H_v = 260$ respectively, which represents the average value for plasma sprayed tungsten coating. However, the tungsten coating Vickers micro-hardness was lower than that of pure tungsten, which is about $H_v = 350$, probably because of the pores in the coatings.

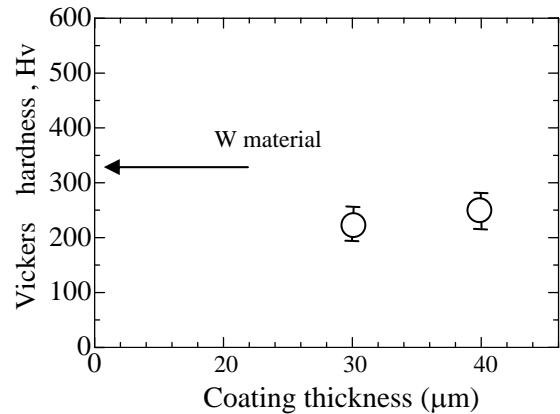


Fig.10 Comparison of Vickers micro-hardness at different coating thickness.

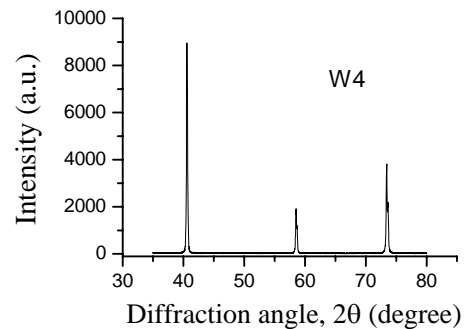


Fig.11 X-Ray Diffraction spectrum of the tungsten coating surface.

3.3 Crystal structure of tungsten coating

XRD measurement of the coating (40 μ m) is shown in Fig.11, which reveals several strong tungsten peaks and indicates the presence of pure tungsten phase. No tungsten oxide was observed by these XRD spectra obtained. The absence of tungsten oxide peaks shows that only minimal oxidation occurred during the deposition processes.

Generally, to avoid oxidation, tungsten or any other refractory metals require spraying under controlled atmospheric conditions, such as an Argon back-filled chamber or use of vacuum plasma spray chambers. It is a significant finding that the high power plasma torch used here must have supplied sufficient argon gas flow to keep the newly deposited tungsten under a shroud of inert gas, thus allowing it to cool quickly enough to avoid oxidation.

4. Conclusion

Pure tungsten coatings were sprayed onto stainless steel substrates using the gas tunnel type plasma spraying method. The following results were obtained.

- (1) Thick (~180 μ m) tungsten coating could be coated onto stainless substrates at short spraying distances of less than 40 mm, when the plasma torch was operating at $P=14$ kW. On the other hand, a thinner (~40 μ m) tungsten coating was also formed by traversing the substrate at about 25 kW.
- (2) The prepared tungsten coatings have a Vickers micro-hardness of $H_v = 260-300$. The Vickers micro-hardness of the thicker coating decreased from $H_v=300$ to 260 from the surface to the interface, which was a little lower than the hardness of pure tungsten of $H_v = 350$.
- (3) Regarding the microstructure of the coatings, a small number of pores were detected by optical microscope. The thinner the deposition, the less porosity, which might be due to substantial re-melting during the deposition.
- (4) The result from XRD shows that the coating consists of pure metal tungsten. The high power plasma torch

used must have supplied sufficient argon gas flow to keep the newly deposited tungsten under a shroud of inert gas, thus to prevent it from oxidation.

Acknowledgement

The authors would like express thanks to Mr. N. Ishibashi for the assistant in data arrangements.

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