

# Research and development of plasma sprayed tungsten coating on graphite and copper substrates

Xiang Liu <sup>a,\*</sup>, Fu Zhang <sup>a</sup>, Shunyan Tao <sup>b</sup>, Yunzhen Cao <sup>b</sup>,  
Zengyu Xu <sup>a</sup>, Yong Liu <sup>a</sup>, N. Noda <sup>c</sup>

<sup>a</sup> Southwestern Institute of Physics, P.O. Box 432, Chengdu 610041, Sichuan, PR China

<sup>b</sup> Shanghai Institute of Ceramics, Chinese Academy of Science, 1295 Dingxi Road, Shanghai 200050, PR China

<sup>c</sup> National Institute for Fusion Science, Toki, Gifu 509-5292, Japan

## Abstract

Vacuum plasma sprayed tungsten coating on graphite and copper substrates has been prepared. VPS-W coated graphite has multilayered silicon and tungsten interface pre-deposited by physical vapor deposition (PVD) and VPS-W coated copper has graded transition interlayer. VPS-W coating was characterized, and then the high heat flux properties of the coating were examined. Experimental results indicated that both VPS-W coated graphite and VPS-W coated copper could endure 1000 cycles without visible failure under a heat flux of approximately 5 MW/m<sup>2</sup> absorbed power density and 5 s pulse duration. A comparison between the present VPS-W coated graphite and VPS-W coated carbon fiber composite (CX-2002U) with Re interface made by Plansee Aktiengesellschaft was carried out. Results show that both Re and Si are suitable as intermediate layer for tungsten coating on carbon substrates.

© 2007 Elsevier B.V. All rights reserved.

PACS: 28.52.Fa; 52.40.Hf

Keywords: Plasma facing components; Heat load; Tungsten coating

## 1. Introduction

Vacuum plasma spray (VPS) is one of the candidate fabrication techniques of the first wall and divertor modules of ITER, for example, plasma sprayed tungsten on Cu heat sink for the divertor modules at the baffle area and plasma sprayed beryllium as the armour material of first wall blankets in

the main chamber. Tungsten coating as plasma facing materials has been successfully performed in ASDEX-Upgrade and W surface is about 24.8 m<sup>2</sup> in 2003/2004 campaign, representing 65% coverage area of all plasma facing surface [1,2]. A good compatibility of tungsten with fusion plasma has been identified [3]. VPS-W coated carbon materials was widely applied at the present stage in order to be compatible with the support structure of current fusion devices, in which rhenium as intermediate layer is a well-established technique, for example, VPS-W coated fine grain graphite and carbon fiber

\* Corresponding author. Fax: +86 28 82850956, +86 28 82850300.

E-mail address: [xliu@swip.ac.cn](mailto:xliu@swip.ac.cn) (X. Liu).

composite [4,5] made by Plansee Aktiengesellschaft. As for VPS-W/Cu coating, thick coating of about 5 mm has been developed by ENEA (Russian) [6] and JAERI (Japan) [7] for divertor modules of ITER, which can sustain 1000 cycles at a heat flux of 5 MW/m<sup>2</sup> without damage.

Recently, plasma discharges with high plasma density and divertor configuration have been performed in HL-2A tokamak machine [8], and the modification of HL-2A for open divertor was being programmed. Tungsten as plasma facing material will be performed in the modified HL-2A. For this purpose tungsten alloy and tungsten coating have been developed. In this paper, we will concentrate on the research and development of VPS-W coating. First, VPS-W coated pure graphite with multilayered silicon and tungsten interface pre-deposited by physical vapor deposition (PVD) and VPS-W coated copper with graded transition interface were prepared, and then the coating was characterized. After that their high heat flux resistance capabilities were evaluated by screening and cycling tests in an electron beam facility. In the present work silicon is selected as the substitute for rhenium, and multilayered Si, W structure pre-deposited by PVD as interface of W/C coating is a new attempt. Therefore a comparison between our VPS-W coated pure graphite with Si intermediate layer and VPS-W coated CX-2002U carbon fiber composite with Re interlayer made by Plansee Aktiengesellschaft was also conducted.

## 2. Experiments and results

### 2.1. Characterization of VPS-W coating

In our previous research, high heat flux properties of VPS-W coated CX-2002U carbon fiber com-

posite prepared by Plansee Aktiengesellschaft were measured and a good thermal load resistance capability has been confirmed [9,10]. The thickness of this coating is 0.5 mm and the density is 85% of the theoretical value. Its thermal conductivity is about 70 W/m K at room temperature (RT) [11]. In the present work, VPS-W coated high pure graphite and copper was developed. VPS-W coating has thickness of 0.3 mm and porosity of about 6%. Bonding strength between tungsten coating and substrates is larger than 40 MPa. Fig. 1 shows the morphologies of VPS-W/C and VPS-W/Cu. As shown in Fig. 1, a multilayered interface consisting of three layers of PVD-W and three layers of PVD-Si was used for VPS-W coated graphite, every layer is 1–2 μm in thickness and total thickness of the interface is about 12 μm. A graded transition interface was used for VPS-W/Cu coating, and the total thickness of this transition layer is about 150 μm, which can also be observed in Fig. 1.

The thermal conductivity of VPS-W coating was measured by laser flash method and the relationship of thermal conductivity with temperature was shown in Fig. 2. The thermal conductivities of pure tungsten and VPS-W coating on CX-2002U CFC substrate were also shown in this figure for comparison. It can be seen that the thermal conductivity of the present tungsten coating is about half of the pure tungsten made by powder metallurgy (PM), and a little higher than that of the tungsten coating made by Plansee Aktiengesellschaft.

### 2.2. High heat flux tests

High heat flux tests of VPS-W coating were performed in an electron beam facility, in which the electron beam has Gaussian-like energy distribution.

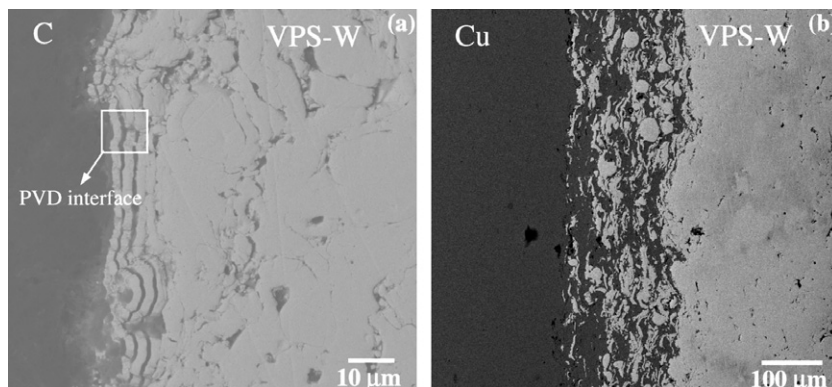


Fig. 1. Cross-section morphologies of VPS-W/C (a) and VPS-W/Cu (b).

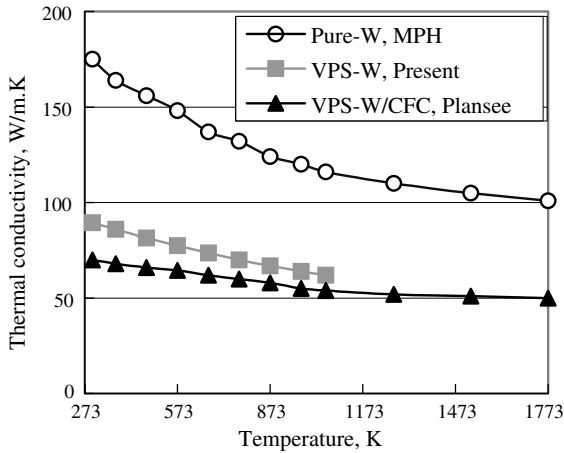


Fig. 2. Thermal conductivities of pure tungsten and VPS-tungsten coating versus temperature.

The samples with size of  $10 \times 10 \times 5 \text{ mm}^3$  were fixed on a copper plate from both side faces of the sample by copper strips, and the copper sample holder cooled by water. In the present experiment the electron beam was defocused to irradiate almost whole top surface of the sample. The surface temperature of VPS-W coating was measured by an optical pyrometer with temperature range of 300–3000 °C.

Before heat load experiments, the optical pyrometer was calibrated by thermocouples and the absorbed coefficient of energy for tungsten coating was measured. An absorbed coefficient of energy of about 57% was obtained. Which is a little larger than the value obtained by Merola for PS-W coating [12]. The reason might be owing to different roughness of PS-W coating and surface compositions. Since the plasma discharge duration is only several seconds in HL-2A tokamak, an experimental condition of 5 s pulse length and 30–40 s intervals (this interval is adequate for sample cooling down) was adopted in the present simulation to examine the heat load limit of the tungsten coating and its suitability for application as plasma facing materials or components in HL-2A. Firstly, screening tests of the coating were carried out, namely increasing the power densities step by step with a step of  $0.7 \text{ MW/m}^2$  until failure of the coating, for example, cracking, local de-bonding or melting, was observed. In screening tests only 3 shots were used for every power density. Results indicated that the heat load limitation was 13, 12 and  $10 \text{ MW/m}^2$ , respectively for VPS-W/CFC, VPS-W/C and VPS-W/Cu. Fig. 3 shows the maximal surface temperature versus absorbed heat flux. At  $4.8 \text{ MW/m}^2$

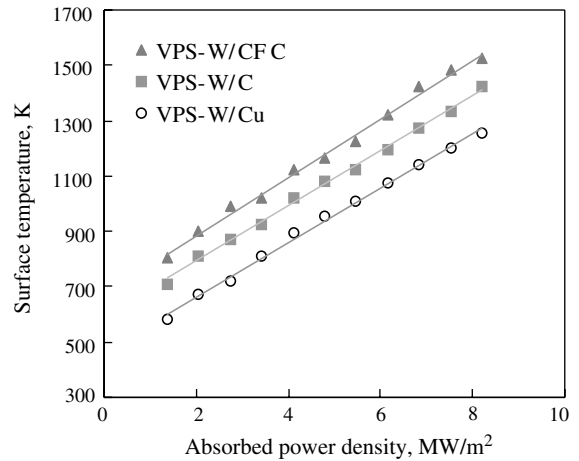


Fig. 3. Surface temperature increase of the coated samples as a function of the absorbed heat flux.

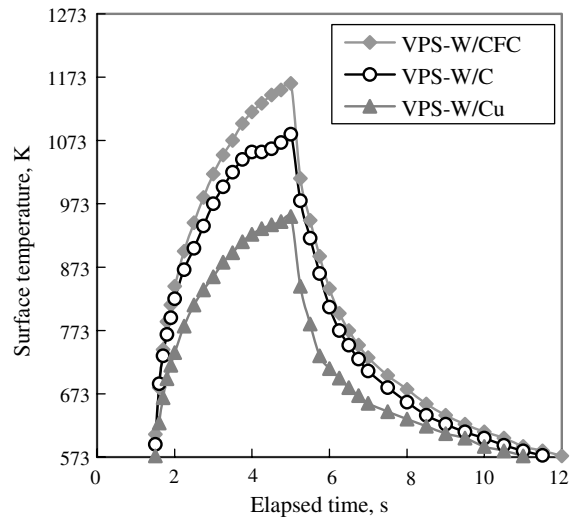


Fig. 4. Time evolution of surface temperature of VPS-W coating.

absorbed power density the thermal fatigue test of VPS-W coating was conducted and the surface temperature response of three kinds of coated samples was shown in Fig. 4. VPS-W/CFC has the highest surface temperature among the three tested samples, which should be due to relatively lower thermal conductivity and larger thickness of the coating than the other ones. The main physical properties of tested samples and the results of heat load tests were concluded in Table 1.

### 3. Discussion

Generally the main factors influencing the thermal conductivity of PS-coating are the density

Table 1  
Summary of the properties of tested coatings and heat load test results

| Materials | Description                   | Thermal conductivity of coating/substrate (W/m K, RT) | Absorbed power density/cycles (MW/m <sup>2</sup> ) | Experimental results                                       |
|-----------|-------------------------------|---|--|--|
| VPS-W/C   | Multi-layered W, Si interface | 90/100  | 5–12/3 for every step <sup>b</sup><br>4.8/1000     | Local de-bonding and/or melting<br>Without visible failure |
| VPS-W/Cu  | Graded transition interface   | 90/380  | 5–10/3 for every step <sup>b</sup><br>4.8/1000     | Local cracking and melting<br>Without visible failure      |
| VPS-W/CFC | Multi-layered W, Re interface | 70/305 <sup>a</sup>                                   | 5–13/3 for every step <sup>b</sup>                 | Local cracking and melting                                 |

<sup>a</sup> Here the thermal conductivity of CX-2002U is an average of the thermal conductivities at *x*, *y* and *z* directions.

<sup>b</sup> Screening tests, and the magnitude of step is 0.7 MW/m<sup>2</sup>.

(porosity) and purity of coating. Therefore VPS-coating has higher thermal conductivity than inert gas plasma sprayed coating. It is reasonable that presented W coating has higher thermal conductivity than the one made by Plansee Aktiengesellschaft as shown in Fig. 2 because the former has higher density than the latter. The reason why we select PVD-Si as intermediate layer for VPS-W coated graphite is that Si has relatively high thermal conductivity (about 130 W/m K at RT) and its thermal expansion is close to the graphite substrate. Furthermore, even though Si reacts with C (substrate materials) and SiC is formed at high temperature, the coefficient of thermal expansion of SiC is very close to that of tungsten, and a stable SiC phase might act as a diffusion barrier of carbon elements. VPS-Si has ever been used as the interlayer of PS-W/C coating in Forschungszentrum Juelich [13], however the result seems not successful. Therefore a detailed investigation on the evolution of the Si interface at high temperature is strongly required.

The thermal load limitation of the tested coating was evaluated by screening tests and the results indicate that VPS-W/CFC is the best and VPS-W/Cu is the worst. However the thermal load limitations of all the coating are beyond 10 MW/m<sup>2</sup> in the present experimental conditions as shown in Table 1. As to HL-2A, the maximum wall load is only several tenth of MW/m<sup>2</sup> at the main chamber (for example, the limiter surface) and a few MW/m<sup>2</sup> at the divertor target plate. Obviously the present VPS-W coating can meet the requirements. However, for the application at next generation fusion experiment reactors, such as ITER, our VPS-W coating, especially VPS-W coated copper as plasma-facing components, is a distance from the requirements of ITER.

The thermal fatigue properties of VPS-W coating were evaluated by cyclic heat load tests. Experimental

results indicate that both VPS-W coated graphite and VPS-W coated copper can endure 1000 cycles without visible failure at an absorbed power density of 4.8 MW/m<sup>2</sup> and 5 s pulse duration. Previous experiments indicated that VPS-W/CFC successfully suffered 1000 cycles at a heat flux of 35 MW/m<sup>2</sup> and 5 s pulse length without failure, where a aperture of 4.5 mm diameter was adopted to limit the electron beam and only the irradiated area was considered at the calculation of this power density [10]. If taking into account the whole surface area of the coating, the previous experimental conditions are quite similar to the present ones. Which is also an indication that both VPS-W coated graphite and VPS-W coated copper presently developed have relatively good thermal fatigue properties. Additionally, it is reported [7] that 6 mm thickness VPS-W coated CuCrZr with 2 mm thickness graded transition interface has been developed in Japan Atomic Energy Research Institute (JAERI) and it can endure 1000 cycles at 5 MW/m<sup>2</sup> heat flux and 7 s pulse duration. Obviously the inherent stress of this coating arising from coating deposition process is much larger than that of the present coating owing to its large thickness. Although this experimental condition is slightly more rigorous than the present one, absolutely its high heat resistance capabilities are much better than the present coating. Therefore further efforts to the optimized design of graded transition layer and the optimization of spray technology are required in order to develop high quality VPS-W coating on copper substrate. In particular, the thick coating has to be developed.

#### 4. Conclusions

VPS-W/C coating with multilayered W and Si pre-deposited by PVD and VPS-W/Cu coating with

graded transition interface were prepared. The physical properties of VPS-W, such as density (porosity), bonding strength between coating and substrates and thermal conductivity, were measured. The density and thermal conductivity were relatively high.

Both screening and cyclic tests of VPS-W coating were performed by means of a Gaussian-like distribution electron beam. Experimental results indicated that the heat load limitation of VPS-W/CFC, VPS-W/C and VPS-W/Cu was 13, 12 and 10 MW/m<sup>2</sup>, respectively. Both VPS-W coated graphite and VPS-W coated copper could sustain 1000 cycles at a heat flux of approximately 5 MW/m<sup>2</sup> absorbed power density and 5 s pulse duration without visible failure. It is also indicated that present coating can meet the requirements of plasma facing materials in HL-2A tokamak machine and PVD-Si is another good choice of the intermediate layer for W/C coating.

#### Acknowledgement

This work is partly supported by core university program between Japan and China, and VPS-W/CFC coating was supplied by professor N. Yoshida of Kyushu University, Japan. Hereby the corresponding author would like to express cordial appreciations.

#### References

- [1] R. Neu, R. Dux, A. Kallenbach, et al., in: 20th IAEA Fusion Energy Conference, November 1–6, 2004, Villamoura, Portugal.
- [2] R. Neu, R. Dux, A. Kallenbach, T. Eich, A. Herrmann, C. Maggi, H. Maier, H.W. Muller, R. Pugno, T. Putterich, I. Radivojovic, V. Rohde and ASDEX Upgrade Team, in: 31st EPS Conference on Plasma Physics, 28 June–2 July, 2004, London, United Kingdom.
- [3] H. Bolt, V. Barabash, W. Krauss, J. Linke, R. Neu, S. Suzuki, N. Yoshida, ASDEX Upgrade Team, *J. Nucl. Mater.* 329–333 (2004) 66.
- [4] K. Tokunaga, N. Yoshida, N. Noda, T. Sogabe, T. Kato, *J. Nucl. Mater.* 258–263 (1998) 998.
- [5] K. Tokunaga, N. Yoshida, N. Noda, et al., *J. Nucl. Mater.* 266–269 (1999) 1224.
- [6] Ph. Chappuis, F. Escourbiac, M. Chantant, M. Febvre, M. Grattarola, M. Bet, M. Merola, B. Riccardi, *J. Nucl. Mater.* 283–287 (2000) 1081.
- [7] ITER Final Report on Task T437, TA No G 17 TT fr 49 FJ, JA Home Team, June 2001.
- [8] X.R. Duan, Z. Cao, C.H. Cui, et al., *J. Nucl. Mater.*, these Proceedings, doi:10.1016/j.jnucmat.2007.01.185.
- [9] X. Liu, L. Yang, S. Tamura, K. Tokunaga, N. Yoshida, N. Noda, Z. Xu, *Fus. Eng. Des.* 70 (2004) 341.
- [10] X. Liu, S. Tamura, K. Tokunaga, N. Yoshida, N. Noda, L. Yang, Z. Xu, *J. Nucl. Mater.* 329–333 (2004) 687.
- [11] S. Tamura, X. Liu, K. Tokunaga, Y. Tsunekawa, M. Okumiya, N. Noda, N. Yoshida, *J. Nucl. Mater.* 329–333 (2004) 711.
- [12] M. Merola, I. Bobin-Vastra, A. Cardella, et al., *Fus. Eng. Des.* 49&50 (2000) 289.
- [13] C. Garcia-Rosales, S. Deschka, W. Hohenauer, et al., *Fusion Technol.* 32 (1997) 263.