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High heat load properties of tungsten coated carbon materials

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Abstract

Tungsten coatings of 0.5 and 1.0 mm thickness were successfully deposited by the vacuum plasma spraying technique (VPS) on carbon/carbon fiber composite, CX-2002U, and isotropic fine grained graphite, IG-430U. High heat flux experiments were performed on the coated and non-coated samples in order to prove the suitability and load limit of such coating materials. The electron beam irradiation experiments showed that there was little difference in temperature increases among CX-2002U and the coated materials below surface temperature of 2200°C. These results indicated that thermal and adhesion properties between the substrate and coatings were good under high heat flux. A few cracks with a width of 15 μm were formed from the surface to the bottom side of the all coated samples, but plastic deformation and microcracks due to grain growth by recrycrallzation were not observed below a surface temperature of about 2200°C. The cracks are expected to be formed due to local thermal stress produced by spot-like beams.

1. Introduction

Although the utilization of low Z material like carbon materials for plasma facing components has enabled the improvement in plasma confinement, their high erosion rates at elevated temperatures is now serious problem. Degradation of thermal conductivity by neutron damage and high tritium retention would be serious problem in next generation of D–T fusion experimental reactor [1]. Owing to its low sputtering yield and good thermal properties, tungsten seems to be a promising candidate material for plasma facing components in next fusion experimental devices but is not easy to machine and weld.

For technical realization of a tungsten material, tungsten coated carbon tiles can be envisaged. Tungsten coatings on graphite by plasma spray or physical vapor deposition (PVD) were produced [2,3]. It is preferable to use of the Carbon/carbon fibre composite (CFC) for high heat flux components owing to its good thermal conductivity and mechanical strength. In this study, thick tungsten coatings on (CFC) as well as isotropic fine grained graphite was newly, successfully produced. High heat flux experiment were performed on the coated samples in order to prove the suitability and load of such coating materials.

2. Experimental

Tiles, 20 mm × 20 mm × 10 mm,were coated by vacuum plasma spraying technique (VPS). The substrate materials were carbon/carbon composite CX-2002U and isotropic fine grained graphite IG-430U made by Toyo Tanso. The CX-2002U and IG-430U received a diffusion barrier layer of rhenium prior to the VPS coating in order to inhabit uncontrolled brittle carbide formation. Heat treatment were performed to stabilize microstruture of the sample. Thickness of the tungsten coating layer was 0.5 and 1.0 mm. Density of VPS tungsten (VPS-W) was 92.5% of that of theoretical density. The high heat load experiments of bare CX-2002U were performed to compare with the results of coated samples.

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The facility used this experiments was an electron beam irradiation test simulator of Research Institute for Applied Mechanics (RIAM) of Kyushu University [4]. The samples were mechanically fixed on copper block actively cooled with water. A carbon sheet with a thickness of 0.38 mm was inserted between the sample and the copper block to make thermal contact between them constant. The electron beam energy used was 20 keV. The beam diameter was 8 mmϕ. Duration of beam was 10 s. The heat load experiments were performed by stepwise increasing the heat flux. The surface temperature of the center region with a diameter of 1 mm of the

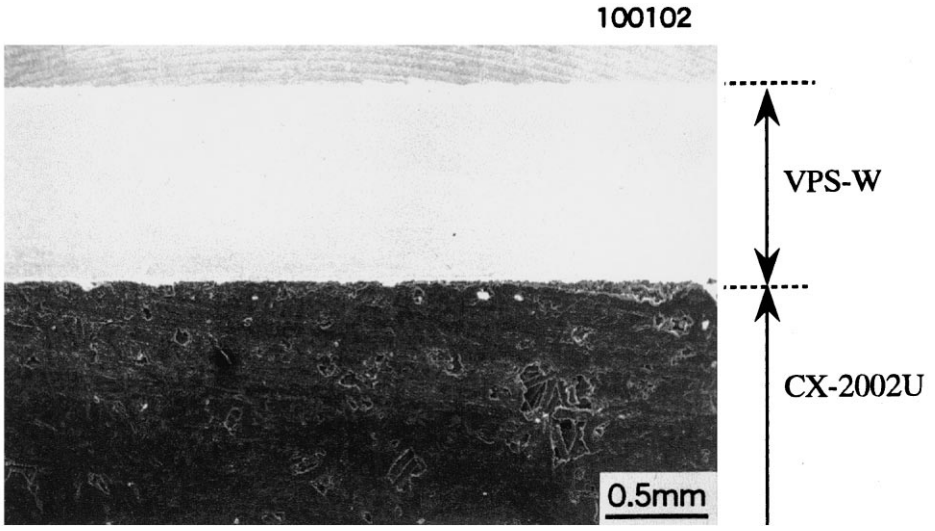

Fig. 1. SEM image of cross section of VPS-W coated CX-2002U (#6) with a thickness of tungsten of 1 mm.

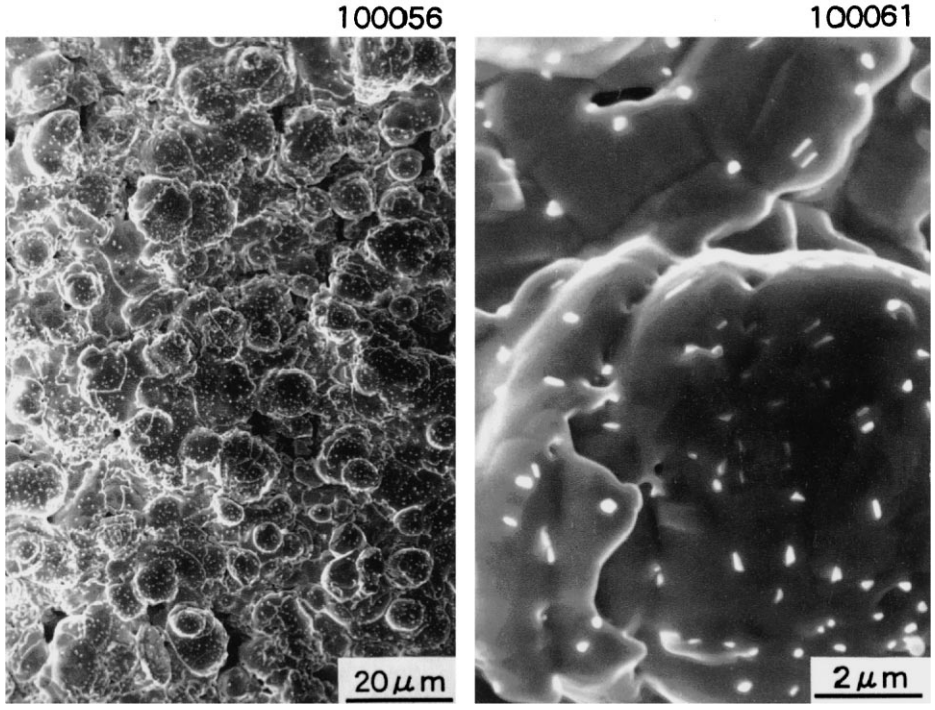

Fig. 2. SEM image of surface of VPS-W coated CX-2002U (#10) with a thickness of tungsten of 0.5 mm.

sample was measured with two-color optical pyrometers (400–1100°C, 1000–3100°C). The gases emitted from the heated sample surface were detected with a quadrupole mass spectrometer (QMS). Heat flux was evaluated by the beam diameter and net electric current of the electron beam irradiated. The net current was measured by applying a bias voltage to the sample to suppress the secondary electron induced by the electron irradiation. Temperature difference between inlet and outlet water of cooled copper block was measured by ΔT system [5] to evaluate heat removable capability of the sample. Water flow rate was also measured. Before and after the irradiation, the sample surface was observed with a scanning electron microscope (SEM).

3. Results

Fig. 1 shows SEM image of cross section of VPS-W coated CX-2002U (#6) with a tungsten thickness of 1mm. It can be seen that the thickness of the tungsten layer is constant and that no cracks can be seen around the joint interface. The Re layer is not visible due to the low magnification. Fig. 2 shows an SEM image of the surface of VPS-W coated CX-2002U (#10) with a tungsten thickness of 0.5 mm. This shows that spherical particles were melted or partially melted and joined each other and pores were formed in the coatings. The same features were observed on the surface of VPS-W coated carbon materials during the SEM observation.

Fig. 3(a)–(d) show the time evolution of the electric current of the sample (a), pressure of vacuum chamber (b), surface temperature (c) and temperature difference between inlet and outlet cooling water (d). Bias voltage was not applied. The sample was VPS-W coated CX-2002U (#10) with a thickness of tungsten of 0.5 mm and the heat flux was 18.6 MW/m^2. The electric current started to increase at the same time as the irradiation started and was almost constant during irradiation. The surface temperature gradually increased and reach about 1800°C and started to decrease when the irradiation ended. The pressure of vacuum chamber gradually increased during irradiation and decreased after the irradiation. Fig. 3(d) shows that maximum temperature difference of inlet and outlet water was 0.58°C. This indicated that a part of incident energy of electron beam flowed to water cooled copper block.

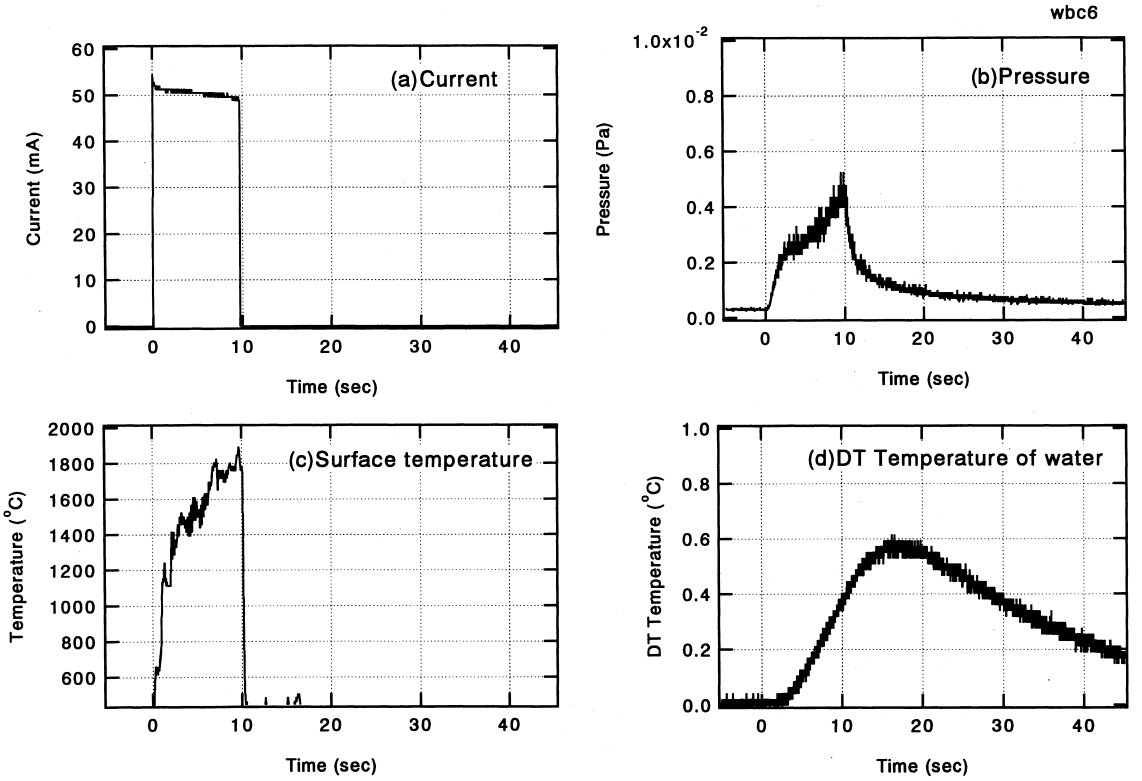

Fig. 3. Time evolution of the electric current of the sample (a), pressure of vacuum chamber (b), surface temperature (c) and temperature difference between inlet and outlet cooling water (d). Bias voltage was not applied. The sample was VPS-W coated CX-2002U (#10) with a thickness of tungsten of 0.5 mm. Heat flux was 18.6 MW/m^2.

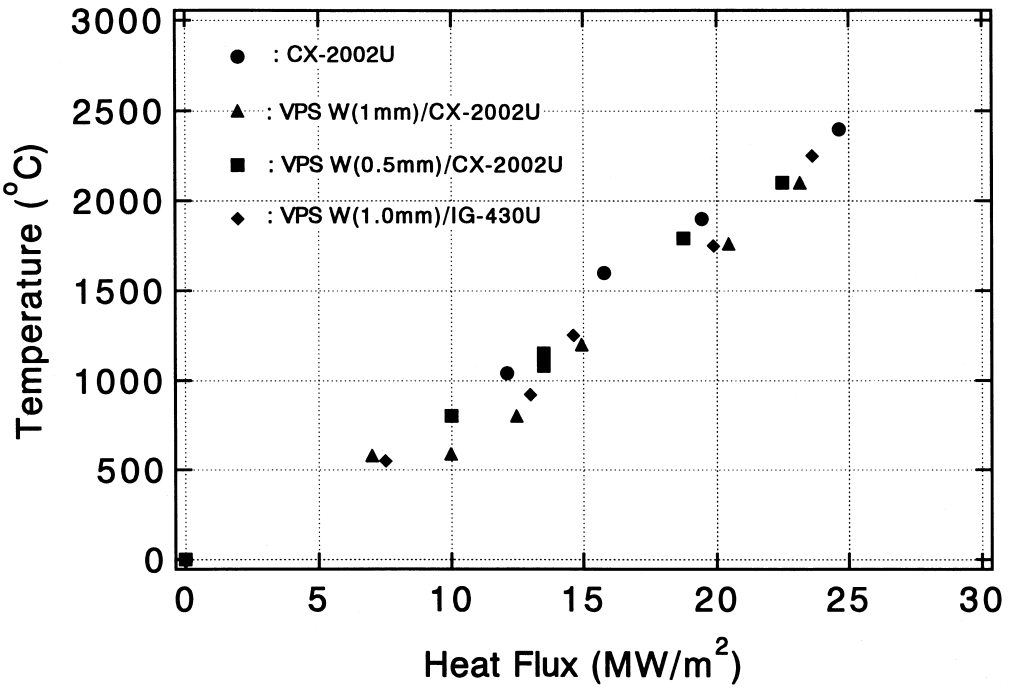

Fig. 4. Surface temperature increase of the samples as a function of the heat flux. The samples were CX-2002U, VPS-W/CX2002U (#10, #14) with a thickness of tungsten of 0.5 and 1 mm, VPS-W/IG-430U (#17) with a thickness of tungsten of 1.0 mm. Duration of electron beam was 10 s.

Fig. 4 shows surface temperature increase of the samples as a function of the heat flux. The samples were CX-2002U, VPS-W/CX2002U (#10, #14) with a thickness of tungsten of 0.5 and 1 mm, VPS-W/IG-430U (#17) with a thickness of tungsten 1.0 mm. The duration of the beam was 10 s. It can be seen from the figure that the temperature linearly increased with increasing heat flux and that there was little difference among the samples. This means that the samples were almost in an adiabatic condition. It is expected that the thermal and adhesion properties at high temperature were good enough because there was no difference of surface temperature increase between tungsten coated and no-coated samples.

QMS spectra before and during irradiation, 6 s after the beam irradiation started, correspond to Fig. 3 are shown in Fig. 5. Emitted gases were mainly H_2, H_2O, CO and CO_2. This result indicated that kind of the emitted gases was almost the same as that of powder metallurgy tungsten and CX-2002U[6].

After the irradiation experiments, the samples were removed from the copper block and observed with an optical microscope and an SEM. Fig. 6 shows SEM images of the surface of the samples after the heat load experiments. A few cracks were observed in the center region on the surface of the all tungsten coated samples as shown in Fig. 6. Fig. 7 shows SEM images of the surface of the VPS-W/CX-2002U (#10) with a thickness of tungsten of 0.5 mm tungsten in higher magnification. It can be seen that a width of the cracks was about 15 μm and the direction of the crack was from the surface to down side. Plastic deformation and microcrack due to grain growth by recrystallization were not observed. Results from high heat flux experiments of same samples by irradiation of uniform profile of electron beam showed that cracks were not formed before melting occurred [7]. Therefore, it is expected that the cracks on the surface were formed due to local thermal stress by the irradiation of spot-like electron beams.

4. Discussion

The covalent bonds of group VIa such as tungsten and molybdenum are strong. Accordingly, they have high cohesive energies and high melting points. However, their grain boundary is intrinsically weak due to electron bonding structure because heterogeneity of bonding is strong due to the covalent bond [8]. It is known that polycrystalline tungsten heated to the recrystallization temperature becomes brittle and that fractures along grain boundaries are formed due to recrystallization embrittlement [9]. Ductile-brittle transition temperature (DBTT) of powder metallogy tungsten

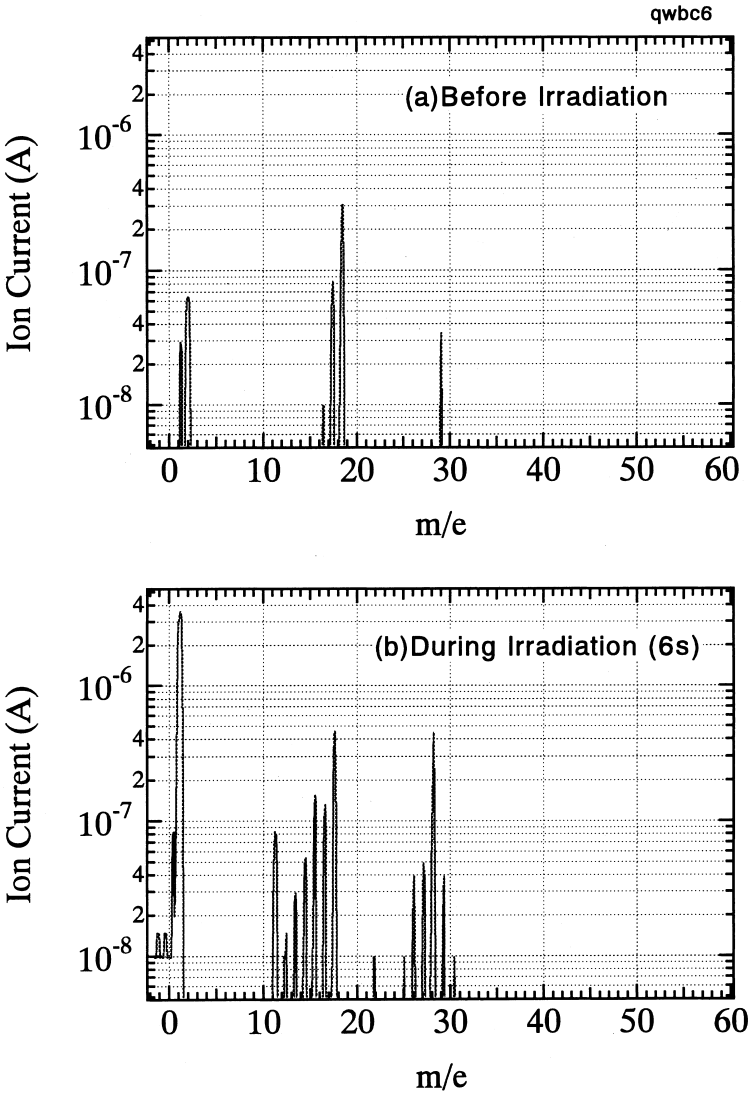

Fig. 5. QMS spectra before and during irradiation (6 s after the beam irradiation started) correspond to Fig. 3.

(PM-W) is between 150°C and 400°C depending on purity, previous deformation and grain size. It is expected that use of below DBTT causes brittle fracture. In these experiments, the surface temperature of VPS-W exceeded over 2000°C but grain growth was not observed. The tungsten coatings produced by the vacuum plasma spraying are built by successive accumulation of molten or partially molten droplets. As a result, the character of VPS-W is expected to be different from that of the PM-Mo. Internal strain energy of the VPS-W, which is driving force of recrystallization at high temperatures, may be weak because the strain energy may be released during the accumulation of tungsten droplets or heat treatments after the product of the sample.

Cracks were formed on the coated samples. The direction of the cracks were from the surface to lower side of the sample and not parallel to heat flow. Therefore, these cracks were considered to hardly influence the degradation of thermal conduction of the sample. Pores were presented in the samples as shown in Fig. 2. The porosity present in the vacuum plasma spraying coating provides a crack-arresting mechanism, so that stresses arising in the tungsten layer during thermal loading can be relieved by pores and limited crack formation without disabling failure of the coating.

Tungsten forms tungsten carbide over 1250°C [10] and brittle carbide is produced. The coating samples had a rhenium-containing diffusion barrier between the tungsten and the carbon. Heat treatment after coating

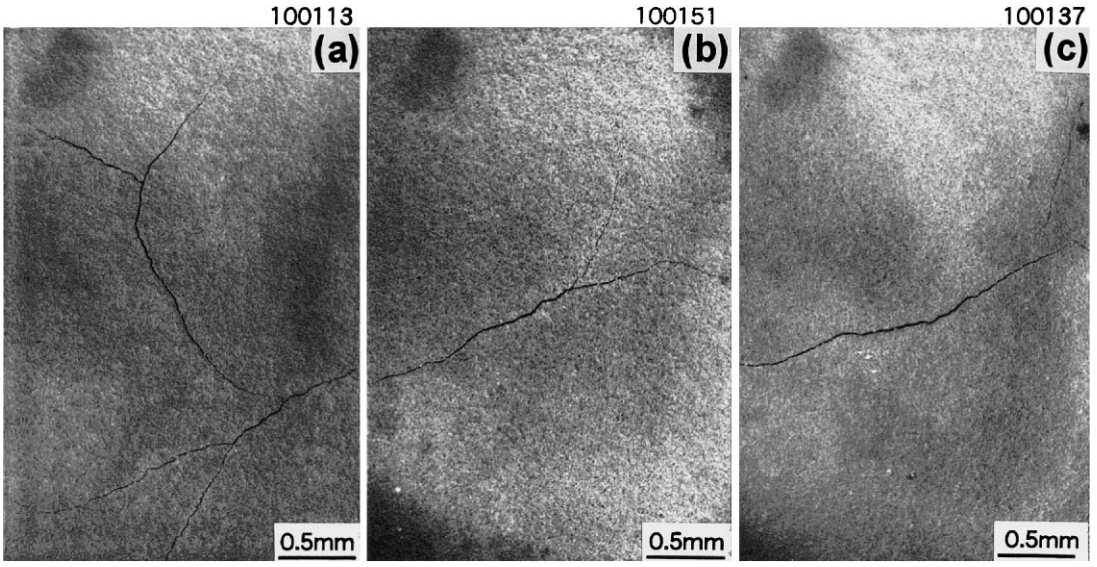

Fig. 6. SEM images of the surface of the samples after the heat load experiments. (a)VPS-W/CX-2002U (#14), thickness of tungsten is 1.0 mm. (b)VPS-W/CX-2002U (#10), thickness of tungsten is 0.5 mm. (c)VPS-W/IG-430U (#17), thickness of tungsten is 1.0 mm

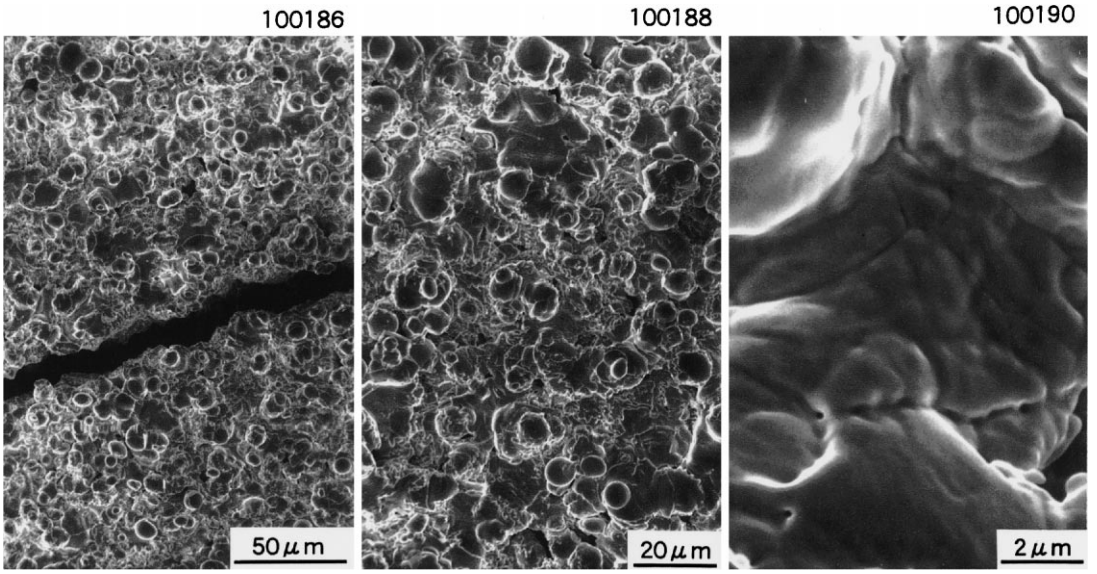

Fig. 7. SEM images of the surface of the VPS-W/CX-2002U (#10) with a thickness of tungsten of 0.5 mm in higher magnification.

produced a stable microstructure. That is expected to suppress the formation of the brittle carbide. In these experiments, the temperature of the interface of the tungsten and the carbon was expected to be rather high, therefore, graphitization of the carbon and formation of new microstructure might occur. It is expected that there is relationship between the formation of the cracks and the microstructural change. Detailed cross sectional observation of the interface is required to confirm this.

5. Summary

Tungsten coatings of 0.5 and 1.0 mm thickness were successfully deposited by the VPS on carbon/carbon fiber composite, CX-2002U, and isotropic fine grained graphite, IG-430U. High heat flux experiments were performed on the coated and non-coated samples. The electron beam irradiation experiments showed that there was little difference between temperature increases

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Acknowledgements

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