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Failure behaviors of vacuum plasma sprayed tungsten coatings for plasma facing application

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ABSTRACT

About 1 mm vacuum plasma sprayed tungsten (VPS-W) coatings were fabricated on the copper chromium zirconium (CuCrZr) alloys substrate. The failure behaviors were studied by means of the steady state and transient heat load using the electron beam facility and the Nd: YAG laser, respectively. The results indicated that the columnar crystals grew up and then micro-cracks between the lamellar layers were observed. Macro-cracks and delamination appeared with the fatigue cycles increase. Finally, the coating failed. The surface cracks were also observed and propagated during the fatigue tests. The failure behaviors of the transient heat load are as follows: the homogeneous melting and micro-cracks, melting tungsten ejection which enhanced the erosion of tungsten due to the splash and evaporation. In addition, the physical properties of W coatings such as porosity, Vickers hardness were degraded. The roughing phenomenon was not easy to be observed due to the rough surface characteristic of VPS-W coatings. © 2009 Published by Elsevier B.V.

1. Introduction

Tungsten is a candidate material for the diverter armor of the international thermonuclear experimental reactor due to its low physical sputtering rate, low tritium inventory, high melting point and high thermal conductivity [1–3]. And it is expected to exposed to the steady state heat flux of 10 MW/m^2 , and the high energy transient heat load event, such as disruption, edge local modes (ELMs), vertical displacement events (VDEs) [4].

Heat flux tests were applied to evaluate the thermal shock behaviors of tungsten as plasma facing material using the electron beam facility and tokamak plasma irradiation [5-8]. The cracks, delamination and melting were observed with the increase of heat flux, though it can withstand about 10 MW/m² heat flux. But few researches were carried out concerning the failure process during the heat flux.

At the present, vacuum plasma sprayed tungsten (VPS-W) coatings were fabricated on copper chromium zirconium (CuCrZr) alloys substrate. And its failure behaviors were studied by means of the heat flux tests using the electron beam facility and the Nd: YAG laser. In addition, the properties of VPS-W coating before and after the heat load tests were measured.

2. Steady state heat load to VPS-W by electron beam facility

Failure behaviors of steady state heat load to 1 mm VPS-W coatings with 200 µm W/Cu compliant layer were carried out using the electron beam facility at ASIPP under the vacuum of $1.5-4 \times 10^{-3}$ Pa. The heat flux of 10 MW/m² was loaded on an area of about 20×20 mm² and beam duration during ramp up, plateau and ramp-down were 20, 200, 5 s, respectively. Each cycle interval was 300 s to make the surface temperature below the DBTT. W/ CuCrZr mock up was directly cooled with a water velocity of 10 m/s and inlet temperature of 20 °C [3]. The surface temperature was measured by an infrared pyrometer (500-2000 °C), and bulk temperature was measured by a thermocouple (0–1000 °C), which was imbedded into the substrate from the side surface and located below the coating/substrate interface.

Fig. 1 shows the failure process of VPS-W with the increase of fatigue test cycles. It can be seen that lamellar structure was the typical characteristic of plasma sprayed coatings, and only several pores were scattered in the vicinity of the laminar layers. The columnar crystals were observed perpendicular to the lamellar direction and the grain boundary was clearly. In the process of fatigue tests, the columnar crystals re-crystallized and grew up, and then micro-cracks between lamellar layers were created due to the repeating heat flux load (see Fig. 1(c)). With the increase of fatigue cycles, the micro-cracks propagated and extended to the macrocracks or delamination, such as Fig. 1(d) and (e). Finally W coating failed. Of course, during the tests, the cracks also appeared on the surface and propagated with the increase of fatigue. Laminar





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Fig. 1. Failure process of VPS-W coating with the increase of fatigue cycles. (a) Original coating, (b) 5 cycles tests, (c) 20 cycles tests, (d) 50 cycles tests, and (e) 100 cycles tests.

structure of VPS-W coating with pore characteristic provides a crack arresting mechanism, and the stresses can be relieved by the limited crack formation, though the laminar structure reduces the thermal conductivity and increases the surface temperature. Columnar crystal structure is favorable to the heat transfer and confines the crack to propagate transversely, so the capability of heat removal was influenced less, even crack appearing along the columnar. No damage was observed at the W/Cu interface.

Fig. 2 shows the temperature evolution of VPS-W coatings with the increase of the fatigue tests at 10 MW/m² heat flux. Before 50 cycles, the mean surface temperature increased gradually from 1252 to 1307 °C, which indicated that crystals growing up and micro-cracks formation had the less influence on the heat transfer.



Fig. 2. The temperature evolution of VPS-W coatings with the increase of fatigue tests at 10 MW/m^2 heat flux.



Fig. 3. The pore size distribution of VPS-W coatings before (a) and after (b) heat load tests.



Fig. 4. Vickers hardness change of VPS-W coatings before and after heat load tests.

But when the macro-crack appeared, the surface temperature increased rapidly to 1523 °C, and then it exceeded 2000 °C after 100 cycles. The thermocouple measurement observed that bulk temperature change did not change much. Even when the coating failed, it was only 830 °C.

The distribution of pore size diameter and the porosity were measured by means of mercury porosimeter instrument (AutoPore IV 9500) at RT. The low porosity of 6.87% and the narrow pore size



Fig. 5. The surface morphology change of VPS-W coatings after laser irradiation. (a-1) 20 pulses, irradiation center, (a-2) 20 pulses, irradiation surrounding, (b) 100 pulses, (b-1) 100 pulses, irradiation center, (b-2) 100 pulses, irradiation surrounding, (c) 300 pulses, (c-1) 300 pulses, irradiation center, (c-2) 300 pulses, irradiation surrounding.

distribution of 0.08–1 μ m were obtained in the W coating. But after 50 cycles, the porosity showed 14.5% due to the defects formation, such as cracks, delamination. The peak increased to about 0.025 mg/l from 0.015 mg/l of original W coating, and a few pores of about 6.5 and 60 μ m were also observed, which is shown in Fig. 3.

Vickers micro-hardness was measured on the cross section from the coating surface to the substrate with the load of 100 N for 10 s. Due to the effect of porosity and the residual stress, the Vickers micro-hardness reveals significant scattering. The mean hardness is 271 MPa, however, after 50 cycles fatigue tests, the mean hardness changes into 234.1 MPa. Vickers micro-hardness distribution from W coating to CuCrZr substrate is shown in Fig. 4.

3. Transient heat load to VPS-W by the Nd: YAG laser

Failure behaviors of transient heat load to VPS-W were performed by means of the Nd: YAG laser. The laser parameters were as follows: a wave length of 1.06μ m, the pulse duration of 5-9 ns, the pulse interval of 0.1 s, Gaussian distribution pulse energy of 2 J on the area of about 1 cm². The temporal evolution of surface temperature during the laser pulse irradiation could not be measured.

Fig. 5 shows the surface morphology change of VPS-W by laser irradiation. Unfortunately, the surface roughing phenomenon was not observed due to the rough surface characteristic of W coatings by means of stacks of many laminar splats. Fig. 5(a-1) and (a-2) shows the obvious surface homogeneous melting and



Fig. 6. Weight loss rates evolution of VPS-W under the transient heat load.

micro-cracks, and the surrounding zone cracks were more severe than center. The compressive stresses caused by surface expansion and the tensile stresses caused by the shrinkage after loading would be responsible for the cracks formation. In addition, except the loaded area the whole surface is at room temperature, therefore, at the surrounding zone cracks were also attributed to the temperature gradual stresses. With the increase of pulses, the loaded area was in a state of being melted, and the melting tungsten was ejected around, which were shown in Fig. 5(b-2) and (c-2). The smooth crater can be seen clearly after 100 pulses (see Fig. 5(b)), and the crater enhanced at 300 pulses. From the cross sectional SEM observation it can be seen that the damage such as coating melting, crystals re-crystallized and grew up only appeared at the near surface.

Weight loss rates were also examined, which is shown in Fig. 6. Tungsten had the less weight loss rates due to its heavy weigh and high sputtering threshold energy. But the melting of W strongly enhanced the weight loss due to the splash and evaporation.

4. Conclusion

About 1 mm VPS-W coatings were fabricated on CuCrZr alloys substrate for the plasma facing application. The heat flux tests and laser irradiation were carried out to study the failure behaviors of the steady state and transient heat load to VPS-W coatings.

When the surface temperature was higher than the re-crystallization temperature, the columnar crystals grew up and microcracks appeared between lamellar layers. With the heat load increase, macro-cracks and delamination were observed. Finally, coating failed.

Under the laser irradiation, the micro-cracks and homogeneous melting were observed, and then melting tungsten was ejected around, which enhanced the erosion of tungsten. Due to the rough surface characteristic of W coatings, the roughing phenomenon of surface was not easy to be observed.

After heat load tests, the certain physical properties of W coating were degraded because of the defects formation, such as microcracks.

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