Material behavior on heat loading and hydrogen penetration of vacuum plasma spray tungsten coatings on reduced activation ferritic/martensitic steel*


Tungsten coating with a thickness of 0.6 mm on reduced-activation ferritic/martensitic steel (RAF/M) F82H (Fe-8Cr-2W) have been produced by Vacuum Plasma Spraying (VPS). Heat flux experiments using an electron beam and quantitative analyses about temperature profiles and thermal stress using FEM have been carried out on the VPS-W coated F82H. In addition, behavior of hydrogen penetration/permeation on the VPS-W coated F82H has been investigated by the tritium (T) tracer technique.

Key Words: Tungsten, Coating, Ferritic steel, Plasma spray, Tritium

1. Introduction

The fusion reactor would be planning to be one of the next-generation infrastructure energy source. Tungsten is potential candidate for an armor of the first wall and the divertor plate of the fusion reactor because of its low erosion yield and good thermal properties. In the case of the fusion demonstration reactor (DEMO), neutron damage will be a critical issue. Structure materials of the first wall/blanket and the cooling channels of the divertor will be made by low activation materials. Tungsten coated reduced activation materials could be convenient for the first wall/blanket because the thickness of tungsten on the first wall/blanket is designed about 1 mm and the coating technique can be used for this. In the present work, tungsten coating with a thickness of 0.6 mm on reduced-activation ferritic/martensitic steel (RAF/M) F82H (Fe-8Cr-2W) have been produced by Vacuum Plasma Spraying (VPS)1). Heat flux experiments using an electron beam and quantitative analyses about temperature profiles and thermal stress using FEM have been carried out on the VPS-W coated F82H to evaluate their possibility as a plasma-facing armor in the fusion device. In addition, behavior of hydrogen penetration/permeation on the VPS-W coated F82H has been also investigated by the tritium (T) tracer technique2) to understand the role of the VPS-W on T permeation of the first wall/blanket of the fusion reactor

2. Experimental procedure

2.1 Samples

Reduced activation ferritic/martensitic steel used in the present works was F82H (Fe-8Cr-2W). The substrate of F82H was coated with W via VPS). The thickness of the W coating layer was 0.6 mm and its density was 89 % of the theoretical value. In order to suppress oxidation and change of the ferritic/martensitic structure during the spraying processes, bulk temperature of the substrate was kept below 577 °C for VPS. The average size of the W powders for VPS was 18 μm. The sample sizes used in the heat loading and the hydrogen penetration/permeation experiments were 10mm x 10mm x 5.6 mm and 7.8mm x 9.4mm respectively. The VPS-W surface was mechanically polished before the experiments.

2.2 Experimental

The facility used for the heat loading experiments was an electron beam irradiation test simulator of Research Institute for Applied Mechanics (RIAM) of Kyushu University3). The samples were mechanically fixed on copper block actively cooled with water. The electron beam energy was 20 keV and the beam diameter was 2.5-3 mm. The experiments were conducted at three irradiation conditions: (1) Heat flux of 7.5 MW/m², duration of
180 s, (2) cyclic irradiations of 60 s on and 140 s off with a heat flux of 12 MW/m$^2$ with 30 cycles in total and (3) cyclic irradiations of 7 s on and 230 s off with a heat flux of 40 MW/m$^2$ with 30 cycles in total. The surface temperature of was measured with two-color optical pyrometers. Thermocouples are inserted in the F82H where is 4mm deep and 2mm underneath the interface.

In addition, the surface of the coating side of the samples was exposed to dc glow discharge (DCGD) of hydrogen including T (T/H = 10$^{-4}$) through a $\phi$= 5mm aperture made of molybdenum. The operating gas pressure of DCGD was 0.27 kPa and the applied dc voltage was 400V. The loading was conducted at 573K for 2 h. Subsequently, the sample was cut in half perpendicular to the plasma-exposed surface to get the cross-section at room temperature (RT). The T distribution on the cross-sectional surface was analysed by TIP for 24 h at 233 K.

2.3 Thermal analyses

The steady state thermal and stress analyses were performed for the VPS-W/F82H in the experimental condition (1) using the finite element code ANSYS. Only 1/4 geometry was considered due to symmetry. The temperature dependence of the materials properties of thermal conductivity, coefficient of thermal expansion (CTE), elastic modulus, Poisson’s ratio and emissivity were taken into account.

3. Result and discussion

Figure 1 shows a time evolution of a heat flux and temperatures of the VPS-W surface and the F82H measured by the thermocouples (Experimental condition (1)). The temperatures of the W surface and the F82H gradually increase and reach at 700 °C and 500 °C corresponding to the heat flux, respectively. Maximum operation temperature of F82H is limited to 550 °C from a viewpoint of irradiation creep and swelling by neutron irradiation. Temperature of small part of F82H near VPS-W was above 550 °C judging from the calculation result, which will be presented later. However, in this experiment, steady state heat loading for 180 s has been performed under the condition that temperature of the most of F82H was below the maximum operation temperature. SEM observation of VPS-W/F82H after the heat loading experiment showed that no modification was observed.

Figure 2 shows a time evolution of heat flux and temperatures of the thermal fatigue experiment of 30 cycles at 12 MW/m$^2$ (Experimental condition (2)). The electric current starts to increase at the same time as the irradiation starts and is almost constant during irradiation. The surface temperature of W gradually increases and reaches about 750 °C and starts to decrease when the irradiation ends. In addition, time evolution of the temperature of the F82H shows the same temperature changing tendency. The peak temperatures did not change during the cycle heat loading. These results indicate that no failure occurred at the interface or in the VPS-W coating during the cyclic heat loading. After the cyclic heat loading, SEM observation has been carried out.

Figure 3 shows the surface before and after the cyclic heat loading. Fine modification is observed, however, macro-cracks and exfoliation between the joint interface of the VPS-W/F82H were not formed after the heat loading experiments. The fine modification on the VPS-W surface may be caused by thermal process or thermal stress on the VPS-W corresponding to micro-structure of the VPS-W. These results indicate that the thermal and adhesion properties between the joint interface of the VPS-W/F82H are good under heat loading at 750 °C of surface temperature.

Figure 4 shows a time evolution of heat flux and temperatures of the thermal fatigue experiment with 30 cycles at 40 MW/m$^2$ (Experimental condition (3)). Figure 4(a) and 4(b) show the
results from 4th to 7th cycles and 20th to 23th cycles. The peak temperatures of VPS-W surface increases from about 1300 °C to 1700 °C as cyclic number increases. On the other hand, the peak temperature of F82H decreases from 480 °C to 300 °C as cyclic number increases. These results mean that departure of temperatures happened and exfoliation for parallel direction occurred.

During and after the cyclic experiment at 40 MW/m² with 30 cycles, exfoliation occurred. Figure 5 shows SEM image of cross section of the interface of the VPS-W and F82H of the sample after the irradiation. It can be seen that exfoliation occurred at interlayer of the VPS-W coatings at about 50μm from the interface of the VPS-W and the F82H and micro-cracks were formed. In addition, cracks were observed on the VPS-W surface and penetrated to bottom layer of the VPS-W, which was exfoliated. These results indicate that the thermal stress caused exfoliation between the VPS-W layer at first, then, the cracks

Fig.3 SEM images of VPS-W surface before (a) and after (b) cyclic heat loading of experimental condition (2)

Fig.4 Time evolution of a heat flux and temperatures of VPS-W surface and F82H of experimental condition (3). Figure 4(a) and 4(b) show the results from 4th to 7th cycles and 20th to 23th cycles.

Fig.5 SEM image of cross section after heat loading of experimental condition (3).

Fig.6 Temperature of the experimental and FEM analyses as a function of distance from the VPS-W surface. The location of parallel plane for the surface, which temperatures is presented in fig. 6, is center on the surface of VPS-W.
from the surface to bottom of the VPS-W layer occurred by the further cyclic heat loading. In addition, it is considered that exfoliation occurred at the location near to the interface between the VPS-W and F82H, where large thermal stress was applied and weak interlayer existed.

The steady state thermal and stress analyses have been performed to evaluate quantitatively thermal behavior under the experimental condition (1). Figure 6 shows a result of temperatures of the experimental and the FEM analyses as a function of distance from the VPS-W surface. The location of parallel plane for the surface, where temperatures are presented in fig. 6, is center on the surface of VPS-W. When the thermal conductivity of the VPS-W is 30% of that of pure tungsten, calculate value gives close agreement with the experimental value. In addition, in this case, thermal resistance between the VPS-W and F82H was not taken account in the calculation. These mean that thermal and adhesion properties between the substrate and the coatings are good.

Figure 7 shows thermal stresses for X, Y and Z direction in the dimension of 5 mm x 5mm x 1.6 mm near the interface in the sample. A thermal conductivity of the VPS-W layer of about 30 % of bulk W was used in the calculation. For X and Y direction, shear stress arises between the VPS-W and F82H and stress for center direction and outer direction are applied in F82H and VPS-W part, respectively. For Z-direction, it seems that shear stress is not applied, however, the F82H part is much swelled comparing with the VPS-W. In addition, Mises stress was also calculated. The Mises stress is scalar value which is projected one axis stress and is used as fracture strength. In this case, temperature of the VPS-W is above DBTT. Therefore, the VPS-W and F82H are regarded as ductile materials. As a result, it is expected that the strength can be evaluated using Mises stress.

The calculation results show that stress of 541 MPa was applied near the interface between the VPS-W and F82H. It is considered that stress of VPS-W is below elastic limit judging from the result of tensile test of pure W. However, evaluation may be difficult because mechanical property of VPS-W may be different from that of pure W. On the other hand, because stress of the part of F82H just below the center of electron beam is slightly above 0.2% proof stress, plastic deformation may occur. In this case, stress is relaxed and become diminished.

Figure 8 shows the T profiles perpendicular to the depth profiles at 573 K. Except for high T concentration within a few hundreds of μm in depth, the T concentration in the coating was nearly uniform along the depth direction. Since the VPS-W coatings have porous microstructures, T can diffuse through open pores or migrate along grain boundaries to be adsorbed or trapped on the W grain surfaces. The T trapped at the W grain surfaces could diffuse into the grains, resulting in uniform distribution of T within the whole depth of the coating. In addition, the diffusion coefficient of T in F82H is large enough to allow T permeation to the back surface, resulting in nearly constant T profiles in F82H.
4. Conclusions

High density W coatings on RAF/M (F82H) have been produced by vacuum plasma spraying technique (VPS) and heat flux experiments on them have been carried out to evaluate their possibility as a plasma-facing armor for the fusion device. In addition, quantitative analyses about temperature profile and thermal stress have been carried out using the finite element analysis (FEA) to evaluate thermal property. Macroscopic cracks and exfoliation have not formed after the heat loading experiments under heat loading condition at 700 - 750 °C of surface temperature. These results have shown that the thermal and adhesion properties between the joint interface of the VPS-W coated F82H are good. These indicate that the VPS-W coated F82H has high potential of these coating as plasma-facing armor under thermal loading of the first wall in the fusion power plant. On the other hand, exfoliation has occurred at interlayer of the VPS-W coatings near the interface of the VPS-W and the F82H by cyclic heat loading at 1300 °C of surface temperature. This means that strengthening of the VPS-W interlayer will be necessary for improvement of thermal property of the VPS-W coated F82H.

In addition, in order to understand the role of a plasma-sprayed tungsten (W) coating on tritium (T) permeation in a W-coated ferritic/martensitic steel (F82H), depth profiles of T in the coating and the substrate have been examined using the tritium imaging plate technique after T loading by a dc glow-discharged plasma at 573K for 2 h. Tritium loaded by plasma exposure is distributed uniformly in the whole coating, while T penetrated to the substrate by diffusion. The former is caused by T diffusion through open pores and/or along grain boundaries followed by adsorption on grain surfaces and dissolution in the grains. The main role of the W coating on T permeation is to reduce the incoming flux at the coating/substrate interface owing to pore diffusion in the coating and the effective area for T dissolution in the substrate.

Reference

2) T. Ostuka et al., PHYSICA SCRIPTA, T145,014035.