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Interface strength measurement of tungsten coatings on F82H substrates

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ABSTRACT

In the current work, hot isostatic pressing is adopted to deposit tungsten coatings on F82H substrates. The interface strength of the W/F82H samples is measured using the Laser Spallation technique and the microstructure is analyzed to determine the strength of the coating. Finally, the failure mechanisms of the hot isostatic pressing versus vacuum plasma spraying tungsten coatings and their different failure strengths are compared. It is concluded that the hot isostatic pressing process ensures a good adhesion for the W/F82H interface while the vacuum plasma spraying process results in relatively lower failure strength for the W-coating itself due to the high porosity in the coating.

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1. Introduction

Tungsten is the primary candidate as an armor material to protect the low activation ferritic steel wall chambers for IFE power reactors. Two of the proposed techniques to apply tungsten coatings on F82H ferritic steel are vapor plasma spraying (VPS) and hot isostatic pressing (HIP). In previous work, Kim et al. [1] evaluated the failure strength of VPS W-coatings and concluded that due to the non-negligible amount of pores produced in the process of coating, the mechanical reliability of the VPS W-coatings is a critical issue. In the current work, we investigate the failure mode and the strength of HIP W-coatings and compare the results with VPS W-coatings.

The failure strength measurements are evaluated using the Laser Spallation (LS) technique. This technique is advantageous over other test methods in the calculation of the failure strength of coatings and interface strength due to its relatively simple test setup and the absence of plastic effects when performing failure analysis. This technique relates the failure strength to the displacement velocity of a coating exposed to compression/tension stress waves propagated through the substrate using a nano-second laser. Detailed information about the experimental setup and the mathematical formulation used in evaluating the failure strength may be found elsewhere [1–4].

2. Sample preparation

The HIP samples were produced at the Japan Atomic Energy Research Institute (JAERI) in Naka, Japan. The ferritic steel starting

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material was heat treated at 1313 K for a duration of 40 min for normalizing and then at 1023 K for a duration of 60 min for tempering. A rod \emptyset 28 mm \times 30 mm was mechanically cut out from a 32 mm thick plate and the bonding interface was polished by 0.03 μ m SiC powder and finally degreased by acetone. The chemical composition in mass% of the F82H is shown in Table 1 [8].

The tungsten material was mechanically cut out from a rod \emptyset 28 mm × 100 mm by electrical discharge machining to dimensions of \emptyset 20 mm × 0.05 mm. The tungsten purity was 99.95% and the disk was degreased by acetone. The specimen was then pretreated by encapsulating it in an SUS304 capsule and heated up to 1373 K for 1 h. The vacuum level inside the capsule was 5×10^{-4} Pa. The W/F82H joint was fabricated with HIP conditions at 1243 K, 143 MPa and 2-h holding time. The temperature and pressure history are shown in Fig. 1 [8].

On the other hand, the VPS samples were produced at the Plasma Processes Inc. (PPI) of Huntsville, Alabama. The initial ferritic steel substrate has dimensions $25 \text{ mm} \times 25 \text{ mm} \times 5 \text{ mm}$ and the dimensions of the free standing VPS W disc is $2 \text{ mm} \times 12.7 \text{ mm}$ diameter. The thickness of the deposited coating was about $125(\pm 25) \mu m$. The bulk density of the W-coating is reported to be 80% and a pore size less than 200 nm was observed. The W-coating was then polished at Oak Ridge National Laboratory (ORNL). The thickness of the specimen was measured at the center to determine when polishing had removed $50 \mu m$ of material, thereby leaving approximately $60-70 \mu m$ of W-coating at the center. Due to curvature of the specimen, less material was removed at the corners leaving a slightly thicker W-coating there.

The VPS technique results in a lower tungsten density, ρ , and a lower Young's modulus, *E*, and thus the material properties used in the analysis of the VPS W-coatings are for 80% dense material properties [1,6]. For the HIP technique the density of the W-coatings does not change much and thus the material properties are those of bulk W.

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Table 1F82H chemical composition in mass%.

С	Si	Mn	Р	S	Cr	W	V
0.095	0.10	0.10	<0.005	0.0030	7.72	1.95	0.018
Ta	Ni	Mo	Ti	B	SolAl	N	Nb
0.040	<0.02	<0.01	0.005	0.00016	<0.001	0.01	0.0001





The HIP samples were impinged with Nd:YAG laser at different laser fluences to determine the critical energy that would result in the W-coating failure. The samples were cross-sectioned at the locations of impingement and were inspected by an optical microscope to determine the coating failure type. Table 2 shows a summary of failure types corresponding to different applied laser fluences. The first indication of failure occurs in the form of small cracks nucleating at the interface. These cracks first appeared when the sample was impinged with a laser fluence of 188.0 kJ/m². Thus, this laser fluence is the critical fluence of failure.

At higher laser fluences (>220.0 kJ/m²), severe damage in the form of delamination at the interface of the W-coating is observed. Fig. 2 shows the optical micrograph of the cross-section at the failure locus of an HIP sample impinged with a laser fluence of 241.6 kJ/m².

Fig. 3 shows the analytical solutions of the input stress pulse profile applied at the bottom of the F82H substrate and the stress profile at the W/F82H interface for the critical laser fluence (188.0 kJ/m^2). The input stress was calculated by measuring the free surface velocity of the F82H substrate using an optical interferometer with a resolution of 0.2 ns. This input stress is used in a one dimensional elastodynamic model to obtain the failure strength of the coating. For more details about the analytical analysis the reader is referred to Ref. [1–4]. The interface strength of HIP W/F82H samples (i.e. the maximum tensile stress at the critical laser fluence) is calculated to be 890 MPa. It should be noted that the observed interface failure, which happens at the maximum

Table 2	
Failure types of HIP W-coating sample corresponding to different laser fluences.	

Laser fluence (kJ/m ²)	86.7	150.7	188.0	223.1	241.6
Type of	No	No	Some crack at	Severe	Severe
failure	failure	failure	W/F82H interface	damage	damage



Fig. 2. (a) Optical micrographs of the cross-section of the failure locus of a HIP sample impinged with a laser fluence of 241.6 kJ/m^2 . Delamination at the W/F82H interface is observed. (b) The left side of the failure locus showing cracking at the interface and (c) the right side of failure locus showing the delamination as well as the cracking at the interface.

amplitude of the stress profile, is caused by the second reflected tensile wave from the W-coating free surface. This maximum amplitude appears 19 ns after the arrival of the first tensile wave reflected from the W-coating free surface.

The VPS samples were also impinged with the Nd:YAG laser at different laser fluences to determine the critical energy that would result in the W-coating failure. The samples were cross-sectioned at the locations of impingement and were inspected by SEM micrograph to determine the failure type. Delamination and cracking were observed in the W-coating at 30 μ m from the interface, while no damage or crack nucleation was observed at the W/F82H interface. These are unique phenomena observed in the VPS coatings and not in the HIP samples. First indication of failure in the VPS W-coatings, characterized by observing small cracks nucleating



Fig. 3. Analytical solutions for the input stress pulse profile applied at the bottom of the F82H substrate and the stress profile at the W/F82H interface for the critical laser fluence (188.0 kJ/m^2).



Fig. 4. (a) SEM micrographs of the cross-section of the failure locus of a VPS W/ F82H sample impinged with a laser fluence of 76.0 kJ/m^2 . Magnified SEM micrograph (b) in the left side, and (c) in the middle of the delaminated area.

in the coatings, was at a critical laser fluence of 28.5 kJ/m^2 . At higher laser fluences, severe damage in the form of delamination in the W-coating as well as partial removal of the coating is observed. SEM micrograph of the cross-section at the failure locus of a VPS sample impinged by a laser fluence of 76.0 kJ/m² is shown in Fig. 4.

For the VPS samples, the stress profiles of the input pulse and the analytical solutions at the interface and at the locus of failure at the critical laser fluence of 28.5 kJ/m^2 are shown in Fig. 5. From these results, the failure strength of VPS W-coating was calculated to be 496 MPa. It is observed that this is only 56% of the interface strength of HIP W/F82H and 33% of the ultimate strength of bulk tungsten reported in Ref. [5]. Kim et al. [1] showed that this



Fig. 5. The analytical solution of the VPS sample input stress pulse profile and the stress history at the interface and at the locus of failure at the critical laser fluence of 28.5 kJ/m^2 [1].



Fig. 6. The stress pulse profiles at the failure locus of both HIP and VPS samples at their critical laser fluences.

low failure strength of VPS W-coatings is mainly due to high porosity.

4. Conclusions

The failure strength of HIP and VPS W-coatings on F82H substrates were evaluated using the LS technique. Fig. 6 shows the stress pulse profiles at the failure locus for the HIP and VPS W/ F82H samples at their critical fluences of 188.0 kJ/m² and 28.5 kJ/ m², respectively. It is concluded that the HIP process ensures a good adhesion for the W/F82H interface. The calculated interface failure strength is reported to be 890 MPa. On the other hand, the VPS process results in a relatively lower failure strength of 496 MPa due to the high porosity in the W-coating itself.

Although HIP W-coatings have higher failure strength than VPS counterparts, the adhesion strength of tungsten to F82H ferritic steel is not the only factor that should be considered in the selection of the appropriate coating process for the first wall armor. As a matter of fact, the high helium implantation and retention in the first wall could result in an unacceptable material loss rate [7]. Thus, VPS W-coatings might be advantageous over HIP W-coatings due to its sub-micron porosities that facilitate large helium recycling rates.

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