

FAILURE STRENGTH MEASUREMENTS OF VPS TUNGSTEN COATINGS FOR HAPL FIRST WALL ARMOR

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The High Average Power Laser (HAPL) project is pursuing development of an IFE power reactor using a solid first wall chamber. Tungsten has been chosen as the primary candidate armor material protecting the low activation ferritic steel chamber wall structure. The tungsten armor is less than 1-mm thick and is applied by vacuum plasma spraying (VPS). The failure strength of the tungsten-armor is critical, which is measured using a state-of-the-art spallation technology developed at UCLA. A nano-second laser is used to propagate a compression/tension stress wave through the composite layered structure. The tensile strength in the coating is then related to the displacement velocity of the free surface of the tungsten coating. VPS tungsten coated steel samples were tested using the laser spallation technique and coating strengths were evaluated and are reported.

I. INTRODUCTION

The High Average Power Laser (HAPL) project is pursuing development of an Inertial Fusion Energy (IFE) power reactor using a solid first wall chamber. Tungsten has been chosen as the primary candidate armor material protecting the low activation ferritic steel chamber wall structure for its high melting temperature and low thermal erosion. The tungsten armor is less than 1-mm thick and is applied by vacuum plasma spraying (VPS). This will result in nano-porous structure that would allow helium to migrate to the surface eliminating any helium build-up that will result in the armor failure.

Failure strengths of coatings are typically measured by pulling on coatings until failure. These types of methods introduce a significant number of uncertainties regarding the accuracy of the resultant interface bond strength. The least of the problem is the introduction of a third material, the “glue”, used to attach coatings to the pulling mechanism. A state-of-the-art technology, the Laser Spallation Technique (LST), has been developed at UCLA, to relate the failure strength to the displacement velocity of a coating exposed to compression/tension stress waves propagated through the substrate using nano-second lasers.¹⁻³ In this technique, a compressive stress pulse is generated on the back side of the substrate disc by exfoliating a constrained metallic film via the impingement of an Nd:YAG laser pulse. Figure 1 shows a

schematic of the interface bond strength measurement set up. This compressive stress pulse propagates through the substrate and reflects into a tensile wave from the free surface of the coating that is deposited on its front surface. The returning tensile pulse pries off the interface if its amplitude is sufficiently high.

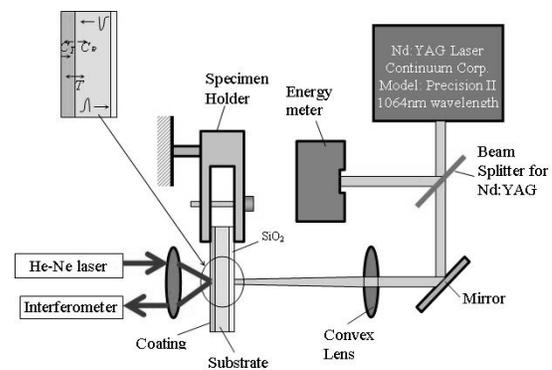


Fig. 1. Basic laser spallation technique set up used to determine the failure strength of thin coatings.

The stress is determined by recording the coating's free-surface velocities using a laser displacement interferometer. Because of the short rise time of the stress pulse, an interfacial region of approximately 70 to 150 micrometers is stressed uniformly. This results in the failure of the weakest link in the region which is spanned by the coating, interface and the substrate material. In addition, such a short pulse is able to invoke a rather local response from the interface such that minute structural and chemical changes are directly reflected in the measured strengths. These aspects of the technique have been demonstrated on a variety of metal/ceramic, metal/polymer and polymer/semiconductor interfaces prepared by using a variety of deposition techniques, including the thermal spray process.

II. EXPERIMENTAL SET UP

II.A. Overall Procedure

A 6-ns long Nd:YAG laser pulse is made to impinge over a $\phi 3$ mm area on the back surface of the ferritic steel substrate which is confined by a $15^{(\pm 0.5)}$ μm -thick

deposited SiO₂ layer (solid water glass). The melting-induced expansion of the ferritic steel under confinement of SiO₂ generates a compressive stress pulse that propagates towards the bonding interface. This compressive stress wave reflects as a tensile stress wave from the free surface of the coating and leads to its spallation at a sufficiently high amplitude. This critical tensile stress, causing failure of the coating, is obtained by measuring the transient displacement history of the coating's free surface (induced during pulse reflection) by using an optical interferometer with a resolution of 0.2 ns in the single shot mode.^{2,3} Figure 2 shows a typical recorded photodiode voltage output (fringe) corresponding to the steel substrate's free surface obtained by the Doppler displacement interferometer and the velocity record obtained at a laser fluence of 213kJ/m². The photodiode output voltage amplitude $A_0(t)$ recorded by the digitizer can be expressed in terms of free surface displacement $u_0(t)$

$$A_0(t) = \frac{(A_{max} + A_{min})}{2} + \frac{(A_{max} - A_{min})}{2} \sin\left[\frac{4\pi}{\lambda_0} \cdot u_0(t) + \delta\right] \quad (1)$$

where t is the time, the A_{max} and A_{min} are the maximum and minimum fringe amplitudes, respectively, and δ is a phase angle in radian. Now, the free surface velocity can be calculated by differentiating the free surface displacement, $u_0(t)$. Furthermore, for a coating of density ρ and thickness h , the stress, σ , at any location, x , in the coating is calculated from the measured transient velocity $v(t)$ as

$$\sigma(x, t) = \frac{1}{2} \rho c \left[v\left(t + \frac{x}{c}\right) - v\left(t - \frac{x}{c}\right) \right] \quad (2)$$

where c is the longitudinal stress wave velocity in the coating. Therefore, the peak amplitude of the stress pulses was recorded as a function of the laser fluence. The generated compressive stress pulses at different laser fluences in the F82H substrate can be obtained and is shown in Fig. 3. The sharp rise time (5 ns) and duration (~100 ns) of the stress wave is notable, and so is the similarity in their profiles at different laser fluences. Finally, the measured compressive stress pulse is used as an input stress for a simple idealized one dimensional elastodynamic model to obtain the failure strength of the coating. For more details about the experimental procedure and the use of one dimensional elastodynamic solution the reader is referred to references 1 through 4.

II.B. Sample Preparation

The Vacuum Plasma Sprayed (VPS) samples were produced at Plasma Processes, Inc. (PPI) of Huntsville, Alabama. The ferritic steel substrate has dimensions 25.0 mm x 25.0 mm 5.0 mm and the dimensions of the free standing VPS W disc is 2mm x 12.7 mm diameter. The

composition of the coating is 99.95% W and the powder particle size is -45/+20 μm .

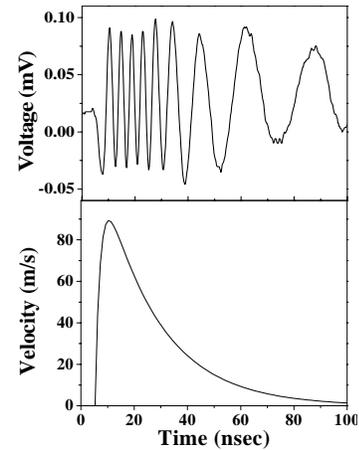


Fig. 2. (a) Photodiode voltage output (fringe) corresponding to the F82H's free surface obtained by Doppler displacement interferometer at a laser energy of 213 kJ/m². (b) The free surface velocity.

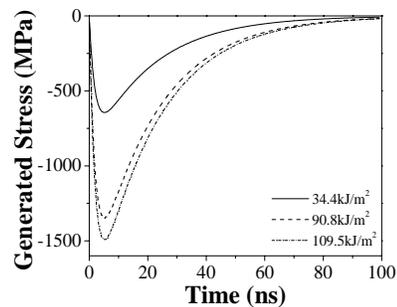


Fig. 3. Stress wave profiles as a function of increasing laser fluence generated in the F82H confined by a 15(±0.5) μm -thick solid water glass

The thickness of the deposited coating was about 100.0 – 150.0 μm . The bulk density of the W-coating is reported to be 80% and a pore size less than 200 nm were observed. It is noted that the characteristic dimensions of the pores are much smaller than the wave length of the propagating stress wave. Thus, the propagating wave sees a homogenous material. The same is true for the interface. The W-Coating was then polished at Oak Ridge National Laboratory (ORNL). The thickness of the specimen was measured at the center, using a dial indicator (± 0.0001 in.), to determine when polishing had removed 50 μm (0.002 in.) of material, thereby leaving approximately 60.0 – 70.0 μm of tungsten coating at the center. Due to curvature of the specimen, less material was removed at the corners leaving a slightly thicker tungsten coating there.

The elastic properties of the coating depend on the coating process. For comparison, since the plasma

spraying technique results in a lower density, ρ , and a lower Young's Modulus, E , the material properties, used in the following analysis, of the W-coating are assumed for two different conditions, namely bulk properties and 80% dense properties. The F82H and the W-coating properties used in the following analysis are shown in Table I.

TABLE I. Material Properties

Properties	F82H	W (Bulk)	80% Dense W-coating (VPS)
Young's Modulus (GPa)	217	410	54*
Poisson's ratio	0.29	0.29	0.1
Density (kg/m ³)	7870	19246	15397
Wave speed (km/s)	6.011	5.284	1.894

* Properties taken from reference 5 while all other properties are from www.fusionnet.seas.ucla.edu

III. RESULTS AND DISCUSSION

The sample was impinged with the laser beam at different locations and different laser fluences to determine the critical (threshold) energy that would result in the failure of the W-coating. Table II shows some of the laser fluences that the sample was impinged with and the type of failure observed.

TABLE II. Laser fluence dependent failure modes (or observations)

Laser Fluence (kJ/m ²)	23.4	25.6	28.5	38.4	75.0	110.4
Type of Failure	No Failure	No Failure	Cracking	Cracking and small Delamination	Cracking and Delamination	Partial removal of coating

The surface of the W-coating was first inspected by an optical microscope to determine any viewable failure. The sample was then cross-sectioned and inspected by scanning electron microscope (SEM) to investigate the types of failure and determine if there is any interlayer failure in the W-coating that may not be observed using the optical microscope. Two types of viewable failure were observed, using the optical microscope, on the W-coating surface. Figure 4 shows the W/F82H sample used in the current analysis and the two viewable types of failure.

The first viewable failure is a partial removal of the W-coating for laser fluences greater than 85.0 kJ/m². Figure 5 shows the SEM micrograph of the cross-section at the failure locus when impinged by a laser fluence of 110.4 kJ/m². The partial removal of about 60% of the

thickness of the W-coating is clearly observed. In Fig. 6-a the micrograph of the partial removed W-coating as viewed from the tungsten side of the specimen shows features that suggest a low ductile failure within the W-coating itself. Consequently, the requirement for failure or non-failure at a particular location is probably an abrupt transition.

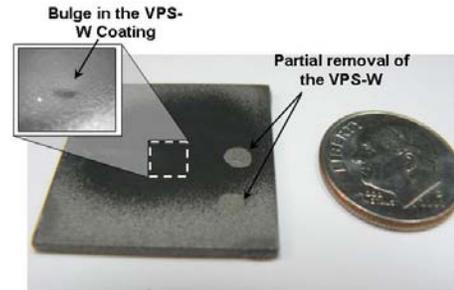


Fig. 4. Optical microscopic images of the failure locus and types of failures (bulged or complete removal).

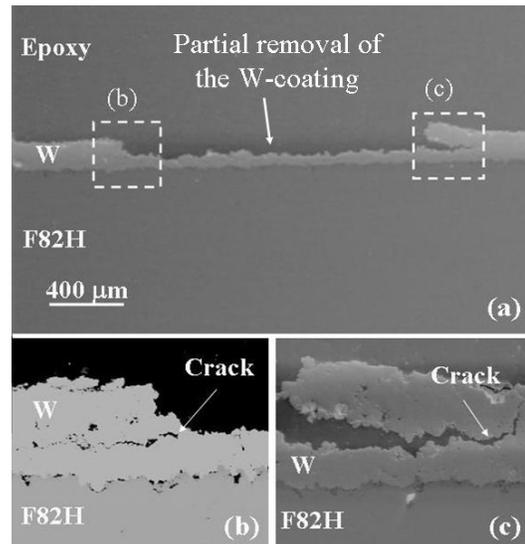


Fig. 5. (a) SEM micrograph of the failed region, at a laser fluence of 110.4 kJ/m², showing the partial dislodged W-coating. (b) Backscatter image of the left corner of the removed W-coat, and (c) SEM micrograph of the right corner of the removed W-coat.

The second type of viewable failure is an observed bulge on the surface of the W-coating. This is the dominant type of failure when the sample is impinged with laser fluences in the range of 35.0 to 85.0 kJ/m². Fig 7 shows the SEM micrograph of the cross-section at the failure locus when impinged by a laser fluence of 76.0 kJ/m². The W-coating is seen to crack and delaminate, 30.0^(±2.0) μm from the W/F82H interface, resulting in an observed bulge on the surface. It is also observed, from

the magnified backscatter image (Fig 7-c), that there is a high density of porosities in the W-coating.

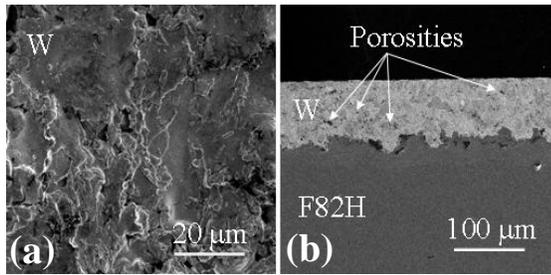


Fig. 6. (a) Surface micrograph of the partial dislodged W-coating for a laser fluence of 110.4 kJ/m²; (b) micrograph of the W-coating indicating pores in the coating near a location that has not failed.

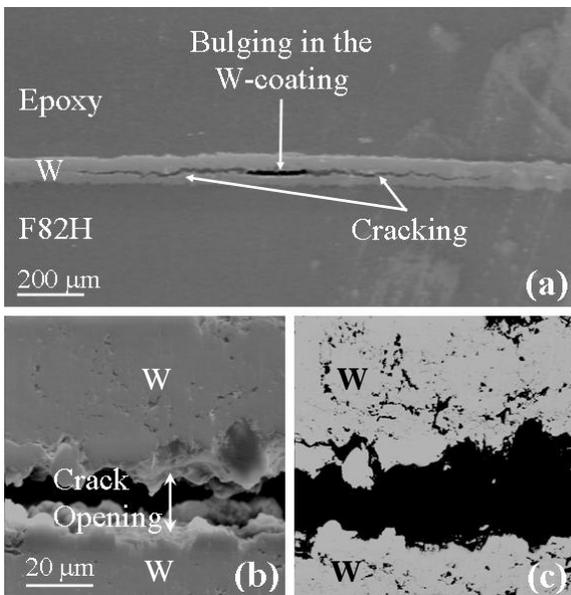


Fig. 7. (a) SEM micrograph showing of failure, at a laser fluence of 76.0 kJ/m²; note bulging in the W-coating; (b) magnified SEM micrograph of the bulge center; (c) backscatter image showing pores inside the W-coating at the center of the bulged area.

By decreasing the applied stress fluence below 35.0 kJ/m², it becomes impossible to observe any failure in the W-coating using the optical microscope and we rely on SEM to identify any failure in the coating. It is observed that for laser fluences in the range of 28.5 to 35.0 kJ/m² cracks form in the W-coating in a similar manner to what was discussed earlier but without bulging. Figure 8 shows the SEM micrograph of the failure locus, at laser fluence of 28.5kJ/m². It is observed in this case as well that the cracks nucleate 30.0^(±2.0) μm away from the interface. Below 28.5kJ/m², there is no observed cracking in the

coating and thus this is considered the threshold laser fluence.

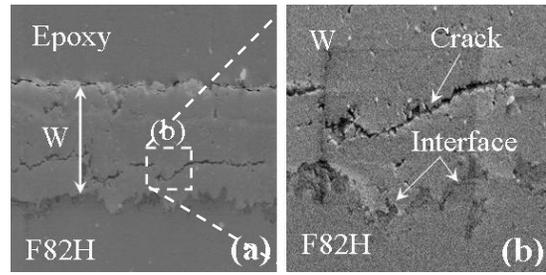


Fig. 8. SEM micrograph of a failed region, at the laser threshold fluence of 28.5kJ/m²; (a) magnification X40; (b) X400.

From the above discussion, it is apparent there is no interface failure observed and instead cracks nucleated then propagate within the W-coating itself creating an interlayer type of failure. This is due to existence of the high porosity density in the VPS W-coating. During the plasma spray coating process, the molten droplets of W-powder experience rapid deformation and solidification at the surface of the F82H substrate target and form disk-like splats. Because the feed stock of the plasma spray is nanosize the W coating is actually particles and not splats. As these splats solidify above each other they create porosities in between and thus weakening the coating as shown schematically in Fig. 9. Mostaghimi et al.⁶ noted that the shape of the splats is one of the key factors to determine the physical properties of the coating. Therefore, it is concluded that the pores and splat deteriorate the mechanical property of VPS W-coating and result in the cohesive failure of the coating as seen in Figs 4 through 8. Also, it is worth to note that most failures (cracking) appear about 30.0^(±2.0) μm away from the interface. This is the location where the reflected tensile wave restores its tension enough to break the coating.

Figure 10 shows the analytical solutions of the stress pulse profile of the VPS W sample at the threshold laser fluence of 28.5kJ/m² for 80% dense coating properties. The shown profiles are the stress pulse profiles at the interface, failure locus (30μm off from the interface toward W coating), and the bottom of the F82H substrate. The failure strength of VPS W coating (the maximum tensile stress at the threshold laser fluence) for this case is seen to be equal 484 MPa. Note that the second peak of the profile represents another reflection from the W/F82H interface.

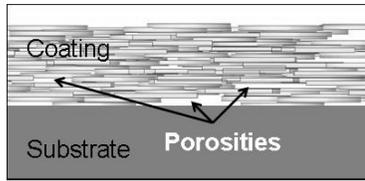


Fig. 9. Schematic illustration of a plasma spray coating showing porosities.

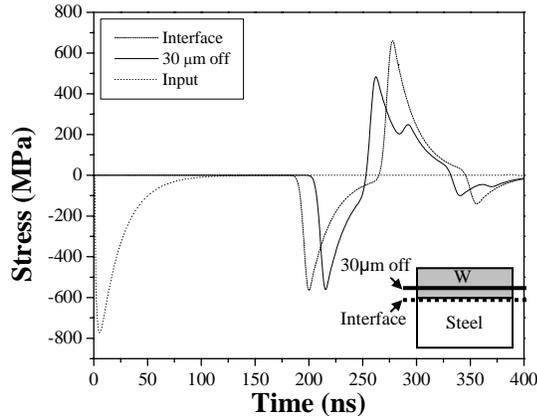


Fig. 10. Analytical solutions of stress pulse profiles at different locations in the sample at a threshold laser fluence of 28.5 kJ/m².

Fig 11 shows the stress hysteresis at the failure locus based on two types of different material properties (80% dense and 100% dense W coating). The case of the 100% dense properties was used to predict an approximated upper bound to the failure strength. The use of the 80% dense properties (which was based on microstructure analysis of the samples)⁵ is the more accurate calculations and gives more realistic failure strength. By changing the material properties of the coating with those of pure W, the maximum tensile stress increases by about 11%. Therefore, it can be concluded that the failure strength of VPS W coating is in the range of 484 to 536MPa. For the accurate calculation of failure strength, it is recommended to experimentally measure the material properties of VPS W considering the amount of porosity, thermophysical properties of the impacting particles, as well as the topology of the substrate.

II. CONCLUSIONS

The Laser Spallation Technique (LST) is advantageous over other test methods in the calculation of the failure strength of coatings due to its relatively simple test setup and the absence of plastic effects when performing the failure analysis. A number of W/F82H samples were tested using the LST experimental set up and it was observed that all the samples showed a cohesive failure occurring within VPS W-coating. As

calculated, based on the failure strength is in the range 484MPa to 536 MPa.

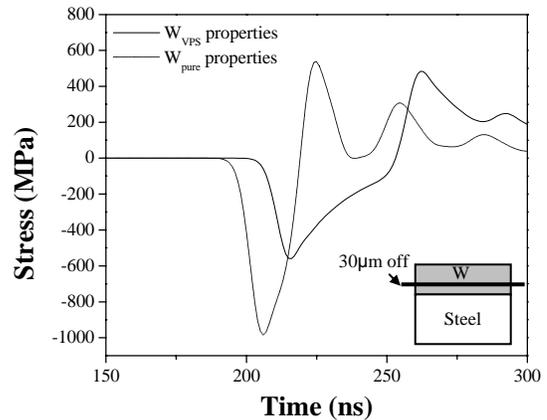


Fig. 11. Analytical solutions of stress pulse profiles at the fracture locus using different material properties of W.

It is interesting to note that Boustie et al.⁷ reported that the adhesion strengths of plasma sprayed tungsten carbide coatings from superalloy substrates to be in the range of 1.0 to 1.3 GPa for short laser pulses (1 and 3ns). However, in the current study, no interfacial delamination of the W-coating was observed. Instead the failure was observed to occur within the coating itself.

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