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Title: MODELING AND TESTING OF THE INTERFACIAL
STRESS STATE OF A TUNGSTEN-CLAD COMPOSITE
USING PUSH-OUT TESTING

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Testing and Evaluation of the Interfacial Stress State of a Tungsten-Clad Composite Using Push-out Testing

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Abstract

To study the properties of a tungsten-clad diffusion bonded interface push-out tests were performed. This method involves pushing out a tungsten rod while measuring the applied load and relative displacement of the tungsten and cladding. Specimens were tungsten in either wrought, annealed, or single crystal form with a 316L stainless steel clad. In the analysis of stress state and its dependence on failure mode for push-out testing, two important parameters are examined: specimen thickness, and support hole size. The measured push-out loads coupled with the finite element modeling were used to yield information about the tungsten-clad bond strength. There is currently no information available on the interface properties of the composite materials under investigation. Therefore, a qualitative/quantitative means of measuring interface strength utilizing push-out testing supported by Finite Element Modeling (FEM) was developed.

Introduction

The Accelerator Production of Tritium (APT) program proposes to produce tritium via the reaction of ^3He gas with neutrons produced by an accelerator-driven proton beam impinging on a tungsten target. In the APT program, metal-clad tungsten tubes and rods are used as a target material to generate neutrons by spallation reactions with high-energy protons (1 GeV) from an accelerator. The APT target design requires tungsten to be clad with a metal for many reasons: to resist corrosion by the coolant, to improve heat transfer, and to contain tungsten in a loss of coolant accident. For heat to transfer efficiently from the tungsten to the cladding, it is imperative that complete bonding occurs and that the interface withstands thermal and dynamic (fatigue) stresses, along with radiation damage that occurs during operation. 316L stainless steel is under investigation as possible cladding materials because of its attractive thermal and mechanical properties, and corrosion resistance. Push-out testing was utilized to examine the role the interface plays in controlling the overall composite strength and toughness. From the data generated it will be possible to quantify interface bond strength and select the best composite materials for safe operation and optimum target life.

There is considerable research on push-out testing as a means to measure interfacial strength. The information to date uses small push-rods compared to the interface diameter and a larger-than-necessary support hole size. The effect of these test parameters is that in addition to placing the specimen in shear, a large tensile stress is imposed on the bottom of the push-out specimen leading to initiation of failure. The tensile stresses can alter the interfacial failure process such that rather than failure initiation by shear stress on the upper surface of the specimen in contact with the push-rod, failure can initiate by crack nucleation along the interface at the bottom surface of the specimen.

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Specimen Preparation

To simulate the actual target geometry, cylindrical specimens, consisting of a tungsten core with a cladding of 316L stainless steel were prepared. Bonding of the two materials was accomplished by Hot Isostatic Pressing (HIP) of the cladding to the tungsten rod. Minimizing the HIP temperature yields a small diffusion zone, avoids the formation of a brittle intermetallic phase, and produces minimal residual stress buildup at the interface during cooling due to the mismatch of CTE between the tungsten and cladding materials.

Three forms of tungsten were bonded to 316L stainless steel to study the effect of tungsten's material properties on the load displacement curve obtained in push-out testing of composite discs cut from HIP samples. Specimens included tungsten in either wrought, wrought/annealed at 1800 °C for one hour, or single crystal form with a 316L stainless steel clad. The HIP assembly consisted of a 0.125 inch diameter tungsten rod inserted into a 0.126 inch diameter hole in the clad material with end caps that were electron beam welded on in a vacuum. The specimens were then HIPed in a cycle that applied 29 ksi of inert gas pressure for 8 hours and reached a maximum temperature of 810 +/- 20 °C. Specimens of roughly 0.05 inch in thickness were cut from the 1 inch long HIPed rods. Specimens were then polished to a thickness of approximately 0.040 inch using a final medium of 1µm diamond.

Experimental Procedures

To study the mechanical properties of the tungsten-cladding bond, push-out tests were performed on 0.125 inch diameter tungsten rods containing a 0.125 inch thick cladding. The push-out apparatus was designed to place the interface of the composite sample in a state of shear stress to measure bond strength. The push-out fixture design incorporated a support plate that restricts the motion of the cladding material while allowing free motion of the inner tungsten core as seen in figure 1. The push-out test fixture was placed in a mechanical testing machine that applied a compressive load to the push-rod, which is in contact with the tungsten core. The test measures the combined resistance of the tungsten and tungsten/metal clad interface to the motion of the push-rod that is driven at a constant displacement rate of 0.005 inches per minute. The specimen is held by the support plate with a concentrically located hole that provides unrestrained tungsten motion. Pushout tests were performed using two identical support plates having different push-out hole diameters. The first support plate hole was 0.135 inch in diameter yielding a 0.005 inch tolerance on the radius of the tungsten. The second support plate hole is 0.130 inch in diameter yielding a 0.0025 inch tolerance on the radius of the tungsten. The punch was made of hardened tool steel with an end diameter of 0.121 inch. Specimens were aligned with respect to the tungsten center and machined to a diameter of 0.374 +/- 0.001inch. The tight tolerance between the push-rod and the support plate hole was designed to help reduce the tensile stresses on the exit surface of the specimen.

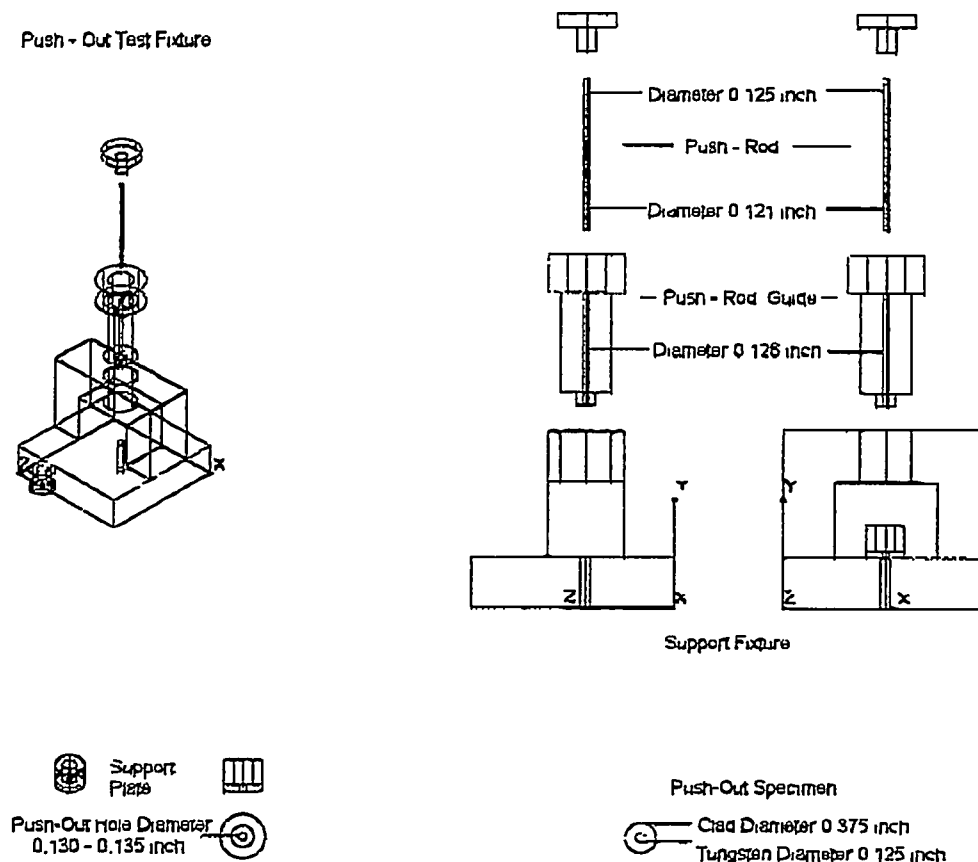


Figure 1. Push-Out fixture schematic and support plate used for testing tungsten-clad rods.

In addition to the information gathered on interface strength from the push-out testing, specimens were also characterized before testing by microhardness measurements on both tungsten and clad materials, energy dispersive x-ray spectroscopy (EDS) for elemental composition across the diffusion zone, and light microscopy for grain size. Scanning Electron Microscopy (SEM) was utilized on complete and partial push-out tests specimens to evaluate the failure mode.

Results

A SEM equipped with EDS capabilities was used to investigate the diffusion zone on tungsten/316L SS specimens uniaxially bonded at temperatures of 800°C and 950°C along with a push-out specimen consisting of annealed tungsten-clad 316L SS that were HIPed at 810°C. The diffusion data indicates that stainless steel elements (Fe, Ni, and Cr) diffuse 1-3 microns into the tungsten for both the uniaxial bond 800°C and the 810°C HIP push-out specimens. Diffusion data for the uniaxial 950°C specimen revealed measurable amounts of Fe, Ni, and Cr 7 μm into the tungsten. There was no evidence in any of the specimens of tungsten diffusing into the stainless steel.

After HIPing, polished and etched pushout specimens were analyzed with optical microscopy (Figure 2 (A, B, and C)). The wrought tungsten exhibits a grain size of 20 – 30 μm , while the grain size in the annealed tungsten varies between 20 to 300 μm . The

single crystal tungsten was analyzed using the Laue back-reflection technique showing a $\langle 111 \rangle$ orientation normal to the surface of the specimen. Micro-hardness examination on specimens before push-out testing yielded Vickers hardness values of $433.3 \pm 2.54 \text{ kg/mm}^2$ for single crystal tungsten, $399.8 \pm 14.44 \text{ kg/mm}^2$ for annealed tungsten, and $427.2 \pm 9.92 \text{ kg/mm}^2$ for wrought tungsten. As expected, the annealed tungsten exhibits the lowest hardness because of the large grain size and the lower dislocation densities from the annealing treatment. In addition, the annealed tungsten shows a large standard deviation because of its large variation in grain size.

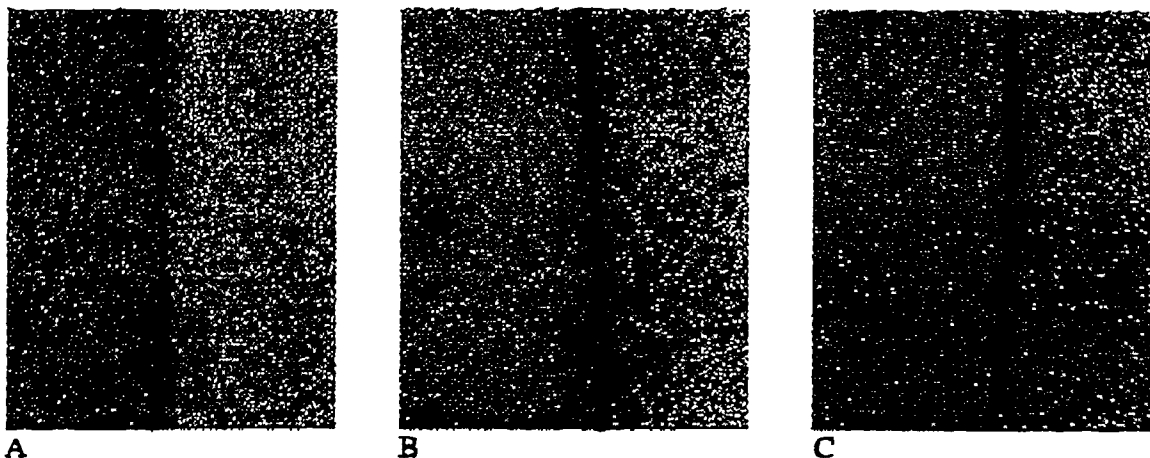


Figure 2 A, B, and C. Morphology of 316L SS clad tungsten rod push-out specimen at 500X. From left to right wrought tungsten / 316L SS with a grain size of 10-20 μm , annealed tungsten / 316L SS with a grain size of 20-300 μm , and single crystal / 316L SS.

Push-out stress/displacement curves were attained by measuring the load vs. displacement during testing and dividing the load by $(\pi * \text{diameter} * \text{thickness})$ to yield the interface stress. Testing was performed on 316L SS bonded to tungsten in the wrought, annealed, and single crystal form. Although load/displacement traces varied from specimen to specimen, there were several distinctive characteristics in the load/displacement traces of all push-out specimens. Tensile stresses induced cracking at the tungsten-cladding interface on the bottom surface of every specimen. These stresses altered the interfacial failure process such that rather than interfacial failure initiation near the upper surface of the specimen adjacent to the push-rod, failure initiated by crack nucleation within the tungsten on the bottom surface of the specimen. Yielding of the clad material was observed on both the top and bottom of every specimen tested. All push-out test specimen's stress vs. displacement curves roughly followed the curves produced by testing a pure 316L SS specimen with no tungsten core. The difference between the load/displacement curves for clad tungsten push-out specimens and that of the pure 316L SS was that for the clad tungsten specimen achieved a higher stress for the same displacement in the push-out specimens. The higher stress results from tungsten having a higher modulus of elasticity than the cladding material. The representation stress/displacement curve for the clad material alone can be seen as a reference on the push-out stress vs. displacement graphs.

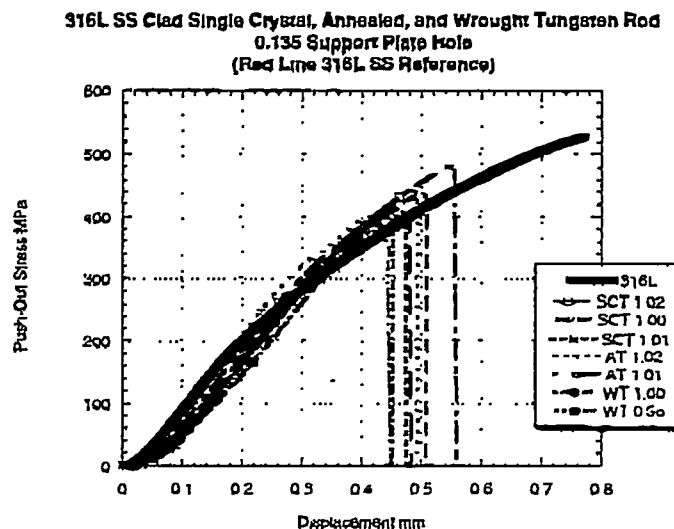


Figure 3. Push-out yield strength vs. displacement for 316L SS clad tungsten. The reference line in the graph indicates the stress vs displacement curve for a 316L specimen without a tungsten core. SCT, AT, and WT in the legend indicates the single crystal, annealed, and wrought tungsten forms respectively, followed by the specimen thickness in millimeters.

The SEM analysis of push-out tests on 316L SS clad wrought show transgranular brittle fractures on the bottom surface as seen in figure 4. 316L SS clad wrought tungsten reached a maximum stress of 406 MPa at a displacement of 0.45mm.

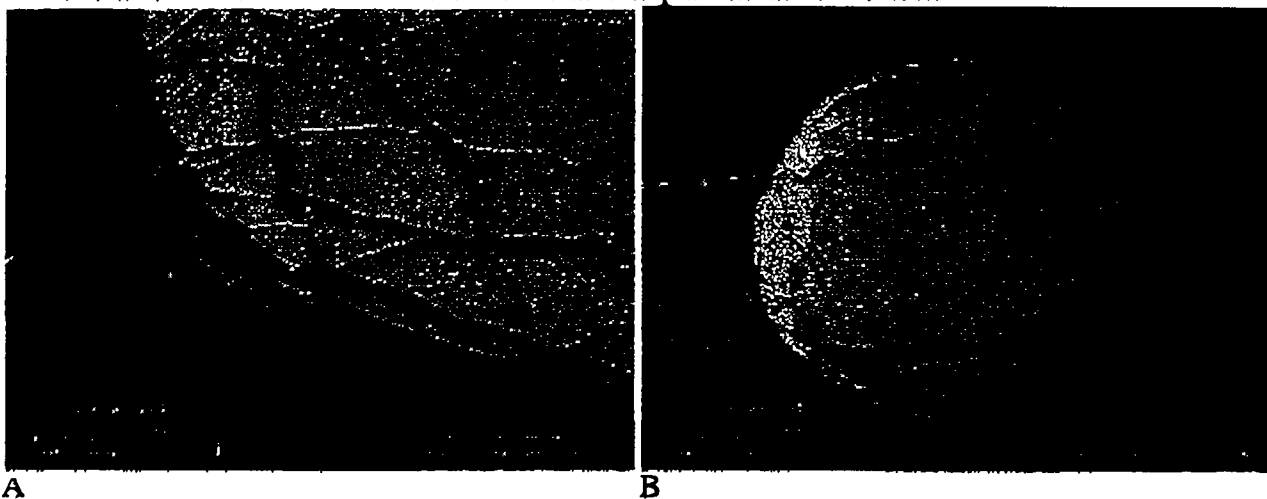


Figure 4 A and B. Transgranular fracture of wrought tungsten core after push-out testing.

The 316L SS clad annealed tungsten push-out tests revealed cracking along the grain boundaries after testing (figure 5). This behavior is probably a result of increased concentration of impurities that accumulated on the boundaries during heat treatment. The push-out stress for 316L SS clad annealed tungsten ranged from 411 to 480 MPa for displacements of 0.48 to 0.56mm.

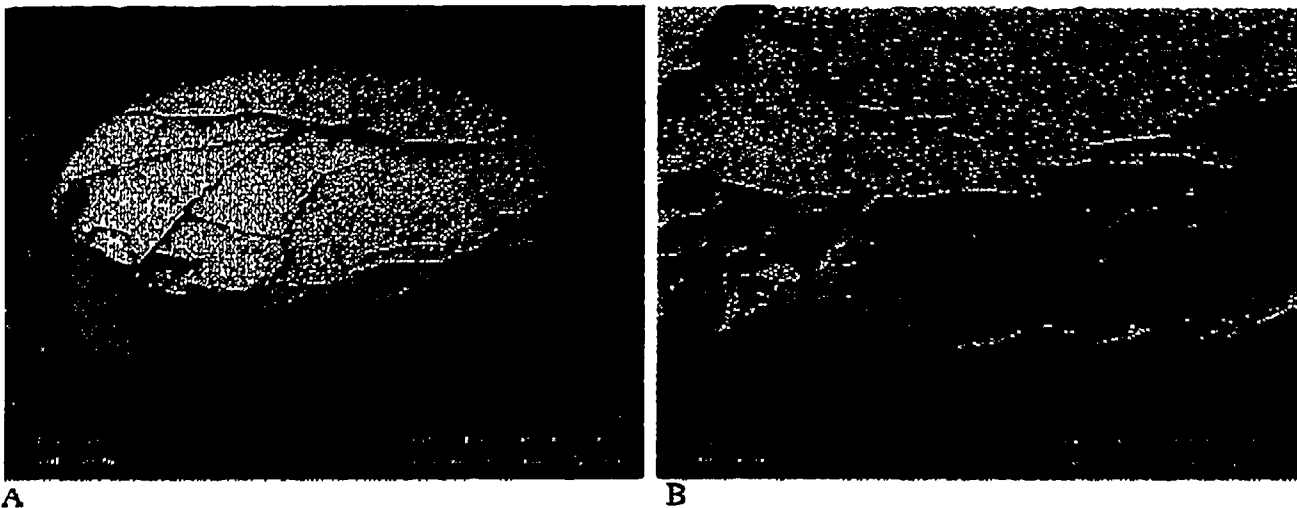


Figure 5 A and B. Intergranular fracture of annealed tungsten core after push-out testing.

The 316L SS clad single crystal tungsten push-out specimens showed cracking along the cleavage planes on the bottom surface after testing. Laue back reflection x-ray analysis revealed that the $\langle 111 \rangle$ crystallographic direction is parallel to the compression direction. Thus it is hypothesized that the cracking is occurring on the (110) close packed planes. Figure 6 shows this cracking on the bottom of the specimen in the single crystal tungsten proved to be at angles of 60 and 120° relative to one another. 316L SS clad single crystal tungsten reached stresses of 402 and 440MPa at a displacement of 0.49mm.

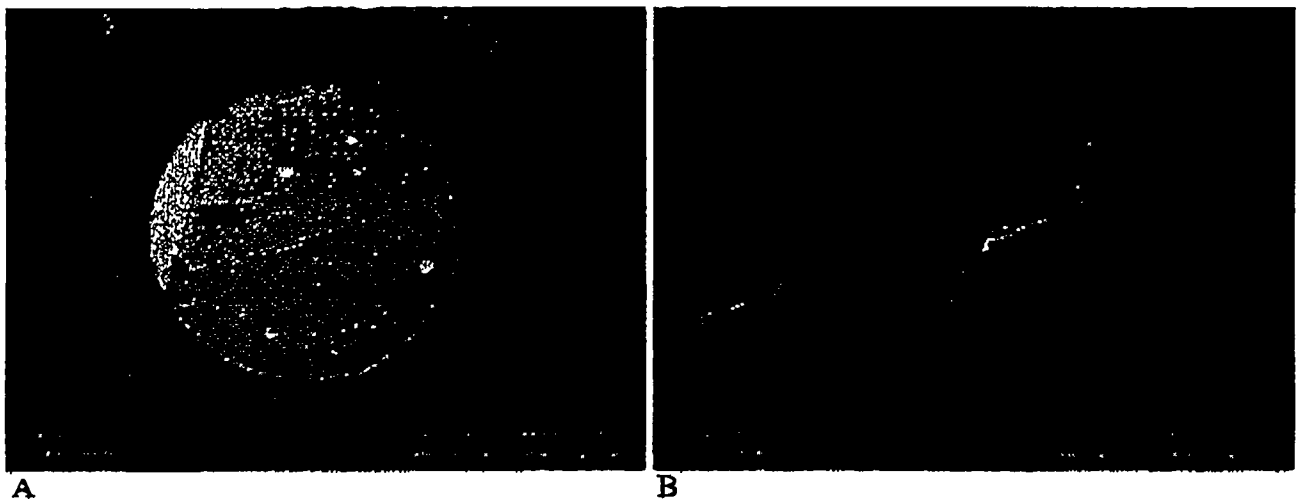
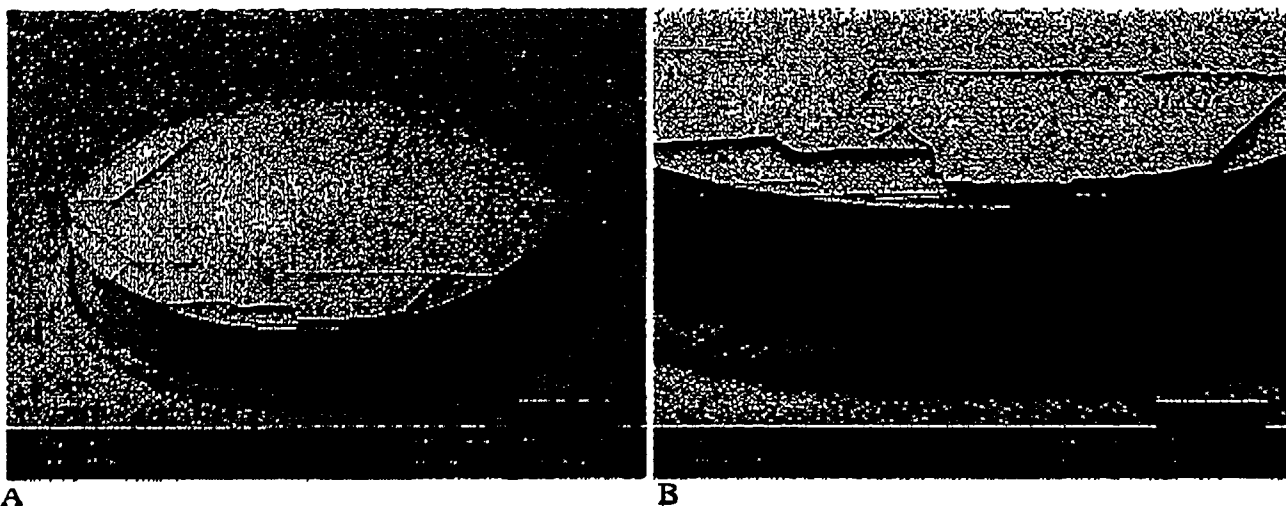


Figure 6 A and B. SEM images revealing cracking at 60° and 120° in single crystal tungsten on the bottom surface of a failed specimen.



Figures 7 A and B. Bottom surface of a 316L SS clad single crystal tungsten partial push-out specimen. Viewing cracking in the tungsten and yielding of the 316L SS clad material.

Partial push-out tests were performed on the three forms on clad tungsten rods to evaluate the failure process. In all partial push-out tests cracking was observed in the tungsten on the bottom surface of the specimen.

Conclusion

EDS of the diffusion zone revealed elements of stainless steel (Ni, Cr, Fe) diffusing 1-3 microns into the tungsten at 800°C increasing up to 7 microns for 950°C. After testing each specimen, it was observed using EDS and backscatter electron imaging mode on the SEM revealed the remains of tungsten on the clad material. The same is true for clad remains being visible on the tungsten core after pushed-out testing. The material remains on both portions of the push-out specimen leads to the conclusion that failure occurred at the interface.

The results showed consistent push-out stress values for both 0.135 and 0.130 inch push-out support hole diameter. Although a larger difference in support hole size will certainly affect the test results, the 0.005 inch difference between support hole diameters showed no noticeable affect.

316L SS clad single crystal tungsten had an average push-out stress of 421MPa. Failure on the bottom surface of the tungsten occurred by cracking on the (110) cleavage planes. Micro hardness testing of single crystal tungsten revealed a Vickers number of 433 with a standard deviation of 2.54.

316L SS clad annealed tungsten, with a grain size of 20-300 μm , showed an average push-out stress of 443MPa. Failure on the bottom surface of the tungsten occurred by intergranular cracking. Micro hardness testing of single crystal tungsten revealed a Vickers number of 399.8 with a standard deviation of 14.4.

316L SS clad wrought tungsten, with a grain size of 10-20 μm , had an average push-out stress of 392MPa. Failure on the bottom surface of the tungsten occurred by transgranular cracking. Micro hardness testing of single crystal tungsten revealed a Vickers number of 427 with a standard deviation of 9.92. The discrepancy between

hardness values for the three forms of tungsten resulted from the affect of variations in grain size between the tungsten specimens.