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Evaluation of the Hot Isostatic Pressed Diffusion Bond of a Clad-Tungsten Composite Using Push-Out Testing

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Abstract

A detailed investigation has been conducted to determine the optimum temperatures that would produce diffusion bonding of 316L stainless steel (SS) to tungsten by a hot isostatic pressing (HIP) method, and to evaluate the strength of bonded specimens fabricated by this method. Initial trial specimens of 316L SS bonded to tungsten by uniaxial hot pressing at 800°C and 950°C were examined by optical microscopy, scanning electron microscopy (SEM), and energy-dispersive x-ray analysis (EDS) to select optimum bonding temperatures for producing HIP-bonded 316L SS clad tungsten rod specimens for determining bond interface strength using a rod push-out test method. Push-out strength testing and complementary microhardness measurements, along with optical microscopy, SEM, EDS, and electron microprobe analysis were then performed on HIP-bonded 316L SS clad tungsten rods. Specimens were HIP-bonded at 810°C using 316L SS and three types of tungsten, including single crystal, wrought, and wrought/annealed tungsten. In addition, 316L SS clad wrought tungsten rods were HIP-bonded at two temperatures, 810°C and 900°C, to assess bonding temperature effects on bond strength. There was a ~50MPa difference between the maximum push-out stresses reached for all three forms of tungsten HIP-bonded at 810°C, indicating that tungsten grain size had little effect on push-out test results. There was a ~25% increase in push-out stress between specimens HIP-bonded at 900°C and those HIP-bonded at 810°C. HIP-bonded 316L SS clad wrought tungsten push-out test specimens were also irradiated in the Oak Ridge National Laboratory (ORNL) High Flux Isotope Reactor (HFIR) at 70°C to a neutron dose of ~1.5 dpa, and tested to investigate the potential for neutron damage of the diffusion bond; push-out test results indicated that the bond was not compromised. SEM analysis was also utilized on fully and partially tested push-out test specimens to evaluate the specimen failure mode. Results indicated that specimen failures were initiated by yielding of the 316L SS near the interface, followed by cracking in the tungsten and finally crack propagation into and failure at the bond interface.

Introduction

The Accelerator Production of Tritium (APT) program is currently developing the design for a system to produce tritium via the reaction of ^{3}He gas with neutrons produced by an accelerator-driven proton beam impinging on a tungsten target. In the APT facility, metal-clad tungsten tubes and rods will be used as target components to generate neutrons in the tungsten by spallation reactions with high-energy protons (1GeV) from an accelerator. The APT target design requires tungsten (the spallation neutron source) to be clad with a metal for many reasons: to prevent corrosion of the tungsten by the water coolant, to improve heat transfer from the tungsten to the cooling water, and to contain tungsten (tungsten oxide vapor) in case of a loss of coolant accident. For heat to transfer efficiently from the tungsten through the cladding to the coolant, it is imperative that the metal cladding be intimately bonded to the tungsten and that the bond interface withstand thermal and dynamic (fatigue) stresses, along with any irradiation damage that may occur during operation. The alloy 316L SS has been selected as a candidate cladding material because of its attractive thermal and mechanical properties, and corrosion resistance in water. A push-out test method has been developed and tests conducted on 316L SS clad tungsten rod specimens to examine the role the bond interface plays in controlling the overall composite strength. The goal of the current effort is to quantify the 316L SS clad tungsten interface bond strength and select the best composite materials and optimum bonding conditions for achieving safe target operation and maximum target life.

Specimen Preparation

To determine appropriate diffusion bonding temperatures for fabrication of the bond interface strength test specimens (push-out specimens), trial specimens of 316L SS were bonded to wrought tungsten by uniaxial vacuum hot pressing at temperatures of 800°C and 950°C. These temperatures were selected based on diffusion calculations performed using readily-available interdiffusion data for nickel and tungsten [1]. The uniaxially-bonded specimens consisted of alternating thin sheets of stainless steel and tungsten hot pressed for one hour at temperature under a pressure of 68MPa. The specimens were then mounted and polished using a final medium of 1μm diamond paste, and examined by optical microscopy to evaluate the bond interface. A SEM equipped with EDS capabilities was also used to measure the width of the diffusion zones developed in the bonded specimens. The results of this study were used to select bonding temperatures for fabrication of push-out test specimens for assessing the strength of the diffusion bond.

To simulate the actual APT target geometry, cylindrical specimens, consisting of a tungsten rod (core) clad with 316L SS were prepared. Bonding of the two materials was accomplished by HIP of the cladding to the tungsten rods. The goal of the HIP-bonding effort was to minimize the HIP temperature which still produce significant diffusion between the two materials, i.e., a reasonable diffusion zone, avoid the formation of potentially brittle intermetallic phases, and produce minimal residual stress buildup at the interface during cooling due to the coefficient of thermal expansion (CTE) mismatch between the tungsten and cladding material which is approximately 3:1.

Three types of tungsten rod materials were bonded to 316L SS to study the effect of tungsten microstructure and material properties on load-displacement curves obtained from push-out testing of composite discs cut from HIP-bonded samples. Specimens included tungsten in wrought, wrought/annealed at 1800°C for 15 hours, and single crystal forms, with a 316L SS clad. The HIP assemblies consisted of a 3.175mm diameter x 25.4mm long tungsten rods inserted into 9.525mm O.D. x 3.20mm I.D. 316L SS tubes fitted with electron beam-welded end caps. These specimens were HIP-bonded in argon at 810 +/-20 °C using a cycle which included increasing pressure and temperature over a several hour period up to the bonding condition (200kpa pressure at 810°C), holding both pressure and temperature constant for 1 hour, and then cooling under pressure to room temperature. Specimens of 316L SS clad wrought tungsten were similarly prepared by HIP-bonding at two different temperatures, 810+/-20°C and 900+/-

20°C, for bonding temperature effects studies. Push-out test specimens, approximately 1.27mm in thickness, were cut from the 25.4mm long HIP-bonded rods, and then ground and final polished to a thickness of ~1 mm using a final medium of 1μm diamond paste. In addition to testing unirradiated specimens, polished ~1mm-thick specimens of 316L SS clad wrought tungsten HIP-bonded at 810°C were neutron irradiated at the ORNL in the HFIR at 70°C to a neutron dose of ~1.5 dpa.

Experimental Procedures

To study the mechanical properties of the tungsten-cladding bond, push-out tests were performed on ~1mm thick specimens prepared as described above. The push-out test method was developed based on a fiber push-out test method previous used to assess fiber/matrix interface strengths in fiber-reinforced composites [2, 3]. The push-out fixture design (Figure 1) incorporated a support plate that prevented motion of the cladding material while allowing loading and free motion of the inner tungsten core, placing the 316L SS clad tungsten interface in a nearly-pure state of shear to measure bond strength. The push-out test fixture was placed in a mechanical testing machine that allowed the application of a compressive load to a push-rod, placed in contact with the tungsten core. The test measured the combined resistance of the tungsten and 316L SS clad tungsten interface to the motion of the push-rod, which was loaded at a constant displacement rate of 0.127mm per minute. The specimen was held by a support plate containing a concentrically located hole that provided unrestrained tungsten motion. The support plate hole was 3.429mm in diameter, providing a 0.127mm radial clearance between the tungsten rod and support plate hole. The push-rod was made of hardened tool steel, with an end diameter of 3.07mm. The outer cladding of each specimens was machined axisymmetric with respect to the center of the tungsten rod to a diameter of 9.525 +/- 0.025mm. The tight clearance between the push-rod and the support plate hole was designed to help minimize the tensile stresses on the exit surface of the specimen during testing [4].

Compliance testing was performed to measure the elastic response of both the push-out fixture and Instron compression testing machine to obtain load-displacement measurements for post-test correction of push-out specimen load-deflection curves for machine/fixture effects. Compliance testing consisted of replacing the support plate with a 10mm thick hardened steel slug (hardness of 434kg/mm^2). The push-out fixture was then placed into the Instron and the load was increased to 12700N. The maximum compliance load used was selected as the maximum force that the push-rod had experienced during push-out testing. The load was then released and the steel slug reloaded to account for any settling in, or deformation, of the steel slug that might have occurred during the initial loading. The load-displacement data obtained from the second loading were fit to a third order polynomial equation which was then used to correct for elastic machine and fixture displacement at any given load in order to yield true push-out specimen load-displacement curves.

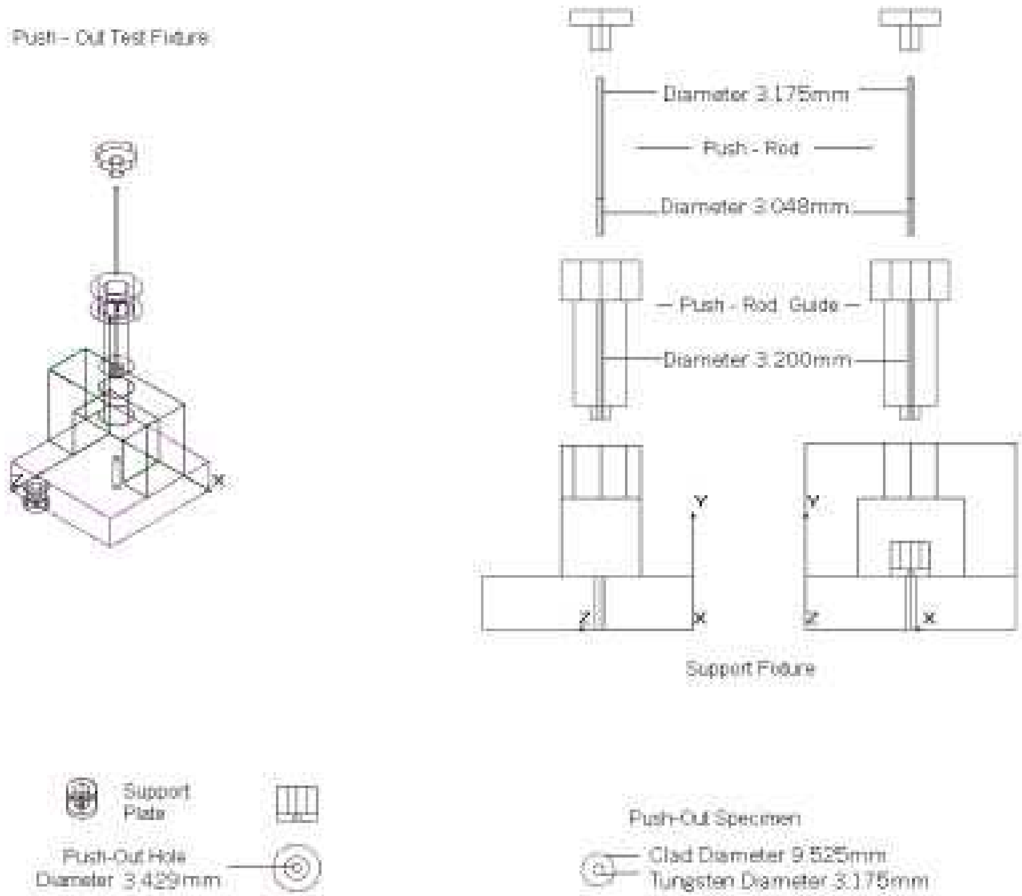


Figure 1. Schematic of push-out fixture and support plate used for testing 316L SS clad tungsten rod specimens.

In addition to the information obtained on interface strengths from push-out testing, specimens were characterized prior to testing utilizing microhardness measurements on the tungsten to assess the relative strengths of the test materials, optical microscopy to determine grain size, assess other microstructural features, and verify diffusion bond development, and electron microprobe analysis to determine elemental compositions across diffusion zones. X-ray diffraction measurements using the Laue back-reflection technique were used to determine the orientation of the single crystal rod material. SEM analysis was also utilized to assess diffusion bond development and to examine fully-tested and partially-tested push-out test specimens to evaluate specimen failure modes.

Results and Discussion

Materials Characterization:

An SEM equipped with EDS capabilities was used to measure the width of the diffusion zone for 316L SS clad tungsten specimens uniaxially bonded at trial temperatures of 800°C and 950°C. The EDS data indicated that stainless steel alloying elements (Fe, Ni, and Cr) diffused ~1-3µm into the tungsten for the 800°C uniaxially bonded specimen, and up to ~7µm into the tungsten for the 950°C uniaxially bonded specimen. However, only limited diffusion of tungsten into the 316L SS was observed. Utilizing the backscattered electron imaging mode on the SEM, a second phase was observed at the interface between the 316L SS and tungsten uniaxially bonded at 950°C that was not visible in the 800°C specimen. Based on these results, 810°C and 900°C HIP-bonding temperatures were selected which were expected to produce sufficient bonding between the 316L SS and tungsten while minimizing the formation of a second, potentially-brittle intermetallic phase.

Elemental diffusion profiles were also obtained by electron microprobe analysis of the interfaces in 316L SS clad tungsten push-out specimens. Analysis of 810°C HIP-bonded specimen revealed elements of 316L SS (Fe, Ni, Cr, and Mn) had diffused ~3-4μm into the tungsten. For the same 810°C HIP-bonded specimen, tungsten was observed to diffuse ~2μm into the 316L SS, thus yielding a total diffusion zone for the 810°C HIP-bonded specimen of ~5-6μm. The elemental diffusion profile for a 316L SS clad wrought tungsten push-out specimen HIP-bonded at 900°C revealed 316L SS elements diffusing ~3-4μm into the tungsten. The microprobe analysis of the diffusion of tungsten into the 316L SS shows measurable amounts of tungsten at ~6μm into the 316L SS for the 900°C HIP-bonded specimen, making the width of the diffusion zone in this specimen ~9-10μm.

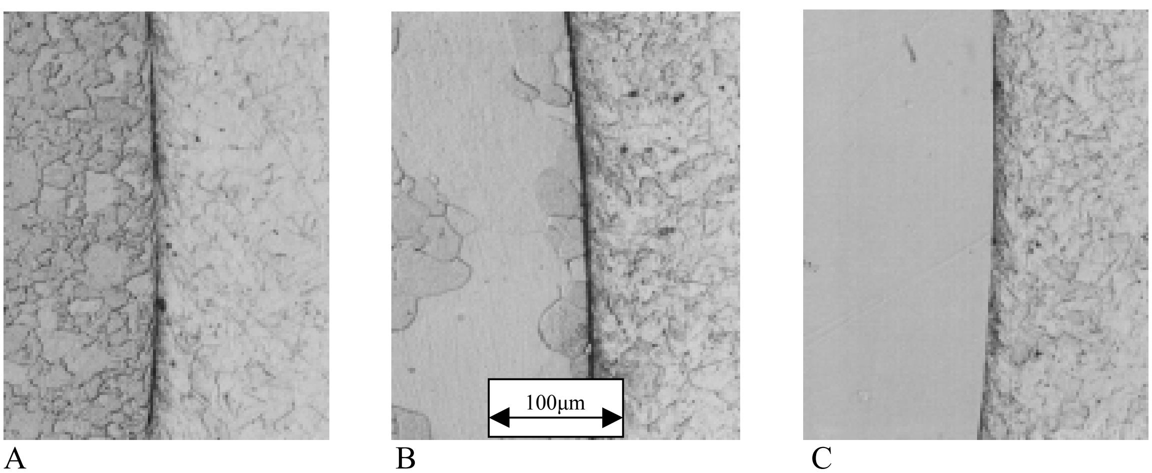


Figure 2. Microstructures of 316L SS clad tungsten rod interfaces in push-out specimens HIP-bonded at 810°C. (A) wrought tungsten (on left)/316L SS (tungsten grain size of 10-20 μm), (B) annealed tungsten (on left)/316L SS (tungsten grain size of 20-300 μm), and (C) single crystal (on left)/316L SS.

Polished and etched push-out specimens fabricated using the three tungsten rod material types were examined using optical microscopy (Figure 2A, 2B, and 2C). The wrought tungsten exhibited a grain size of 20 to 30μm, while the grain size in the annealed tungsten varied between 20 to 300μm. The orientation of the single crystal tungsten was determine using the Laue back-reflection x-ray technique. Analysis of the x-ray pattern indicated that the axis of the single crystal rod had a <111> orientation normal to the surface of the specimen. Microhardness measurements on un-tested push-out specimens HIP-bonded at 810°C yielded Vickers hardness values of 433.3 ±2.54kg/mm^2 for single crystal tungsten, 399.8 ±14.44kg/mm^2 for the wrought/annealed tungsten, and 427.2 ±9.92kg/mm^2 for wrought tungsten. As expected, the wrought/annealed tungsten exhibited a lower hardness than the wrought tungsten because of the larger grain size and the elimination of the wrought (hot worked) microstructure (lower dislocation density) resulting from the annealing treatment. In addition, the wrought/annealed tungsten exhibited a larger variation in grain size than the wrought tungsten and yielded a larger standard deviation in the microhardness measurements. The microhardness of the single crystal tungsten was similar to that for the wrought tungsten. Microhardness measurements on push-out specimens HIP-bonded at 900°C revealed a Vickers hardness values of 419.5 ±10.6 kg/mm^2 for the wrought tungsten.

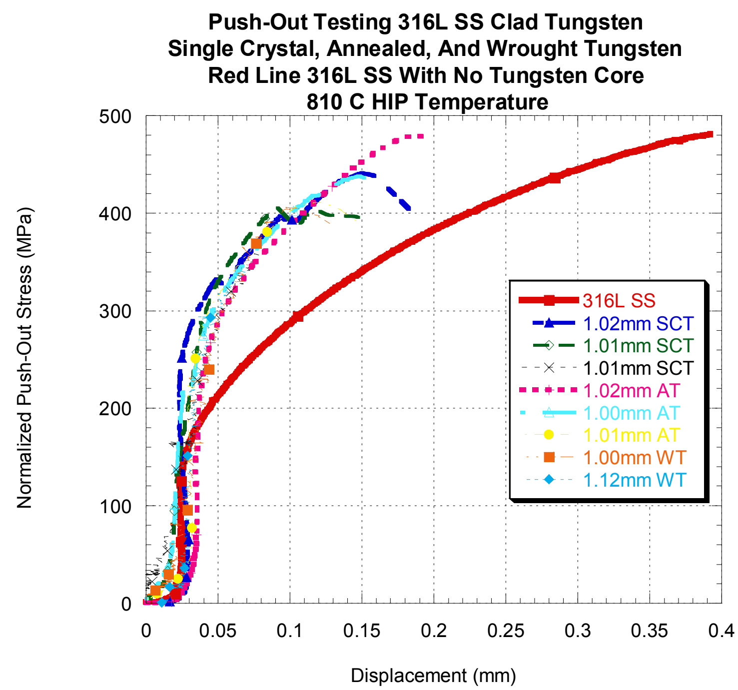
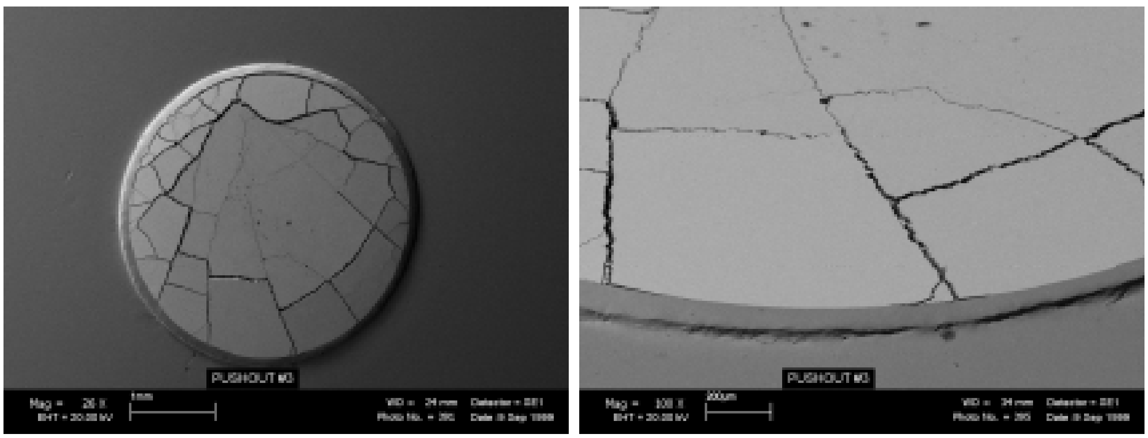


Figure 3. Push-out stress (interface area-normalized load) vs. displacement curves for 810°C HIP-bonded 316L SS clad tungsten rod material types. SCT, WT, and AT in the legend indicates the single crystal, wrought, and wrought/annealed tungsten types, respectively. Values preceding the material identifiers are the specimen thicknesses in millimeters. The red reference line in the graph indicates the stress vs. displacement curve for a solid 316L SS specimen, i.e., without a tungsten core.

Push-Out Testing

Push-out stress/displacement curves were obtained by measuring the load vs. displacement during testing and dividing the loads by the interfacial area of each test specimen (π x diameter x thickness) to yield an "interface area-normalized load". Testing was performed on 316L SS bonded to tungsten in the wrought, wrought/annealed, and single crystal forms at 810°C. A load-displacement test was also performed on a solid disc sample of the 316L SS cladding material. Although the load/displacement traces varied somewhat from specimen to specimen, all of the traces were similar in shape (Figure 3). The ranges of maximum pushout loads for different specimen types also overlapped, averaging 392MPa for the wrought tungsten specimens, 421MPa for the single crystal tungsten specimens, and 443MPa for the wrought/annealed tungsten specimens. Cracking of the tungsten was observed for all specimen types, and was primarily confined to the bottom surfaces of the specimens. Yielding of the clad material in a narrow zone (~100μm in width) near the interface was observed on both the top and bottom surfaces of all tested specimens. The push-out stress vs. displacement curves for all specimens generally followed the curve produced by testing a pure 316L SS specimen with no tungsten core up to a maximum stress of ~160MPa. Above approximately half the maximum loads achieved, the clad tungsten specimens achieved a higher stress for the same displacement as that obtained for the pure 316L SS test specimen (solid red curve in Figure 3). The higher stress results from testing of a composite material (316L SS clad tungsten) which contains a material (tungsten) possessing a modulus of elasticity approximately twice that of 316L SS.



A B

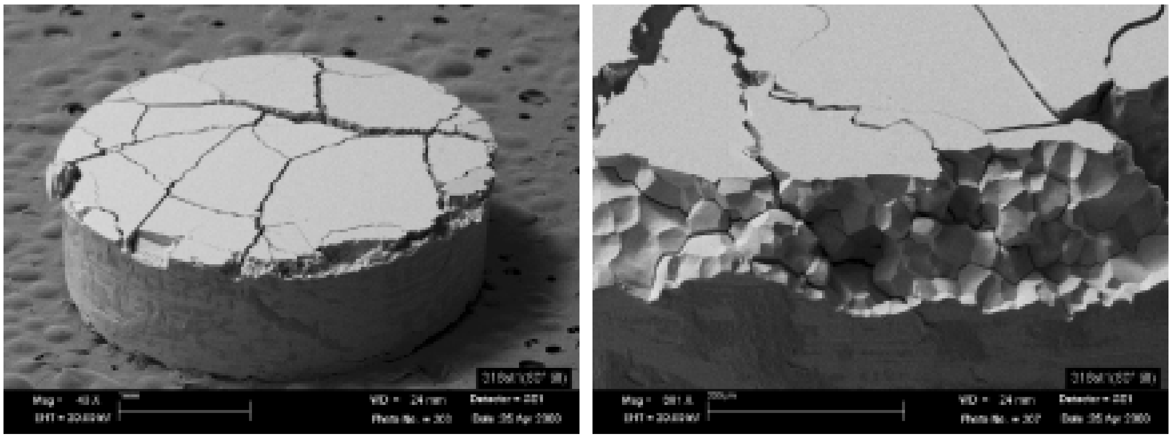
Figure 4 A and B. SEM image showing brittle fracture pattern on the bottom surface of a of wrought tungsten core and ductile yielding of the surrounding 316L SS cladding from a partially-tested 810°C HIP-bonded 316L SS clad wrought tungsten push-out specimen.

<u>Fracture Analysis</u>

An SEM analysis of a representative 810°C HIP-bonded 316L SS clad wrought tungsten specimen after partial push-out testing revealed brittle fractures on the bottom surface of the specimen along with yielding in the 316L SS cladding, as seen in Figure 4. This specimen was stopped before it reached the maximum push-out stress needed for complete debonding.

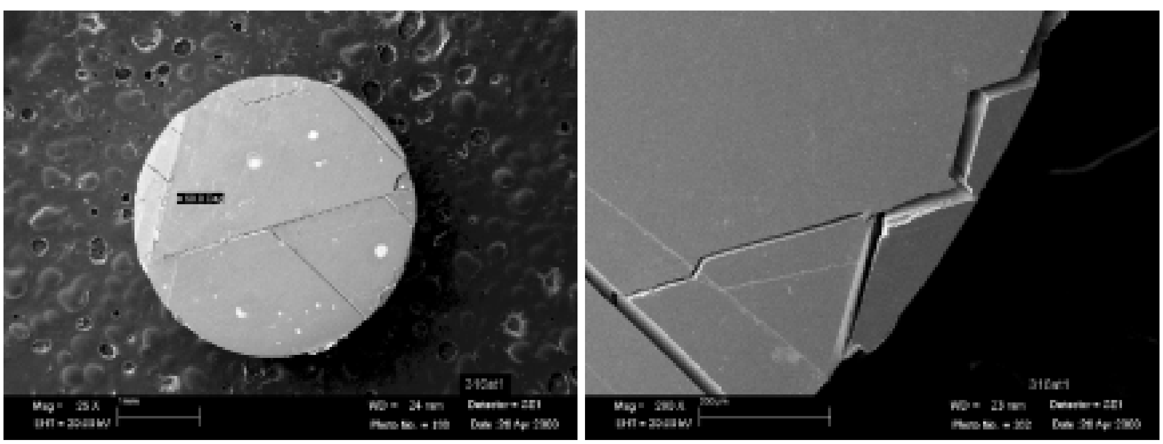
SEM analysis of a 810°C HIP-bonded 316L SS clad wrought/annealed tungsten push-out test specimen revealed cracking and fracture features on the bottom surface of the specimen which were consistent with intergranular failure (Figure 5). The overall numbers of visual cracks in this specimen were smaller than that for the 316L SS clad wrought tungsten specimen (Figure 4), and is consistent with the larger grain size in this specimen. The push-out stresses for 316L SS clad wrought/annealed tungsten ranged from 410 to 480 MPa for displacements of 0.10 to 0.15mm as shown in the AT curves in Figure 3.

SEM analysis of a 810°C HIP-bonded 316L SS clad single crystal tungsten push-out specimen revealed cracking on the bottom surface of the specimen at angles of 60˚ and 120˚ (Figure 6). Laue back reflection x-ray analysis had previously revealed that the <111> crystallographic direction was parallel to the loading axis of the specimen. The cracking is, therefore, consistent with traces of (110) type planes, the cleavage planes in tungsten. 316L SS clad single crystal tungsten specimens reached stresses of 400 to 440MPa at a displacement of 0.10 to 0.15mm, as seen in the SCT curves in Figure 3.



A B

Figure 5 A and B. SEM image showing intergranular fracture on the bottom surface of a wrought/annealed tungsten core from a fully-tested 810°C HIP-bonded 316L SS clad wrought/annealed tungsten push-out specimen.



A B

Figure 6 A and B. SEM images revealing cracking at 60° and 120° on the bottom surface of a single crystal tungsten core from a fully-tested 810°C HIP-bonded 316L SS clad single crystal tungsten push-out test specimen.

Repetitive loading tests to successively higher loads, followed by SEM imaging between each loading step, were performed on a 316L SS clad wrought tungsten specimen HIP-bonded at 810°C (Figure 4). The tests revealed failure initially occurred by yielding of the cladding material followed by crack development in the tungsten at the bottom surface at ~1/3 of the ultimate push-out load. The cracks gradually opened, grew in size, and multiplied as the push-out load was increased. At approximately 1/2 to 2/3 of the maximum push-out load, cracking along the interface was observed. Based on the results of previous finite element modeling analysis of push-out tests [2, 3], it appears that the cracking observed on the bottom of the tungsten resulted from tensile stress developed normal to the bond interface from bending moments induced in the specimen due to the testing configuration [4]. After testing each specimen, observations using EDS of areas examined in the SEM backscattered electron imaging mode revealed the remains of tungsten adhering to the cladding material in some areas and the remains of cladding adhering to the tungsten core in other areas.

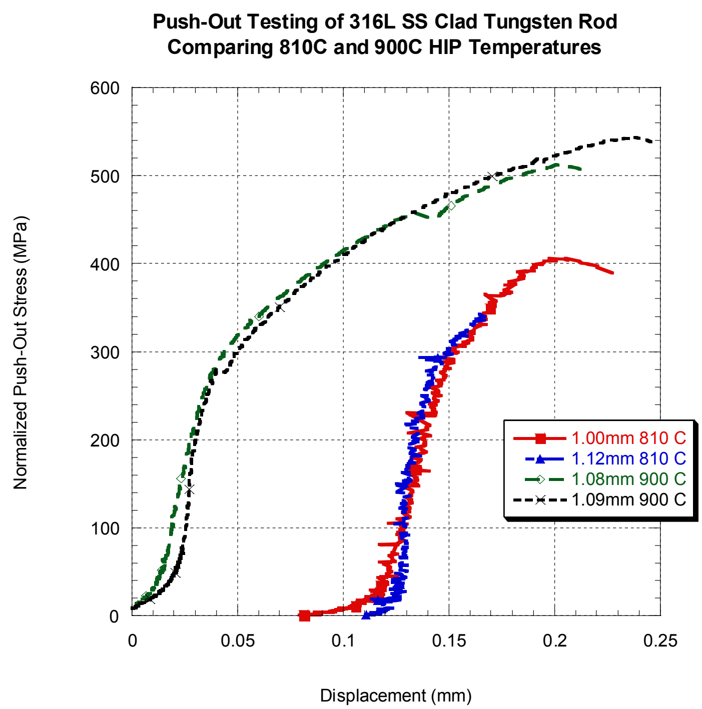


Figure 7. Comparison of push-out stress vs. displacement curves for 810°C and 900°C HIP-bonded specimens. The 810°C results have been shifted by a displacement of 0.1mm to the right to separate the 810°C and 900°C data for viewing purposes. The legend indicates specimen thickness followed by the HIP temperature.

Effect of HIP Temperature

Figure 7 compares the push-out stress vs. displacement curves for push-out specimens HIP-bonded at 810°C and 900°C. Up to the failure loads for the 810°C specimens, both sets of curves exhibit very similar behavior. There was a ~25% increase in strength for the 900°C HIP-bonded specimens over that for 810°C HIP-bonded specimens that can be associated with the increase in thickness of the diffusion zone observed for the 900°C HIP-bonded specimen (Figure 7).

Effect of Irradiation

The push-out stress vs. displacement curves for unirradiated and irradiated 316L SS clad wrought tungsten specimens HIP-bonded at 810°C are shown in Figure 8 along with push-out test results for unirradiated 316L SS and a sample of 316L SS irradiated in the same HFIR irradiation test. There was a ~11% decrease in the maximum push-out stress after irradiation which may lie outside the error of measurement. More tests would be needed to substantiate the decrease. The data for the solid 316L SS specimens indicate a substantially hardening effect, which is consistent with irradiation-induced increases in the tensile yield strength of 316L SS previously observed [5, 6].

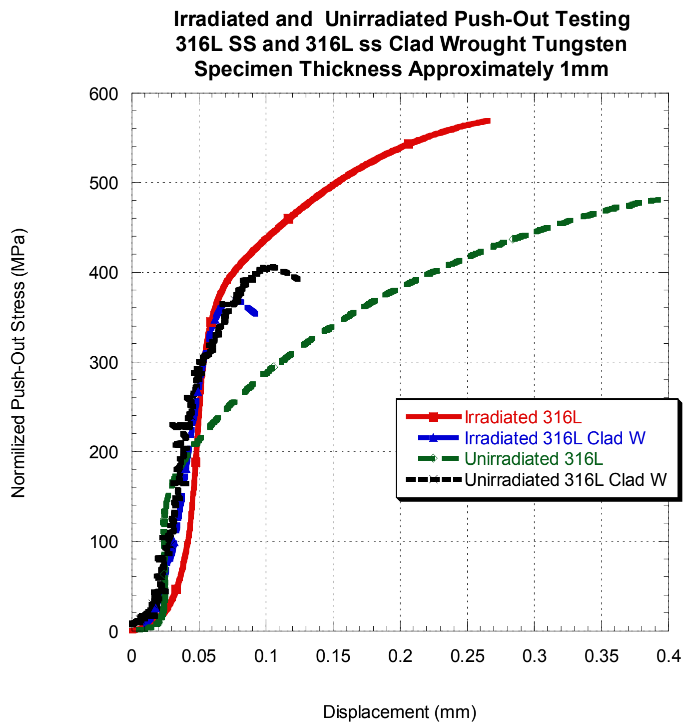


Figure 8. Push-out stress vs. displacement curves for both unirradiated and irradiated 316L SS and 810°C HIP-bonded 316L SS clad wrought tungsten.

Summary and Conclusions

An investigation was conducted to determine the effect of diffusion bonding processing variables on the interfacial strength of 316L SS clad tungsten rods to assist in determining optimum processing parameters for fabrication of tungsten target components for the APT program. A test specimen configuration and test method were developed and specimens were fabricated to measure the interfacial strengths of diffusion-bonded 316L SS clad tungsten specimens. Test specimens were fabricated by HIP-bonding at several temperatures and using several tungsten material types. The HIP-bonding method and 316L SS cladding material were previously selected for fabrication of target components for the APT program [1]. A push-out test method was selected similar to that used for determining the fiber/matrix interface strengths in fiber-reinforced composites [2, 3].

Trial bonding temperatures of 800°C and 950°C were initially selected based on diffusion calculations performed using interdiffusion data for nickel and tungsten [1], and samples of 316L SS bonded to wrought tungsten were fabricated at these temperatures using a uniaxial vacuum hot pressing method. Analysis of diffusion zones in uniaxially-bonded 316L SS/tungsten specimens revealed that alloying elements of the 316L SS (Ni, Cr, Fe) had diffused into the tungsten for both bonding temperatures and a second phase had developed at the 316L SS/tungsten interface for the 950˚C bonded sample. Based on these results, HIP temperatures of 810°C and 900°C were selected for fabrication of push-out test specimens.

Clad assemblies of 316L SS bonded to tungsten were prepared by HIP-bonding at 810°C and 900°C and thin sections were cut, ground, and polished on both ends to provide push-out test specimens. Electron microprobe analysis of HIP-bonded push-out specimens revealed interdiffusion zones between the 316L SS and tungsten of ~5µm for a HIP temperature of 810°C and ~9µm for a HIP temperature of 900°C.

To evaluate the effect of tungsten material type on bonding, three types of tungsten rod materials (single crystal, wrought, and wrought/annealed at 1800°C for 15 hours) were HIP-bonded to 316L SS, and push-out test specimens were fabricated and tested to measure bond strength. A ~50MPa difference was observed between the maximum push-out stresses reached for the three forms of tungsten, indicating that tungsten grain size had little effect on bond strength. A ~25% increase in push-out stress was observed between specimens HIP-bonded at 900°C and those HIP-bonded at 810°C. This increase in push-out stress is attributed to an increased bond strength resulting from an increased interdiffusion zone thickness for the 900°C HIP-bonded specimens.

SEM analyses of composite parts (tungsten rod and 316L SS cladding) from fully-tested push-out test specimens showed significant cracking in the tungsten for all specimens, generally being confined to their bottom surfaces, and yielding in the 316L SS cladding material in narrow zones near the interface on both top and bottom surfaces. Cracking in HIP-bonded 316L SS clad single crystal tungsten specimens occurred on (110) type cleavage planes. Cracking in wrought and wrought/annealed tungsten specimens appeared to be intergranular in nature, with an increased density of cracks in the wrought tungsten, consistent with the smaller grain size observed in this material type.

Based on the examination of push-out test specimens which were step loaded in successive increments followed by SEM analysis after each increment, specimen failure was observed to initiate by yielding in the 316L SS cladding material at both top and bottom surfaces of the specimen, followed by initiation of cracking in the tungsten at the bottom surface at ~1/3 of maximum push-out load, and finally by crack propagation in the tungsten and into the 316L SS clad tungsten interface at between 1/2 and 2/3 of maximum load. It is proposed that cracking in the tungsten in this test specimen and other fully-tested and partially-tested push-out test specimens resulted from failure of the tungsten from tensile stresses generated at the bottom of test specimens from a bending moment imposed in that region. This mechanism is also supported by finite element modeling and analysis [4]. Cracking was constrained to the tungsten due to tungsten's lower strain tolerance for fracture compared to that for the 316L SS cladding.

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