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FAILURE ANALYSIS OF PB-1(EBTS Be/Cu MOCKUP)

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ABSTRACT

Failure analysis was conducted on PB-1 following a tile failure during a high heat flux experiment in the EBTS facility (SNL/NM). PB-1 consists of a series of beryllium tiles joined to a copper alloy substrate. This actively cooled assembly was subjected to a high heat flux loading condition to simulate the ambient conditions inside ITER. The beryllium tiles were bonded to the copper alloy substrate using a low temperature diffusion bonding process being considered for use in fabricating plasma facing components in the ITER project. Using a coating technique developed at SNL/CA, copper was electrodeposited on beryllium. This copper coated surface was then diffusion bonded to a wrought copper alloy (Hycon-3) at 450°C in a hot isostatic press. The bond microstructure was examined using optical and electron microscopy. The bond strength was evaluated using tensile tests and high heat flux experiments in the electron beam test system at SNL/NM. The results of this analysis show contrasting differences between the failure during a room temperature tensile test and the failure experienced in the EBTS facility.

The room temperature tensile specimens failed at the copper-beryllium interface in an intermetallic phase formed by the reaction of the two metals at the bonding temperature. The fracture strength values measured by these tests were in excess of 300 MPa. The high heat flux specimens failed at the copper-copper diffusion bond. The fracture morphology in both cases was a mixed mode consisting of dimple rupture and transgranular cleavage. Several factors, which could provide reasons for this difference in failure mechanism, are proposed.

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FAILURE ANALYSIS OF PB-1 (EBTS Be/Cu MOCKUP)

I. Introduction

One of several processes being evaluated as a method of bonding copper to beryllium for first wall fabrication on the ITER project utilizes a relatively low temperature to diffusion bond copper to copper ($<0.5 T_{mp}$). The process consists of the following steps: beryllium samples are cleaned and electroplated with approximately 0.8 mm of copper. This coating is then machined back to approximately 0.5 mm to provide a planar surface. Two copper alloy samples (Hycon-3) are machined to a thickness of approximately 19 mm and match the footprint of the coated beryllium sample. A stainless steel can is machined which will contain the assembly to be bonded. All copper surfaces are machined flat and parallel, then etched to remove oxides just prior to encapsulation in the container. This assembly is then evacuated and sealed by welding in an electron beam welding chamber. The assembly is then placed in a hot isostatic press (HIP) and subjected to a 450°C iso-thermal hold for three hours at a pressure of 103 Mpa (argon) to accomplish the diffusion bond.

II. Experimental Procedure

Two pressing geometries were processed in the manner detailed above. The first is a cylindrical geometry (50 mm dia x 75 mm). Material from this pressing is used to provide tensile bars and metallographic samples for bond evaluation as part of a screening test used to select candidate bonding processes for more extensive testing. The tensile bars discussed in the next section of this report came from a pressing from this geometry (EDDB-5). EDDB-5 was processed using the same procedures as the PB-1 sample used for testing in the electron beam test system (EBTS) facility located at SNL/NM. PB-1 is designed to simulate a section of the ITER first wall (Figure 1). It was during testing that one of the tiles (tile C) from PB-1 failed after ten cycles at a heat load of 3 MW/m². Cu-Be interface temperatures during these tests was measured at 275-300°C. The test was eventually aborted after the other three tiles showed evidence of failing. The only tile discussed in this report is tile C.

Metallography

A metallographic sample was prepared to evaluate the microstructural features in the bond region using material from EDDB-5. The diffusion bonded interface between the electrodeposited (ED) copper and the wrought copper alloy (Hycon-3) is shown in Figure 2. The Hycon-3 grain morphology is seen as a fine grained, heavily-worked microstructure produced during the conversion from billet to wrought product and subsequent heat treatments. The ED copper microstructure exhibits an extremely small grain morphology with clear evidence of voids. This microstructure is expected given the low temperature used in the bonding process (450°C). Bonding temperatures in the recrystallization range ($>0.75 T_{MP}$), would result in a void-free, wrought grain structure in the ED copper. These microstructures has been observed in previous applications which deploy this ED copper process (reference 1). The absence of this grain morphology and the presence of voids suggests minimal mass diffusion during the bonding process. However, the bond interface appears to be relatively void free (Figure 2).

The bond interface between the beryllium and ED copper is shown in Figure 3. In this photomicrograph the intermetallics formed between the copper and beryllium are clearly evident.

The BeCu intermetallic is seen as a continuous phase with a thickness of 3 μm . The darker phase (hardly discernible) in the reaction zone is the Be_2Cu . The phases have been identified in earlier studies using similar materials and temperatures (references 2, 3).

Tensile Bars

Tensile bars were machined from material produced in the EDDB-5 pressing. The cross section of the tensile bar in the gauge section was approximately 5 mm x 3 mm with a gauge length of 15 mm. The bonded section is shown schematically in Figure 4. The mechanical strength of the bonded sections (i.e., across the intermetallic phase) was very high. The fracture stress for the two bars tested at room temperature was 322 MPa and 331 MPa. This is compared to 397 MPa for the ultimate tensile strength (typical) for S-65 C beryllium in the transverse direction at room temperature (reference 4) and 675 MPa (minimum) for the Hycon-3 (reference 5). In both specimens, fracture occurred at the Be_2Cu intermetallic/beryllium interface. The fracture morphology is shown in Figures 5-7. In one case, the fracture initiated at the intermetallic/beryllium interface and propagated into the beryllium as the crack front progressed (Figure 5). In the other test, the fracture initiated at the interface and continued along the intermetallic phase until failure occurred. The fracture morphology in the beryllium is seen as a cleavage fracture which is the normal fracture mode at room temperature (reference 6). The fracture morphology in the intermetallic is a mixed mode fracture consisting of cleavage and quasi-cleavage rupture.

PB-1(tile C)

Metallographic samples were not prepared for this assembly. The fracture morphology of the tile C failure is shown in figures 8-10. In contrast to the room temperature tensile tests, failure occurred at the ED copper-wrought copper alloy interface (i.e., the low temperature diffusion bond). The fracture propagates along the original bond interface with a mixed mode fracture morphology. The fracture surface exhibits areas of ductile rupture and areas where features are poorly defined (Figure 9). As noted in Figure 8, a small corner section of the tile was fractured during removal of the thermocouple. This fracture is similar to the room temperature tensile fractures. Here the fracture initiates at the intermetallic/beryllium interface and propagates either through the intermetallic or into the beryllium.

III. Discussion

The intermetallics formed at the beryllium-copper alloy interface are reason for concern. The bonding process is produced by heating to 450°C for 1-3 hours. Aging studies using similar materials and processing parameters have shown that the presence of these intermetallics will exist. Although a continuous layer of the BeCu and Be_2Cu intermetallics may produce a strong bond, which is clear from the room temperature tensile tests, the ductility and fracture toughness of this bond is questionable. No references on the mechanical properties of the Be-Cu intermetallic phases could be found; however, intermetallics such as the Ni and Ti-based intermetallics have high strength, low ductility properties near room temperature (reference 7).

Several factors may explain the differences observed in the two test failures. Clearly, the differences in test parameters are large. The test temperature is different. At room temperature, the residual stresses generated by the thermal expansion gradient in the two materials (beryllium and copper) is greatest. These stresses combined with the external stresses generated by the tensile tests may force the failure to the beryllium-copper interface. This is in contrast to the tile C failure

where the residual stresses due to the thermal expansion gradient are lower (i.e., approximately zero at the bonding temperature, 450°C). In such a case, the fracture origin may shift to the copper-copper interface. Also, the loading conditions are dramatically different. In the case of the tensile tests, dismissing any misalignment, the loading is in tension. In the case of tile C, the loading conditions are complex and certainly consist of both tension and shear components. Finally, there is the possibility that the HIP processing for the two assemblies was significantly different and therefore changed the resulting bonding strengths.

IV. Conclusions

1. Fracture in the room temperature tensile bars occurred in the Be₂Cu-BeCu reaction zone layer at the copper-beryllium interface. This bond exhibited high fracture strength values (322, 331 MPa). The fracture was a mixed mode consisting of dimple rupture and transgranular cleavage.
2. Fracture in tile C occurred in the diffusion bond between the electrodeposited copper and the wrought copper alloy.
3. A second failure on tile C occurred at the beryllium-copper interface during removal of the thermocouple. This failure looked similar to the tensile bar fractures.
4. The contrasting fracture events are different as are the test temperatures, loading conditions, and HIP cycles. Any of these factors or a combination may provide the reason for a change in the fracture mechanism from the Cu-Be interface at room temperature to the Cu-Cu interface at approximately 275°C.
5. A continuous layer of intermetallics is formed between the copper and beryllium at the diffusion bonding temperature (450°C). These phase(s), although exhibiting high strength also may exhibit poor ductility and fracture toughness.
6. Results from the room temperature tensile tests suggest that these tests are not a good measure of the performance in the EBTS tests. These tests were initiated to provide a strength value and act as a rough screening test for candidate joining processes. Strength may not be the limiting parameter for this application. Perhaps an elevated temperature tensile test or thermal cycling prior to tensile testing would provide a better screening test in preparation for the more expensive tests at the EBTS facility.

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VI. References

1. W. D. Bonivert, H. Johnson, B. C. Odegard, Sandia National Laboratories, Livermore, CA, private communication (June 1996).
2. H. Kawamura and M. Kato, *Reactivity Test Between Beryllium and Copper*, Proceedings 2nd IEA International Workshop on Beryllium Technology for Fusion, September 6-8, 1995, at Jackson Lake Lodge, WY, edited by G. R. Longurst, INEL, CONF-9509218, pp 204-211.
3. B. C. Odegard, C. Cadden, and N. Yang, *Beryllium and Copper Reactivity in an ITER Joining Environment*, to be presented at the Fourth International Symposium on Fusion Nuclear Technology, Tokyo, Japan (April 6-11, 1997).
4. D. Dombrowski, E. Deksnis, and M. Pick, *Atomic and Plasma-Material Interaction Data for Fusion*, 1994 IAEA Conference Proceedings, Supplement to the Jnl of Nuclear Fusion, Volume 5, Vienna, Austria.
5. J. Harkness, W. Spiegelberg, and W. Cribb, *Beryllium-Copper and Other Beryllium-Containing Alloys*, Metals Handbook, Vol 2, 10th edition: Properties and Selection: (ASM International, Materials Park, OH), pp 403-427.
6. A. Goldberg, J. Hanafee, and R. Scott, *Mechanical Properties Evaluation of Some Commercial Beryllium Materials*, UCRL-87392, Lawrence Livermore National Laboratories, (June 30, 1982).
7. Eds. J. Horton et al, *High-Temperature Ordered Intermetallic Alloys VI*, MRS Symposium Proceedings, Volume 364, November 28-December 1, 1994, Boston, MA, published by Materials Research Society, Pittsburg, PA, (1995) pp 837-842, 629-634.

Figure 1. Schematic of the EBTS sample configuration. Tile C is the third tile from the left. Failure occurred at the Hycon-3 - ED copper interface (not shown).

Figure 2. Micrograph showing interface between the wrought copper alloy (Hycon-3) on the left and the ED copper on the right. Although there is a distinct demarcation at the interface due to the different grain morphologies, there is relatively little evidence of the original pre-bonded interface. Note the porosity and the extremely fine grain morphology in the ED copper. The lower bonding temperature (450°C) and pressures used in the HIP cycle are insufficient to entirely eliminate this porosity and initiate grain growth. This microstructure is common to both the tensile specimen and tile-C.

Figure 3. Micrograph showing the interface between the beryllium (on the left) and the ED copper (on the right). The intermetallic phase is clearly evident at this magnification. The BeCu intermetallic is approximately 3 microns thick (a). The Be_2Cu intermetallic is seen as the darker phase at the beryllium interface (b). At this magnification, the Be_2Cu is hardly discernable. Microcracks (c) seen in the beryllium are most likely due to high residual stresses generated during cool-down from the bonding temperature. This microstructure is common to both the tensile specimen and tile-C.

Figure 4. Schematic showing tensile bar geometry and location of the beryllium-copper bond.

Figure 5. Low magnification micrograph showing fracture path in the tensile bar. The fracture initiated in the intermetallic phase at the bottom right hand side of the photograph and propagated into the beryllium. The lighter phase is the intermetallic phase (determined by secondary electron imaging). The beryllium is in the upper portion of the fracture. Convergence of the river patterns in the beryllium aid in determining the fracture origin.

Figure 6. Tensile bar: fracture in the beryllium is seen as cleavage along specific crystallographic planes.

Figure 7. Fracture in the intermetallic region of the tensile bar. The fracture path is likely a mixed mode consisting of cleavage and quasi-cleavage through the intermetallic phase. (See Figure 10 for higher magnification of a similar area)

Figure 8. A low magnification photograph of the tile C failure. This corner was ripped away during removal of the thermocouple. The lighter area (a) is fracture in the intermetallic phase. The darker area (b) is fracture in the beryllium. The layer that is pulled away is the ED copper. The top surface (c) is the main fracture in tile C and is located at the interface between the ED copper and the Hycon-3.

Figure 9. Micrograph showing the main fracture in tile C. This is the surface of the ED copper. The higher magnification shows some evidence of a ductile rupture. However, there are areas that indicate lack of bonding (darker areas).

Figure 10. Fracture in the small corner section (Fig. 6) of tile C. This is the fracture region going from area b (left) to area a (right). The fracture morphology in area a (beryllium) is cleavage along specific grain orientations. The fracture morphology in area b (intermetallic) is mixed mode consisting of cleavage and quasi-cleavage through the intermetallic phase.

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