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Evaluation of Cu as an interlayer in Be/F82H diffusion bonds for ITER TBM

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ABSTRACT

Copper has been investigated as a potential interlayer material for diffusion bonds between beryllium and Reduced Activation Ferritic/Martensitic (RAFM) steel. Utilizing Hot Isostatic Pressing (HIP), copper was directly bonded to a RAFM steel, F82H, at 650 °C, 700 °C, 750 °C, 800 °C and 850 °C, under 103 MPa for 2 h. Interdiffusion across the bonded interface was limited to 1 μm or less, even at the highest HIP'ing temperature. Through mechanical testing it was found that samples HIP'ed at 750 °C and above remain bonded up to 211 MPa under tensile loading, at which point ductile failure occurred in the bulk copper. As titanium will be used as a barrier layer to prevent the formation of brittle Be/Cu intermetallics, additional annealing studies were performed on copper samples coated with a titanium thin film to study Ti/Cu interdiffusion characteristics. Samples were heated to temperatures between 650 °C and 850 °C for 2 h in order to mimic the range of likely HIP temperatures. A correlation was drawn between HIP temperature and diffusion depth for use in determining the minimum Ti film thickness necessary to block diffusion in the Be/F82H joint.

1. Introduction

In the design of ITER, a 2 mm thick layer of beryllium was originally considered as an armor material for the plasma facing surfaces of the Test Blanket Modules (TBM). The requirement for all TBMs to be coated with beryllium was later alleviated due to the recess of the TBMs in the equatorial port modules. And while this material joint may not be used specifically in ITER, there is a notable gap in current knowledge concerning the joining technique and characterization for direct joining of plasma facing armor layers to structural steels for future fusion reactors [1,2]. It is not completely settled how these armor layers should be attached, though one proposed manufacturing method is Hot Isostatic Pressing (HIP). The HIP process has been commonly used for joining parts and fabrication of the ITER First Wall (FW) panel and has, therefore, been the subject of a significant amount of research. The work presented here is a preliminary study intended to focus on the bonding characteristics of S65-C beryllium to the Japanese manufactured F82H.

Some research has been conducted by the Japan Atomic Energy Agency (JAEA) on a joining methodology that involves a chromium interlayer to block diffusion of unwanted elements into the highly reactive beryllium substrate [3]. While some success was found with the Cr interlayer, other interlayer combinations were performed by Lee et al. [4], including one sample to utilize a copper/titanium interlayer regime. This interlayer technique was used with promising results during the joining of the US ITER FW mock-up, and has therefore been chosen as the focus for this research [5–7].

Titanium acts as a diffusion barrier and copper as a bonding aid and strain accommodating layer [2,8]. This has the benefit of increasing the overall ductility in the joint, with the purpose of reducing local stresses during thermal expansion in the region. Prior diffusion bonding research has shown that titanium is a good bonding aid in bonds between copper and beryllium [9]. However, in order to ensure that the Ti is thick enough to mitigate the formation of brittle BeCu intermetallics, experiments were conducted to measure the diffusion of Cu into Ti.

Additionally, previous studies have shown that copper often does not easily bond to RAFM steel [10,11]. Ferritic steel has a BCC structure, while copper has an FCC crystal structure, both of similar lattice size. This difference in crystal structure makes diffusion between the couple difficult, which could potentially lead to a low strength bond. For this reason, the strength of a pure copper to RAFM steel interface was investigated.

2. Cu to RAFM joining

2.1. Experimental

For the HIP bonding of Cu to ferritic steel, commercial oxygen free copper (OFC), grade C10200, was HIP bonded to RAFM steel,

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Fig. 1. HIP cans after closure, prior to HIP cycle.

grade F82H. After chemical cleaning, the copper and steel substrates were placed inside cans machined from stainless steel schedule 80 pipe. The HIP cans are shown in Fig. 1.

There are three conditions in the process of HIP'ing that can be varied: temperature, pressure, and time. Of the three conditions, only the effect of temperature was explored. The same HIP pressure and hold times, 103 MPa for 2 h, were used as was employed during manufacturing of US mockups of the ITER blanket FW [3]. In this way, it was possible to determine the HIP temperature that created conditions at the interface corresponding to maximum tensile and shear strength. Five samples were HIP'ed at Bodycote in Andover, MA: one each at 650 °C, 700 °C, 750 °C, 800 °C and 850 °C.

Tensile and shear test coupons were wire cut via electric discharge machine (EDM) by Axsys Technologies Inc. (Cullman, AL), from the bonded Cu/F82H joints. The shear test specimen design was based on a modified DIN 50162/ASTM A263 standard designed to measure the strength of a beryllium to copper interface. The specimens were mounted using a fixture that provided lateral support while loading the copper in pure shear akin to the aforementioned standards. The tensile specimens were of a simple, flat dogbone design. Tests were performed on a SATEC Model 22EMP electromechanical test frame. Depth profiling via Electron Microprobe (EMP) and Auger Electron Spectroscopy (AES) was conducted in order to characterize interdiffusion across the bondline.

2.2. Results and discussion

Typical tensile tests results are shown in Fig. 2. For a specimen machined from the 650 °C HIP bond, failure occurred at 134 MPa with no apparent plastic deformation. Fracture occurred along the original bond interface with no evidence of local ductility. Indeed, observation of the fractured surfaces of the 650 °C HIP sample revealed polishing markings from the original preparation of the substrate surfaces. The presence of these markings indicated that there was no appreciable interdiffusion and therefore no metallurgical bonding across the HIP joint at this temperature. Had substantial diffusion occurred, the flowability of the material would have eliminated any polishing lines and some amount of copper would be expected to be seen still attached to the steel after fracture. In fact, samples cut from the material bonded at 700 °C

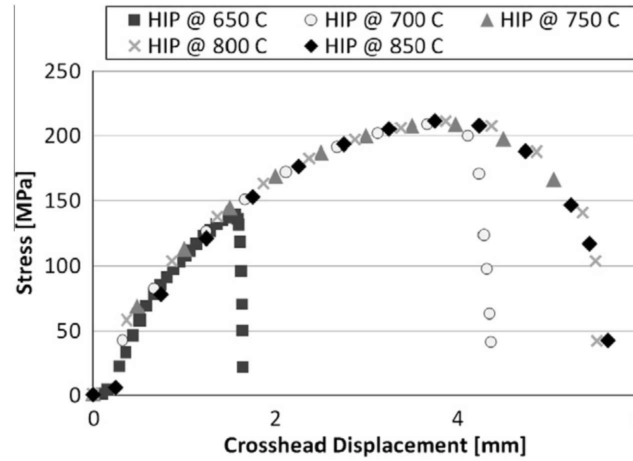

Fig. 2. Stress versus crosshead displacement of tensile tests of bonded samples.

showed exactly this type of behavior. These samples debonded under an approximately 211 MPa tensile load at the material interface. Inspection of the fracture surfaces revealed chunks of copper remaining attached to the steel, as is shown in Fig. 3. This implies that at HIP temperatures around 700 °C, significant diffusion does occur, though not quite enough to produce a joint stronger than the parent materials. In contrast, samples HIP'ed at 750 °C, 800 °C and 850 °C exhibited ductile failure in the bulk copper well away from the HIP interface at peak tensile strengths of approximately 210 MPa. From this information it can be inferred that for temperatures above 700 °C the bond successfully diffuses to create a joint with higher tensile strength than the soft copper. Views of failed specimens from the 650 °C and 850 °C HIP bonds are shown in Fig. 4, in which the location of failure can be seen. Shear testing revealed similar deformation and fracture characteristics with respect to bonding temperature.

An AES line scan from across the 850 °C bondline (Fig. 5) reveals the limited extent of interdiffusion between the steel and copper substrates. For this highest bonding temperature, the interdiffusion zone is only on the order of 1 μm, and no clearly visible intermetallics were observed.

3. Ti/Cu diffusion zone

3.1. Experimental

For studying Ti/Cu diffusion, commercial grade C11000 copper was used to fabricate ten 1-in. diameter cylindrical samples on

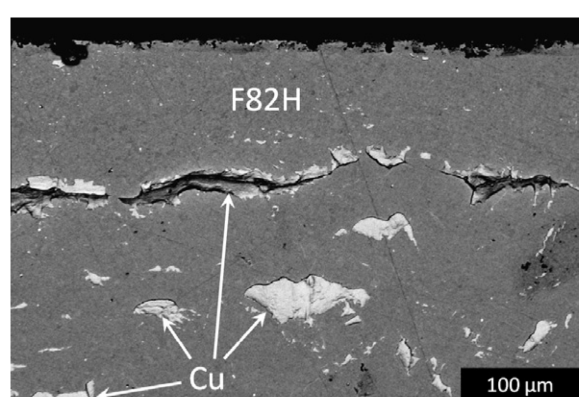

Fig. 3. Backscattered electron image from the fractured surface of the F82H side of a tensile sample bonded at 700 °C.

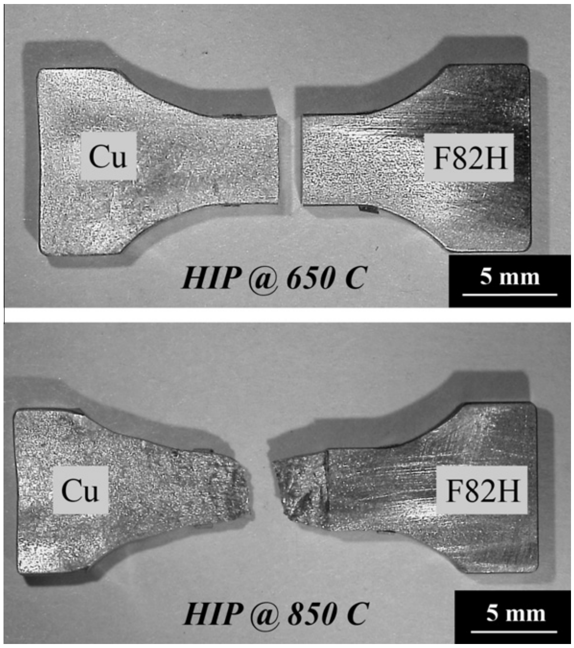

Fig. 4. Tensile samples HIP bonded at 650 °C (top) and 850 °C (bottom).

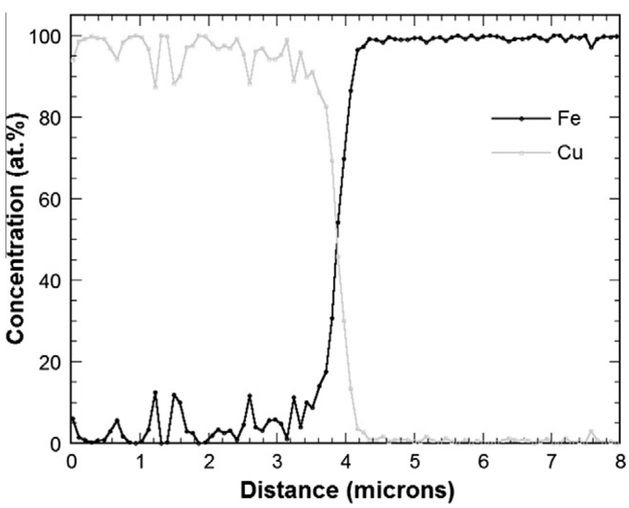

Fig. 5. Auger line scan across Cu/F82H bondline after 850 °C HIP at 103 MPa for 2 h.

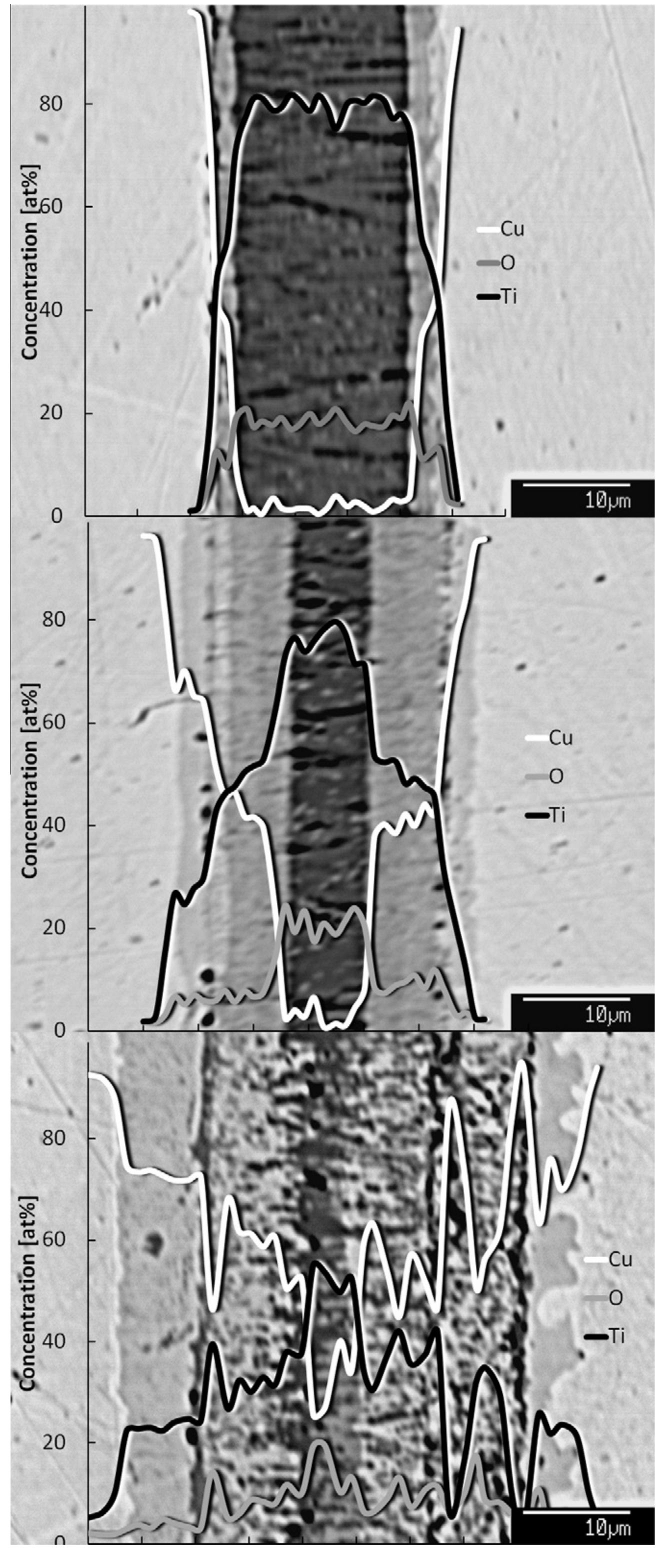

Fig. 6. Backscattered electron images of Cu/Ti diffusion zone, with EMP elemental line scan overlay. The three images correspond to samples heat treated for 30 min at 650 °C (top), 750 °C (middle), 850 °C (bottom), where Cu substrate is at left in each image, and PVD Cu is at right.

which thin films of both Ti and Cu would be deposited. Metallization via Plasma Vapor Deposition (PVD) of 20 μm of 99.99% pure titanium followed by 3 μm thick 99.99% pure copper were deposited at Thin Film Technology (Buellton, CA). To create a sufficient thick Cu film, 20 μm of copper were electroplated onto the PVD Cu. This approach prevented substantial oxidation of the titanium, and also allowed for comparison of Cu/Ti interdiffusion with both bulk Cu and thin film Cu.

Samples were heated to temperature under high quality vacuum in a quartz tube using a Thermolyne tube furnace. Using this set-up, samples were annealed for 30 min at 650 °C, 700 °C, 750 °C, 800 °C and 850 °C under a vacuum better than 1 mtorr. After annealing, the substrates were mounted and metallographically prepared for EMP and AES analysis. Once polished, a line scan was taken across the interface of the samples, revealing the extent of interdiffusion.

3.2. Results and discussion

Fig. 6 shows backscattered electron images taken from the EMP showing the Cu/Ti films after heat treatment for 30 min at temperatures of 650 °C, 750 °C and 850 °C. Overlaid on each of these

Table 1
HIP cycle temperatures performed and their respective strengths and diffusion depths.

HIP temp (°C) (2 h, 103 MPa)	Max tensile strength (MPa)	Max shear strength (MPa)	Ti/Cu diffusion depth (μm) (30 min anneal)	Predicted Ti/Cu diffusion depth (μm)
650	134.73	103.81	4.6	9.1
700	209.23	115.89	7.8	15.6
750	210.22	115.33	11.8	23.6
800	209.38	114.74	Through Ti	–
850	211.56	114.26	Through Ti	–

images are the concentration profiles obtained from the EMP indicating the atomic percentage of titanium, copper, and oxygen present as a function of distance across the interface. Up through the 750 °C anneal, the diffusion front between the Cu and Ti appears planar. There is no appreciable difference in the width of this diffusion front with respect to either the wrought Cu substrate or the finer grain size PVD electrodeposited Cu. EMP elemental line scans reveal the presence of unreacted Ti after both 650 °C and 750 °C anneals (oxygen in the unreacted Ti is present as a deposition artifact). The reaction zone after a 30 min anneal at 850 °C is much more complex and it appears that the all or nearly all of the titanium has reacted to some extent with the copper such that little to no pure Ti remains.

4. Conclusions

The results presented above are the preliminary data for developing a viable method of bonding beryllium to RAFM steel. Table 1 summarizes the findings for both the diffusion studies as well as the HIP bonding experiments. The data from the Cu/F82H HIP bonds show clearly that higher temperature HIP cycles result in stronger bonds and identify a practical lower limit on a HIP bonding temperature for Cu metallized Be. HIP bonding significantly below 700 °C will be problematic with respect to the strength and ductility of the interface region. It can also be seen in the table that the minimum Ti film thickness that will be required to completely prevent the formation of deleterious Cu–Be intermetallics is approximately 9 μm, 16 μm and 24 μm for 650 °C, 700 °C and 750 °C HIP bonds, respectively, assuming 2 h hold times. With these preliminary tests showing promising results, Be will be metallized with Ti and Cu in preparation for HIP bonding between 700 °C and 750 °C.

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