Progress towards a Better Be/Cu Joining for ITER First Wall in China

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Abstract. Technology for Be/Cu joining for fabrication of ITER first wall (FW) panels was investigated. Many types of interlayer materials, such as Ti, Cu, Al, AlSiMg alloy in forms of foil and coating film, were screening tested. Coatings of Ti on Be and pure Cu on CuCrZr substrate were finally selected as the interlayer to fabricate Be/Cu mockups by hot-isostatic-pressing (HIP). Measures to thin the Be/Cu interface for better joining were extensively investigated. It was found that lowering HIPing temperature and time is effective to thin the interface and increase the bonding strength. Other effective approaches include post-HIPing annealing and the use of solid solution annealed (SA) CuCrZr alloy instead of the aging-hardened one. So far the highest bonding strength (shear strength) is 143MPa at room temperature. NDT method to inspect the defects in the interface of the Be/Cu mockup has been built, and a facility for the high heat flux test (HHFT) of the mockup is in construction.

1. Introduction

ITER is to be constructed and will have its first plasma in ~2016. The reactor will demonstrate the integration of many technologies for reactors and serve as a test facility for advanced components such as blankets [1]. China has joined the program and will have her contribution to its construction, operation and so on. One of the in-vessel components that China will fabricate is the shield blanket, which consists of FW panels and shield blocks. The panel is a joint product of Be/Cu/SS, with Be as the plasma facing material.

Diffusion bonding by HIP is usually used to join Be to Cu heat sink. One of the key issues for the bonding is the formation of brittle Be/Cu intermetallic phases. Efforts are made to inhibit or reduce the formation. There are mainly two approaches [2]. One is to place interlayer between the Be tile and the Cu plate. Another is to decrease the diffusion bonding temperature. There are many choices of the interlayer. Typically they are classified into two categories. One is the metals that could not form any new phase with Be or even not dissolved into Be. Another is the ones that could act as a barrier for the diffusion of Cu into Be.

In the present paper, joining Be to Cu by HIP was investigated. Optimization of the interlayer, HIP parameters and the base materials has been investigated and much progress has been made towards a good Be/Cu joining. The technology for the joining will be qualified by HHFT of Be/Cu/SS joint mockups in EU and US in 2008. This qualified technology will be used for the fabrication of ITER shield modules.

2. Screening tests of interlayer materials for the joint

2.1. Material preparation

For the Be/Cu joint, Chinese HIP-Be of 98.5% in purity was used. Pebbles from reduction of beryllium fluoride were vacuum melted and cast into cylindrical ingot that was then machined into small pieces. Different grades of beryllium powders in purity of more than 99% were manufactured from these pieces through mechanical attrition, impact grinding and classification. The powder was firstly cold isostatically pressed into blocks, followed by HIPing at elevated temperature to get high density of ~1.84g/cm^3. Plates were cut from the blocks. The yield and ultimate tensile strength of the Be plates are more than 340 and 450 MPa at ambient temperature, respectively, while the total elongation is more than 3.0%.

DS-Cu or CuCrZr alloy was used as another material for the Be/Cu joining. The DS-Cu is the commercially available product that contains 0.6% Al_2O_3 in mass. CuCrZr alloy was developed in laboratory in the scale of several hundred kilograms. Pure raw metals were melted in an inductive vacuum furnace and cast into ingots, which were then hot and cold rolled into plates. The final plates used for the joining were solid solution annealed (SA) and aged (SAA), at 960^{o}C for 20min. and 475^{o}C for 60 min, respectively. The final chemical composition analysis showed the alloy contains 0.61%Cr, 0.10%Zr and 12-15 ppmO in mass. Its tensile properties were measured. Results are reported in table 1.

TABLE 1: THE TENSILE PROPERTIES OF THE CUCRZR ALLOY IN SAA STATE.

Temperature (^{o}C)	YS* (MPa)	UTS* (MPa)	Elongation (%)
RT	290	423	32
200	250	340	28
400	205	260	35

*YS: Yield strength, UTS: Ultimate tensile strength.

Interlayer material used includes Ti, Al, AlSiMg alloy and Cu in forms of foil and coating. The AlSiMg alloy contains 5.06%Si and 1.88%Mg in mass. Thickness of the foil is 50-100μm. For the coating, PVD (Physical Vapor Deposition) method was employed.

2.2. The procedures for the joint

Small Be/Cu joints were fabricated by HIP in cylindrical shape in diameter of 38-60mm. A photo is shown in Fig.1. Detailed parameters for the fabrication are reported in Table 2. Be and Cu blocks were machined from the plates and were mechanically polished. After cleaned in acetone, Be was degassing in vacuum of 5x10^{-3}Pa at 700^{o}C for 1h. Before PVD coating, sample surface was bombed by energetic Ar ions to remove oxidized layer at surface. Interlayer Foils were polished with sandpaper, etched in acid solution and cleaned in acetone by sequence.

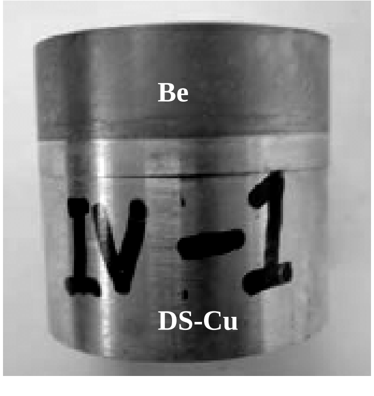

Fig. 1. The Be/Cu joint by HIP.

These cleaned materials were canned and sealed by TIG or electron beam weld after degassing at 400^{o}C for 4h. Sheets of various materials for the can were used in thickness of 1.5 mm. After canning and He leakage test, a HIP process was conducted at 500-850^{o}C for 2h with pressure of 120-150MPa.

TABLE 2: DETAILED PARAMETERS FOR THE FABRICATION OF THE BE/CU JOINT.

Joint No	Pre-HIP coating (μm) on				Interlayer (μm)	HIP Condition* (^{o}C/MPa/h)	Result A: attached F: Failed
	Be	CuCrZr or DS-Cu					
	Al	Al	Ti	Cu			
I	5	5	5	5	AlSiMg-50-100	555/140/2	F
II	5	5			AlSiMg-100	555/140/2	A
III	5			5	AlSiMg-50 +Al-50+Ti-100	540/140/2	A
IV	5					620/140/2	F/A
V				5		620/140/2	A
VI					Ti-100	850/140/2	A
VII					Ti-50	850/120/2	A
VIII					Ti-50	800/120/2	A
IX					Ti-50	700/120/2	A

*For sample of No.I-VI, a stress-release annealing was performed at 420^{o}C for 4h at pressure of 3 MPa.

2.3. Examination and test of the joint

NDT (non-destructive test) of ultrasonic type was performed to inspect cracks on the Be/Cu interface. A standard test specimen from 12mm thick Be plate was fabricated with 10mm deep flat bottom holes and was inspected by the NDT device for calibration of cracks. The holes are 0.3-2 mm in diameter. The inspection results showed that cracks of more than 1 mm in diameter could be successfully detected.

Destructive test of the Be/Cu joint includes microstructure observation by OM (optical microscope) and SEM (scanning electron microscope), and the tensile and/or shear test of the joint to measure the bonding strength at room temperature (RT). Another test of the joint is the thermal fatigue test by pulse electron beam heating Be surface. Sample dimension is 20x20x32mm^{3}. The sample was periodically heated by the electron beam with pulse/interval duration of 16s/36s, while the Cu heat sink was cooled by cold-water flow of 3L/min. The power density at the Be surface was 3.2 MW/m^{2}.

2.4. Results of the screening tests

For most of the joints, Be tile was successfully attached to the Cu alloy. Results are reported in Table 2. A few tests failed to join the two materials, which is due to the leak of the can in the HIP process. So it seemed that any interlayer material listed in Table 2 in forms of either foil or coating is possible for the joining. Fig. 2 shows the tensile or shear stress of the joint.

The strength ranges from ~25 to 68MPa. The joint with interlayer of Ti and AlSiMg alloy foils (No.III, VI and VII-IX) are relatively promising for their relatively higher strength. In the case of Ti foil interlayer, No. VI and IX samples exhibited higher strength. For these two samples, either there was a stress-relief annealing during the cooling phase from the HIP temperature or the HIP temperature is relatively low. Because of this, the residual stress at the Be/Cu interface should be much lower than the others. In another word, the bonding strength could be increased for No. VII and VIII sample with a stress-release annealing after the HIPing.

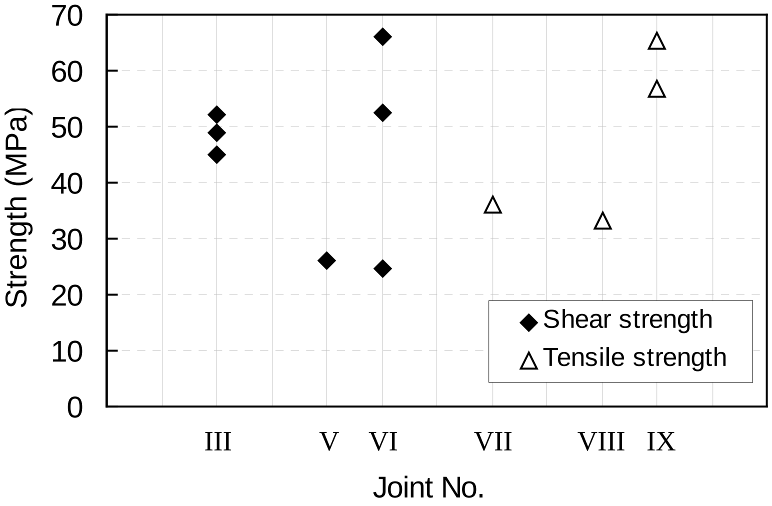

Fig. 2. The shear or tensile strength of the Be/Cu joints at room temperature.

Fig. 3 (a) shows the effect of the post-HIPing annealing on the bonding strength. The annealing was performed at 400°C for 2 h or 420°C for 4 h. Obviously the strength increased significantly due to the annealing. The effect becomes stronger with the increase of the annealing temperature and the annealing duration. The highest strength has reached to 113 MPa after the annealing, which is several times of the as-received one. In this point of view, stress-release heat treatment is very important for a good bonding of Be to Cu alloy.

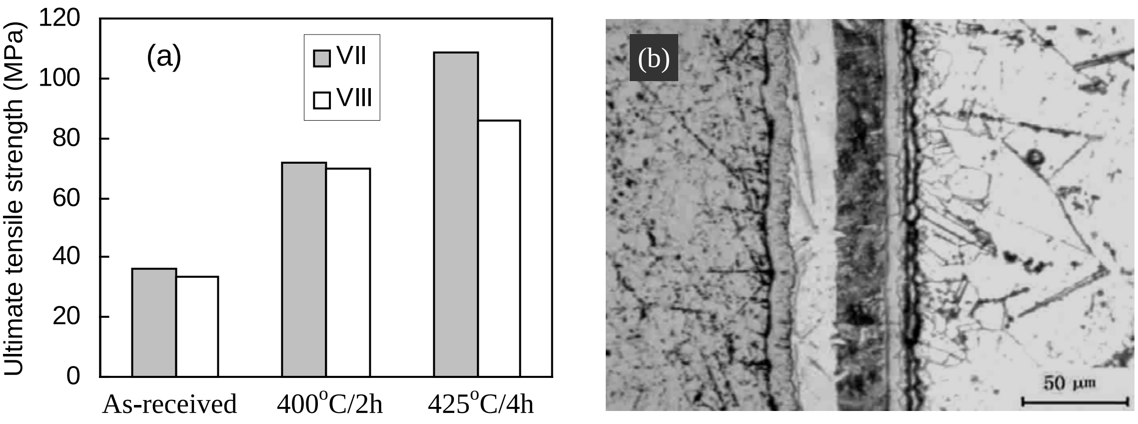

Fig. 3. The RT ultimate tensile strength (a) of No. VII and VIII joints after the post-HIPing annealing treatment, and the microstructure (b) of No. VII joint.

A $20x20mm^2$ specimen was cut by EDMA from No. VI joint. With a cooling tube in the Cu heat sink, the block was thermal fatigue tested in a pulse-operation electron beam machine. The result showed it withstood 1000 cycles without obvious damage at a power density of 3.2 MW/m^2 [4]. So the lifetime of the joint should be longer and could satisfy the ITER FW heat load requirement.

OM observation showed that the interface was very thick, containing several layers in microstructure. Figure 3 (b) is a photo showing the microstructure across the Be/Cu interface for No. VII joint after the post-HIP annealing. The interface contains at least 5 layers with a width of ~70μm in total, which is 20μm wider than the thickness of the Ti foil used. It means the diffusion of the Ti into CuCrZr and Be. The structure is quite similar to what reported by F. Saint-Antonin [3]. According to the report, the interface may consist of intermetallic phases of $TiBe_{12}$, $TiBe_2$, Ti_2Cu, TiCu, Ti_2Cu_3, Ti_3Cu_4 and $TiCu_4$.

3. Optimizing the HIP process for a better performance of the Be/Cu joint

Thick interface containing many intermetallic phases is not good for the performance of the Be/Cu joint at high temperature because the phases are mostly very brittle. To improve the property, the thickness should be reduced. There are usually two ways to reduce the thickness. Based on the theory of solid diffusion, one is to decrease the HIP time, and another is to lower the HIP temperature.

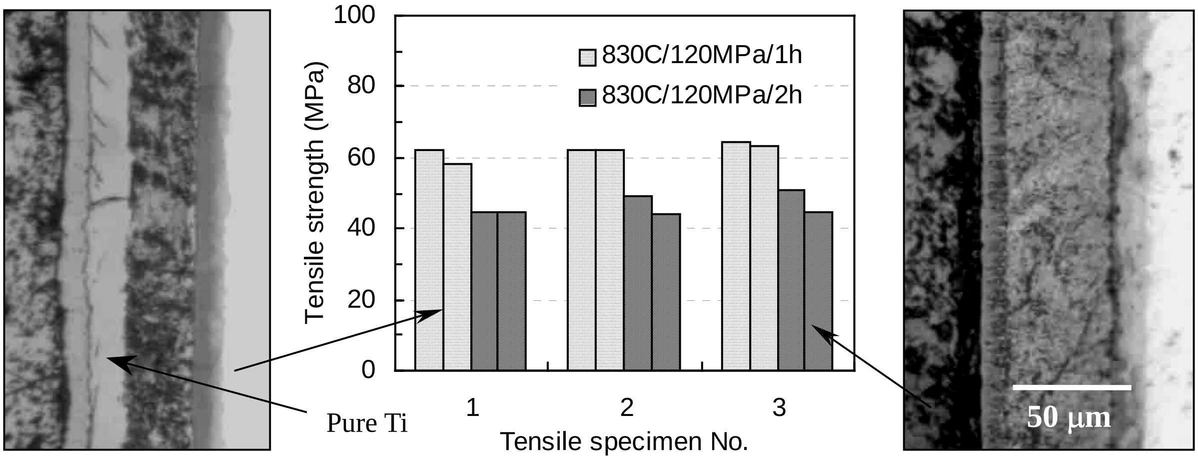

Fig. 4. The RT tensile strength and microstructure of the Be/Cu joints fabricated by HIP at 830°C/120MPa for 1-2h. Ti foil was used as the interlayer.

Figure 4 shows the effect of HIP time on the Be/Cu bonding strength for the HIPing at 830°C/120MPa. Here Ti foil was used as the interlayer material. Though the strength is not high, but the effect is significant. Shortening the time from 2 h to 1 h, the strength had an increase of more than 25%. OM observation showed a little thinning of the interface, which should not affect the bonding strength so significantly. Further analysis by SEM indicated there is a layer of pure Ti remained after the 1h-HIPing, not any more for the case after 2 h. Comparing to the formed intermetallic phase, the pure Ti must be much soft, and thus could act as a compliant layer to release thermal and residual stress at the interface during cooling down from the HIP temperature. As a result, the measured strength is much higher.

Figure 5 shows RT shear strength of the joint fabricated by HIP at 580°C/140MPa/2h. No matter with or without interlayer, the joint displayed much higher strength as compared to others stated above. The highest strength has reach up to 143MPa. SEM observation showed the Be/Cu interface is very thin, about 9μm and 6μm respectively for the one without interlayer and with Ti/Cu interlayer. That the one with Ti/Cu interlayer has thinner interface indicates the barrier role of the Ti to the diffusion of Cu and Be to each other. EDS analysis of the Cu distribution across the interface is shown in Fig.6. For the direct bonding Be to CuCrZr, a sharp increase of Cu at the interface is observed. For the one with Ti/Cu interlayer, obviously the Cu distribution is rather gentle, gradually increasing from Be into CuCrZr alloy.

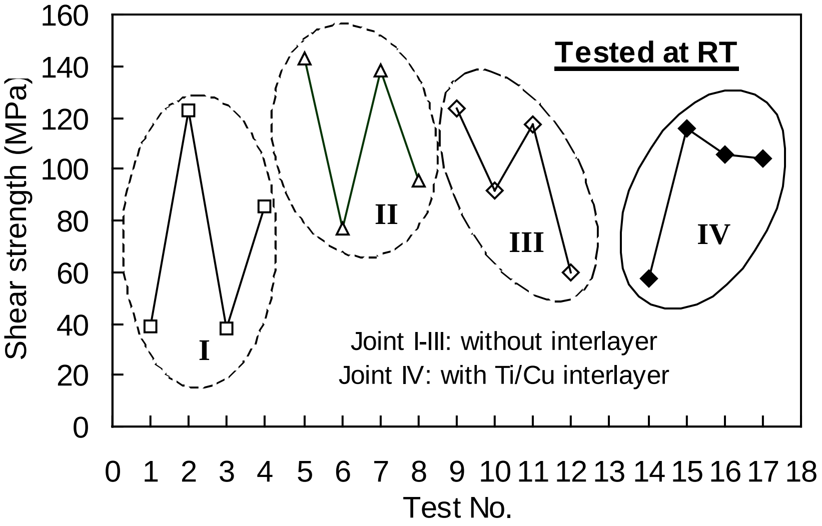

Fig.5. RT Shear strength of the Be/CuCrZr joint at RT.

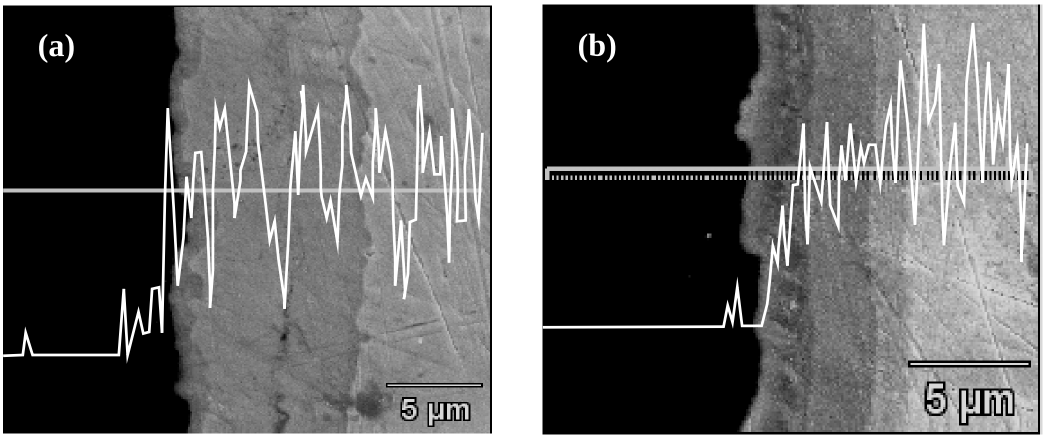

Fig. 6. Cu distribution across the Be/CuCrZr interface, (a) without interlayer, (b) Ti/Cu interlayer.

The thermo-mechanical state of the CuCrZr alloy also has effect on the bonding strength. When the used alloy was in 475°C/4h aging hardening state, the bonding strength is much lower as compared to the joints stated above where SA state CuCrZr was used. For the former, the shear strength at RT was only 67MPa on average.

4. Fabrication of ITER FW mockup for qualification and the testing

Mockups of Be/Cu/SS joint in dimension of 80x244x84 will be fabricated and delivered to IT for HHFT for qualification of the joining technology. For the task, we have designed a mockup with three Be titles and cooling tubes in the CuCrZr heat sink. The technology for the CuCrZr/SS tube joining has been successfully developed. The bonding strength (ultimate tensile strength) reached up to more than 300MPa at room temperature. The Cu/SS mockup fabricated is shown in Fig. 7 (a). Optical microscope observation indicated good joining between the two materials without any crack in the interface.

The fabrication of three Be titles joining to CuCrZr heat sink is still in the process. Before this, technology for joining single Be title to CuCrZr for 50x50x35 mm^3 mockup was studied. Figure 7 (b) is a photo showing one of these small mockups. HIPing parameter was 580^{o}C/150MPa/2h. The maximum shear strength was about 75 MPa at room temperature. OM observation showed good diffusion bonding of the two materials but there were micro-cracks in the diffusion layer.

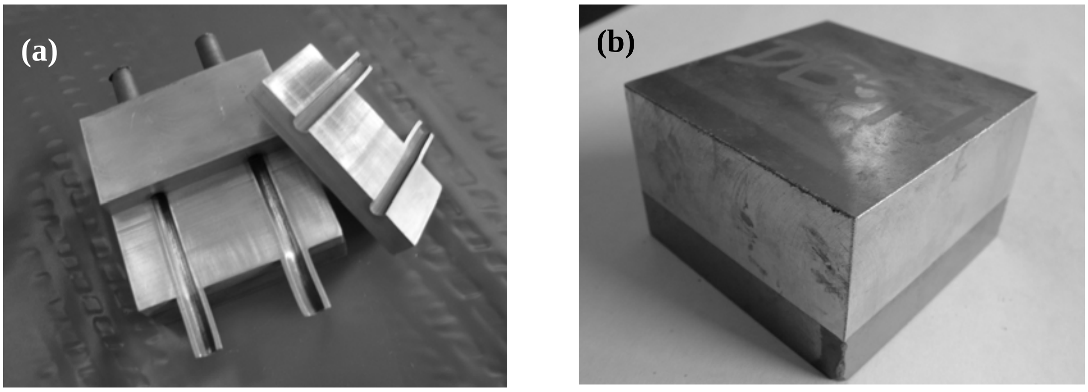

Fig. 7. The Cu/SS (a) and Be/Cu (b) mockups fabricated .by HIP.

New VHP-Be has been produced for the Be/Cu joint according to the ITER requirements. The material has a purity of more than 99% and good ductility. BeO content was controlled in the range of 0.75-0.93% in mass. The total tensile elongation at RT is more than 3%. This VHP-Be has not been used in the previous mockup but will be used in the fabrication of the FW mockups for qualification.

Methods to test the mockup have been being built. One of it is the non-destructive test (NDT) technology for Be/Cu joining. The NDT utilizes ultrasonic to inspect the mockup to find and measure cracks along the bonding area. Calibration Be samples with flat-bottom holes of 0.3-2 mm in diameter were inspected. The holes were taken as the simulation of cracks in the same size. The device for the NDT could find cracks larger than 1mm in diameter. Many Be/Cu joints were inspected with this method before any mechanical tests. The one with less defects usually showed higher bonding strength. Technology to detect cracks in the cylindrical interface of the Cu/SS tube joint is still studying.

Another test of the mockup is the HHFT. A new electron beam facility is under construction for the purpose. The facility has been designed in a scanning heating manner with a frequency of 10hz and a power of 3 kW. Scanning area is more than 80x80mm^2. Thus the facility is capable for the HHFT of the 1/3 of the mockup for qualification. Details could be found in Ref. [4].

5. Summary

Be/Cu joining by HIP has been extensively investigated in China recently. Much progress has been made for a better joining. Both the Be/Ti/Cu/CuCrZr and the direct bonding of Be/CuCrZr exhibited good performances, especially those HIPed at low temperature. Small mockups of CuCrZr/SS joint and Be/CuCrZr have been fabricated with the establishment of the NDT method and HHFT test facility for testing the qualification mockup. The technology will be further studied and be used in the fabrication of the ITER FW panels. With the aid of these studies, following measures should be taken for a better Be/Cu joining.

(1) Ti and Cu coating on Be and CuCrZr substrate, respectively, to thin the interface and mitigate the formation of Be-Cu intermetallic phases.
(2) Lower the HIPing temperature to obtain a thin Be/Cu interface.
(3) Using SA-state CuCrZr alloy instead of the aging-hardened alloy.
(4) Post-HIPing annealing to release the residual stress at the interface.

Acknowledgement

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