ITER first wall fabrication technology in China

J. M Chen 1), X. Liu 1), J. H. Wu 1), L. R. Shen 1), Z. J. Xu 1), N. M. Zhang 1), Y. Liu 1), C.H. Pan 1), X.S. Wang 2), D.H. Xie 2), X.B. Xian 2), P.C. Zhang 2), Z.H. Wang 3), L. Wang 3), Z.N. li 3), Z. Li 3), X.C. Mu 4), Z.J. Wan 4), Y.M. Tang 4)

1) Southwestern Institute of Physics, P.O. Box 432, Chengdu 610041, China
2) China Academy of Engineering Physics, Mianyang 621900, China
3) Ningxia Orient Non-ferrous Metals Group Co. Ltd., Ningxia, China
4) Non-destructive Detect Center, Nuclear Power Institute of China, Chengdu 610041, China.

e-mail contact of main author: chenjm@swip.ac.cn

Abstract. China has done R&Ds on the fabrication technology for ITER first wall (FW). The FW materials, such as the vacuum-hot-pressing (VHP) Be and ITER-grade CuCrZr alloys, have been developed in China. The Be plate showed good physical and mechanical properties, similar to S65C. The effect of fabrication process on mechanical strength of CuCrZr alloy was studied. Several FW qualification (FWQ) mock-ups have been fabricated and tested. Good bonding quality has been achieved between Be, CuCrZr and SS. The RT shear strength of Be/Cu joint has reached up to 244MPa, and no defects of larger than 2 mm in diameter was observed at Be/Cu interface. A high heat flux test (HHFT) of the mock-up shows that the mock-up survived 500 cycles at 1.47 MW/m^2 and a number of cycles at higher power density up to 2.47 MW/m^2 without significant damage.

1. Introduction

China will manufacture 10% ITER FW panels as her in-kind contribution to ITER. The panel is composed of three kinds of material in the forms of plate and tube. They are bound together through diffusion processes for good heat load transfer performance. To ensure the manufactured panels in required quality, the fabrication technology is requested to be qualified by fabrication and testing FW qualification mock-ups and semi-prototype before the procurement, which has been specified by ITER international organization [1]. One way for the bonding is hot isostatic pressing (HIPing). The materials are Be tiles as the top material facing plasma, CuCrZr alloy as the heat sink beneath the tiles and 316L(N) as the back plate. Inner the heat sink there are 316L(N) cooling tubes. During normal operation, the heat load on the Be tiles is about 0.5MW/m^2.

One of the key issues for the fabrication is the formation of Be/Cu intermetallic phases that increase the brittleness of the joint [2], while the key property of the joint is its performance under high heat flux (HHF) that the ITER-FW will face in the normal and abnormal (transient) operation. The main objective of this study is to find ways to reduce the brittle phase formation to increase the performance, and the technology to fabricate mock-ups satisfying the ITER-FW requirements for qualification. China started the study of the joining technology in 2005 [3]. Various interlayers to mitigate the formation of Be-Cu intermetallic phases were studied. By analysis of the interface microstructure and measurement of bonding strength, a relatively optimized joining technology was obtained in 2006. For the Be/Cu joints, the highest bonding strength of 143MPa was achieved at room temperature (RT) in the shear test mode. For the CuCrZr/SS joints, more than 300MPa of ultimate tensile strength was achieved. The Be/Cu joint sample of small size showed good performance in a HHFT [4].

2. Developing materials for ITER FW

2.1 Be tiles for ITER FW

High purity CN-G01 Be blocks were made by VHP in China with impact grinding powder in size of 7-14μm. The VHP was conducted at 1050-1175℃ in vacuum of less than 1×10^{-3} Pa in the pressure range of 10-30MPa. Test samples were cut from the blocks for mechanical and physical analysis. More than 15 samples were chemically analyzed. The maximum and average content of impurities in the samples were listed in Table 1. According to the average values, the purity of the material is about 99.1%.

Table 1 Impurities in CN-G01 Be (ppm in mass).

Impurities	BeO	C	Fe	Al	Si	Ni	Cr	Mn	Cu	Mg	Pb	Zn	Cd	Co	Ag	Li
Maximum	10000	980	500	90	110	38	50	22	78	65	8	110	0.2	7.0	1.36	0.24
Average	7855	566	441	56	100	27	33	15	35	35	5	26	0.15	4.8	0.45	0.2

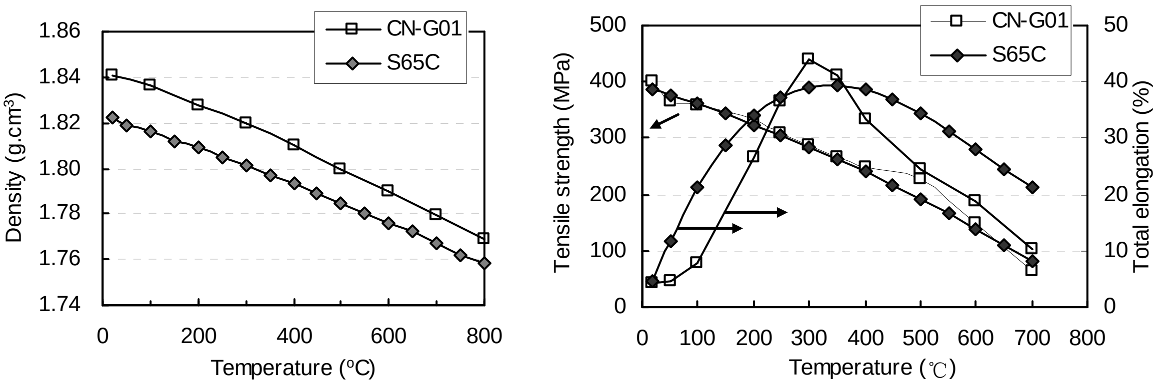


Fig.1 The average density, ultimate tensile strength and total elongation of CN-G01 and S65C. Data of S65C is from Ref. [5].

Physical properties of Young's modulus, specific heat, thermal expansion coefficient, thermal conductivity and density were measured at RT to 800°C. The measured results showed that CN-G01 has similar properties to that of the ITER reference VHP-Be of S65C [5], with an exception of its density. Its tensile properties were also measured at elevated temperature to 700°C. The CN-G01 exhibited higher density, similar strength but lower total elongation at in lower and higher temperature range than ~300°C as shown in figure 1. The minimal total elongation is 2.6% at RT. It is clear that the elongation of S65C changed gently with temperature, while that of CN-G01 changed more drastically, causing a sharp peak around 300°C. The reason for it is not known at the moment and the property needs further improvement.

2.2 CuCrZr alloy

This study aimed at the effect of thermal process of FW mock-up fabrication on the tensile properties of the alloy. Two kinds of CuCrZr alloys were studied, SY-Cu made in China and EU-Cu from Europe. Both meet the ITER requirement of 0.6-0.9%Cr and 0.07-0.15%Zr in mass. In a case, the EU-Cu was forged (EU-Cu-FG). The copper alloy blocks were vacuum canned by stainless steel sheet, followed by HIP1, solid solution annealing (SA), aging (A) or HIP2 plus stress-release annealing (SRA). HIP1 was conducted at 1040^{o}C/130MPa for 2 hrs with furnace cooling. SA had a temperature of 980^{o}C and duration of 1 hr followed a fast cooling by water quench. Aging temperature was 580^{o}C and time was 2 hrs. HIP2 was performed at 580^{o}C/150MPa for 2 hrs. SRA temperature was 400^{o}C and time for it was 4 hrs with furnace cooling. Tensile specimens were cut from the treated blocks and were tested at RT at a strain rate of 3.3x10^{-4}/s according to the Chinese standard of GB/T228-2002.

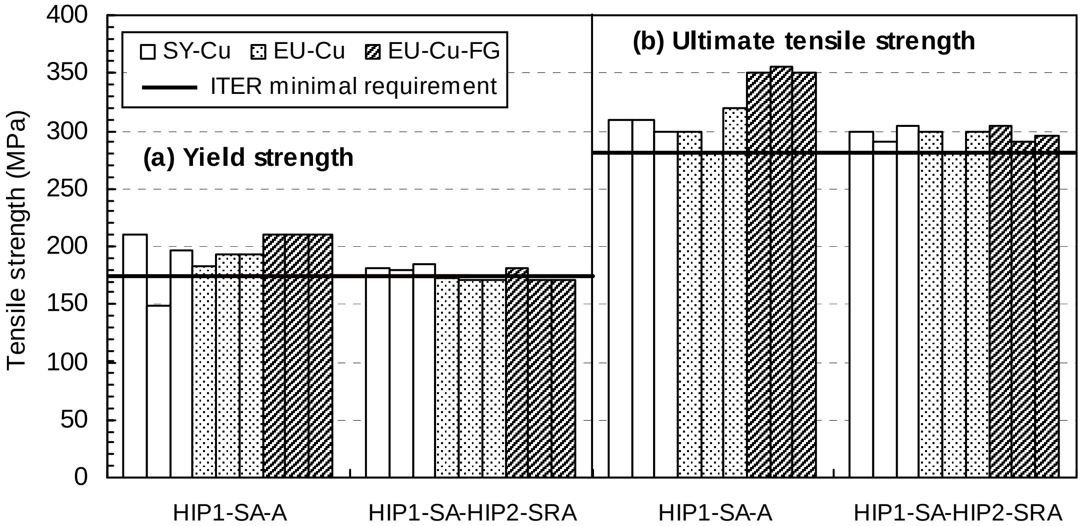
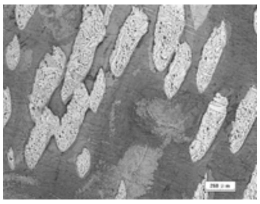
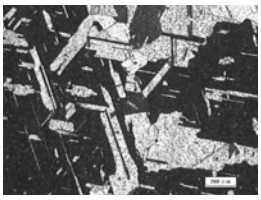
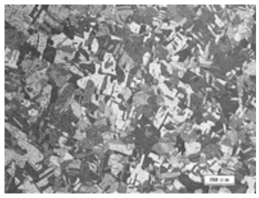
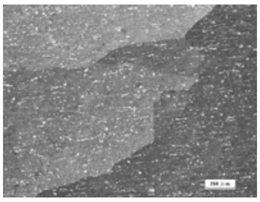
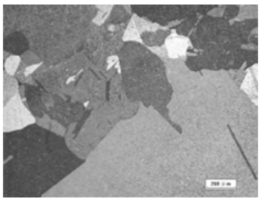
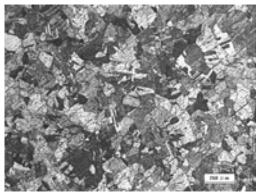


Fig.2 The RT yield strength (a) and ultimate tensile strength (b) of CuCrZr alloys.

Figure 2 shows the tensile strength at RT. No matter what alloy or in which state, after the heat treatment the ultimate tensile strength could satisfy the ITER minimal requirement. The yield strength almost meets the requirement with a few exceptions, for which the deviation is merely several MPa. As the FW mock-up fabrication will undergo the same procedure as HIP1, SA and HIP2, it is thought that these alloys could be used for the qualification mock-up. Another that could be deduced from the figure is that the one after HIP1-SA-A seemed to have higher strength than the one after HIP1-SA-HIP2 and SRA. This may be reasonable because the one in the aging experienced faster cooling than that in HIP2 and SRA.

Table 2 shows the microstructure of the alloys. Both alloys in their as-received condition showed much coarsened grains, larger than 1mm in size for many of them. The grain of the forged EU-Cu was much smaller. It seemed that recrystallization occurred in the forged Cu alloy during the SA heat treatment. According to the photos, only the forged EU-Cu could satisfy the ITER minimal requirement that no grain is larger than 500μm. Its mean grain size is about 130μm after the thermal cycle.

Table 2 The microstructure of the CuCrZr alloys after various thermal treatments.

Treatment	SY-Cu	EU-Cu	EU-Cu-FG
HIP1-SA-A			
HIP1-SA-HIP 2-SRA			

3. FW mock-up fabrication

The CN FWQ mock-up was fabricated by a two-step Hot-Isostatic Pressing (HIPing) method (HIP1 and HIP2 as stated above), together with an intermediate SA treatment at 980^{o}C. Cu/SS joint, including the inner cooling tubes and the tubes to the SS plate, were bound together by HIP1, and three Be tiles were mounted onto the Cu surface of the Cu/SS joint by HIP2. The mock-up has the same structure as IO specified [1], but the slit between Be tiles is 2 mm deeper into the heat sink Cu alloy. The dimension of the mock-up (Be/Cu/SS joining part) is 80x244x84mm^3. Two SS cooling tubes of 12mm in outer diameter are in the Cu alloy heat sink plate, 14 mm down from the Be/Cu interface.

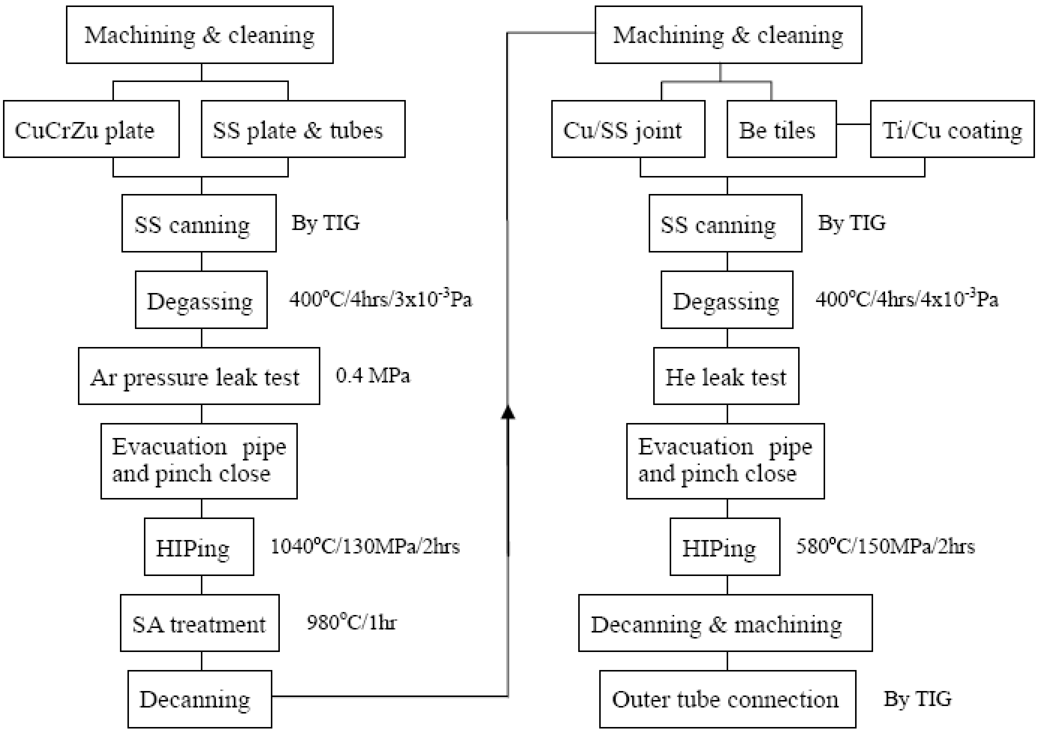


Fig.3 The flow chart for the mock-up fabrication.

Figure 3 shows the CN FWQ mock-up fabrication route. After machining each part of the mock-up, they were cleaned in acid solution to remove the surface oxidized layer, then cleaned in ion-free water, alcohol and acetone. To protect the Cu/SS joint from oxidization in the SA heat treatment, the HIPed canning block was heated and water quenched. For the He leak test, usually a leak rate of lower than $1x10^{-8}Pa/s.m^3$ was acceptable. Ti/Cu coating was placed on the joining surface of Be tiles, first oxygen-free Cu and then Ti by ion plating. The Ti and Cu coating thickness were about 10 and 40μm, respectively. This is a modification to the previous technology in which the Ti was coated on Be and Cu on CuCrZr alloy separately.

The materials used for the CN FWQ mock-up were S65C VHP-Be, EU-Cu in forged state, and 316L plate and tubes. In addition to the mock-up for qualification test, small mock-ups in 1/3 dimension but similar structure were fabricated in the same technology, which was used for a Pre-HHFT. In figure 4 there are two photos showing the structure of the CN FW qualification mock-up and the 1/3 mock-up for HHFT. There are three thermal couple (TC) holes in the FWQ mock-up, drilled from the back of the mock-up toward the center of the Be tiles. The holes terminated at 2mm into Be tile, have a diameter of 1.2mm.

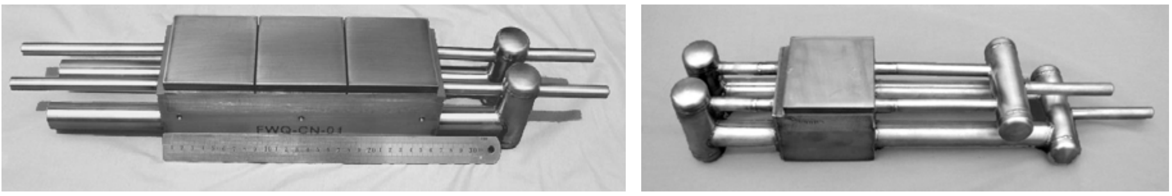


Fig.4 The CN FW qualification mock-up (left) and the HHFT mock-up (right).

4. Testing the mock-up

The tests include non-destructive test (NDT) of the mock-up that will be delivered to the third party for qualification test and destructive tests of spare mock-ups or test coupons.

4.1 NDT

This test contains visual examination of outer surface, the He leak test of the cooling tubes, the pressure examination of the cooling tube connections and the ultra-sonic inspection of Be/Cu bonding interface. So far several mock-ups has been examined at RT by water at pressure of 5MPa for 1 hr, none was failed in the process. The pressure kept unchangeable in the examination. He leak test result showed that the leak rate was lower than $0x10^{-10}Pa/s.m^3$, far away from the required maximum rate of $1x10^{-8}Pa/s.m^3$. So the TIG welding could ensure the quality of the cooling tubes. Ultra-sonic inspection utilized Be calibration blocks with various size flat-bottom holes. Mock-ups were merged in water under an ultra-sonic probe, which has a focused spot of 1mm. The microwave frequency was 10 MHz. In the test, the probe scanned the Be surface to find the defects in the Be/Cu interface. According to the image of the flat –bottom hole in different size, the defect size was estimates. The results showed that no defect at the interface is larger than 1.7mm for one of the mock-ups, others no larger than 2mm. A detailed description of this NDT method could be found in Ref. [3].

4.2 Destructive tests

Be/Cu and Cu/SS interface were observed by scanning electron microscope (SEM) and optical microscope. There found no crack at the interfaces except for a few of <20μm crack at the edge area of the sample, which was thought to be caused by EDM (electrical discharging machining) cutting. The Be/Cu interface looks to have two layers. Good diffusion of Ti into Cu could be identified, while the Cu was successfully barred from the CuCrZr side into the Be tiles. Thus no brittle inter-metallic phase of BeCu could be formed. A shear test of the Be/Cu sample resulted in fracture at Be/Cu interface. XRD analysis of the surface indicated an inter-metallic phase of Ti_3Cu_4. For Cu/SS joint, SEM observation indicated that the interface also contains multi-layers, which is shown in Fig.5. EDS analysis indicated that there was enrichment of Cr and Zr in or adjacent to the interface layer.

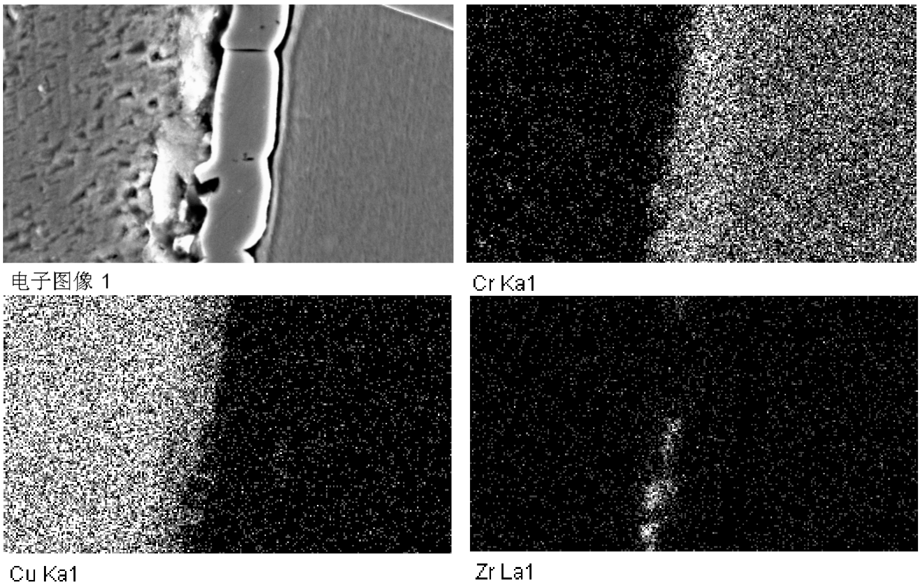


Fig. 5 SEM photo and EDS analysis of Cr, Cu and Zr distribution across Cu/SS interface.

For Be/Cu joint, shear strength was measured at RT. The test specimen was fabricated according to what recommended by IO in the "Standard Test Methods for the Measurement of the Strength Properties of Joints during Manufacturing of the ITER First Wall". The shear test specimens were cut from an additional mock-up to the delivery ones. It has its longitude parallel to the mock-up length direction. Tests were conducted on a CM75105 testing machine. Loading rate was 0.5mm/min for this test. Eight specimens were tested. The measured shear strengths are more than 214MPa and 233.8MPa on average. The shear test curves are shown in Fig. 6. The Highest strength reached to 244MPa.

Tensile test of the Cu/SS joint test coupons that have gone through the whole thermal process of CN FWQ mock-up fabrication was performed at RT. The specimen has a gauge length of 20mm and the diameter is 3mm. The test had a tensile strain rate of 8.3×10^{-3}/s. It was found that fracture always occurred in the CuCrZr alloy region, indicating the Cu/SS bonding strength is higher than the copper alloy. Four specimens have been tested. The average ultimate tensile strength was 266.5MPa, while the minimal value was 241MPa.

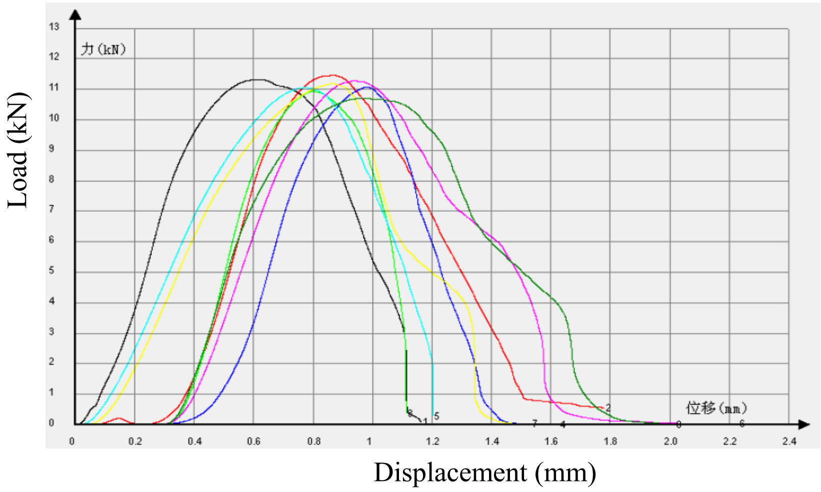


Fig. 6 The Load curves of the shear test of Be/CuCrZr specimens. Test was conducted at RT.

4.3 HHFT of the 1/3 mock-up

The test was conducted in Judith 1，an electron beam facility for HHFT. It started with a screening test from 0.6MW/m^2 to 1.5MW/m^2 by a step of ~0.5MW/m^2, followed by cycling test at 1.47MW/m^2 for 500 cycles, then increased to 1.92MW/m^2 for 10 cycles and finally 2.47MW/m^2 for 2 cycles, each with a prior screening test at a little higher power density. The power density is the absorbed one obtained from water calorimetry. Cooling water flowed from the 10mm diameter cooling channels in the heat sink to the 21mm ones in the SS back plate at RT and the flow rate in the 10mm channel was 5.1m/s. Pulse was set as 60s on and 60s off. The surface temperature of the Be tiles was measured during the cycle.

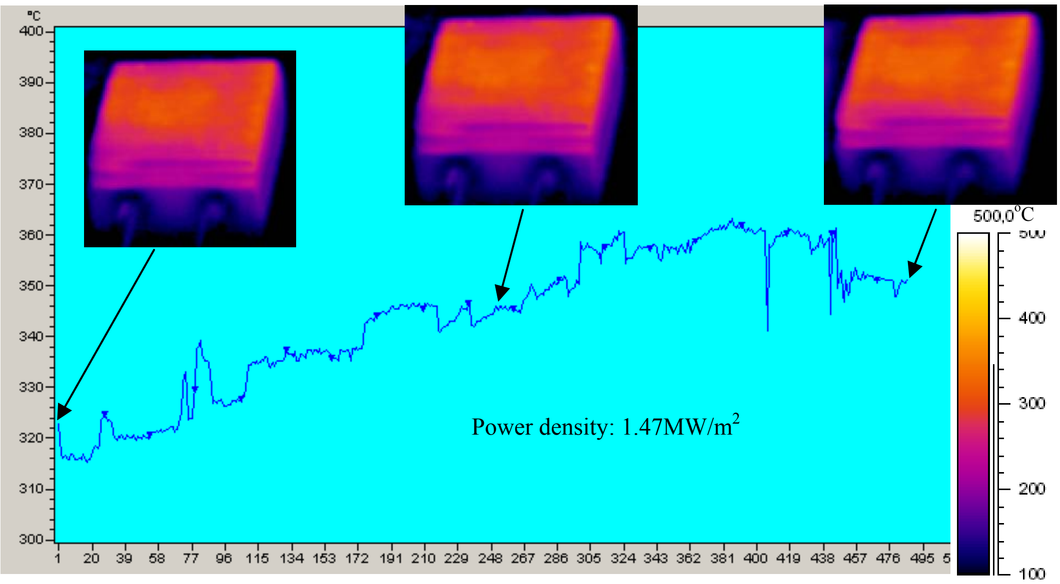


Fig. 7 The maximum surface temperature and the surface image during cycling.

The maximum surface temperature increased with the cycling at 1.47MW/m^2 as shown in figure 7. It seemed that the heat transfer capability decreased during the cycle, possibly implying some changes at the material joining interface. But the temperature didn't exceed

365°C at any cycle. Even after the higher power density cycling as described above, the mock-up still survived according to the nearly homogeneous temperature decreasing during the cooling-down after cycling at 2.47MW/m^2. On the other hand, the tested mock-up was inspected by ultra-sonic to verify the changes at the Be/Cu interface. Result shows many defects in the edge area. As the NDT of the mock-up before the test didn't show any defects, these defects should be produced during the thermal fatigue cycling. Therefore, although the mock-up didn't fail in the test, the cycling did cause damage to the Be/Cu interface.

5. Summary

Several FW mock-ups have been fabricated and tested for the technology qualification. The materials were studied to show the effect of HIPing process on mechanical properties. The HIPing was conducted to join CuCrZr to SS plate and tubes at 1040°C and Be tiles to the HIPed Cu/SS joint at 580°C. The Be/Cu interface didn't show any defects larger than 2 mm in diameter. SEM observation showed no crack at interface, EDS analysis indicates the well diffusion of the material elements but Ti_3Cu_4 formed at Be/Cu interface, while Cr and Zr segregated at Cu/SS interface.

Coating both Ti and Cu on Be tiles was successful, improved the bonding strength of Be/Cu, which reached up to more than 214MPa in shear test mode at RT. The tensile strength of Cu/SS joint was more than 240MPa, higher than that of the Cu alloy. The mechanical properties of CuCrZr alloy varied in the HIPing process. Both EU and China Cu alloy could satisfy the ITER requirement. However, only the alloy used in forged state could meet the requirement to grain size. The Be/Cu/SS mock-up survived 500 cycles at 1.47MW/m^2, 10 cycles at 1.92 MW/m^2 and 2 cycles at 2.47 MW/m^2 didn't cause the failure of the mock-up. Anyhow, there was NDT-identified damages at the Be/Cu interface.

Acknowledgment

The authors would like to thank Dr. Linke and Dr. Rodig in FZJ for their help in the HHFT of Chinese FW mock-up. Dr. Yan Desheng in Institute of Metal Research Chinese Academy of Science in Shengyang supplied one of the CuCrZr alloys for the study.

References

[1] V. Barabash, P. Chappuis, Specification for manufacturing First Wall qualification mock-ups, ITER document, ITER_D_24K TBS v2.0.
[2] ITER Material Assessment Report, Armour/Heat Sink Joining Technology, G 74 MA 10 01-07-11 W 0.2, PP. 1-45.
[3] J.M. Chen, X.S. Wang, et al., Progress towards a better Be/Cu joining for ITER first wall in China, IT/P2-5, IAEA FEC 2006, Oct. 16-21, Chengdu, China.
[4] LIU, X., et al., High Heat Flux Tests of Small-scale Be/Cu Mock-ups for ITER, IT/P2-6, IAEA FEC 2006, Oct. 16-21, Chengdu, China.
[5] ITER Material Assessment Report, Appendix C, G 74 MA 10 01-07-11 W 02, P.12.