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### HIGH HEAT FLUX TESTS OF W-W BRAZED JOINTS FOR DIVERTOR COMPONENTS

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#### Keywords

Tungsten, brazing, fusion reactor, high heat flux, plasma facing component, thermal fatigue

#### Abstract

The divertor component of the future DEMO fusion reactor will be exposed to severe conditions, which includes high thermal loads, neutron irradiation and particle exposure. In order to qualify the materials and joining, it is necessary to expose them to similar service conditions than those expected during the service life of the reactor. In the present work, W-W brazed joints (tungsten block:  $8 \times 8 \times 4$  mm joined to an actively cooled copper heat sink) were exposed to steady state heat loads to study the effect of the thermal fatigue on their microstructure and mechanical integrity. Two different surface temperatures were tested (1000 °C and 1250 °C) varying the number of applied cycles (100 and 1000). The results indicated that a surface temperature of 1000 °C represents the limit condition for the joints to be used in the DEMO fusion reactor, which corresponds to a braze temperature in the range of 562-599 °C according to thermal simulation results. The increase of the surface temperature to 1250 °C caused degradation of the joint and a reduction of sustained cycles and accordingly lifetime.

#### 1. Introduction

The divertor component of the DEMOstration fusion reactor (DEMO) will be exposed the most severe conditions among all plasma facing components of the vacuum vessel. High thermal loads, neutron irradiation and particles exposure will be the most relevant phenomena which the materials have to withstand. In order to select the materials and the joining technologies necessary for the design of the reactor, it is necessary to expose them to similar conditions than those expected during the service life of the reactor. The divertor design proposed by Norajitra et al. included one tile made of tungsten, which will face the plasma joined to a thimble structure made of a tungsten alloy [1, 2]. The joint is thought to be fabricated using an intermediate filler alloy to stop the propagation of the crack formed in the tile during its service life. The selected filler has to meet some requirements such as thermal stability, up to certain temperature, and restrictions in the filler compositions [3].

The present paper proposed 86Fe-14Ti alloy as filler material for W-W brazed joints, whose suitability has been demonstrated in previous works. The filler allows providing high strength joints with a low distortion of the base material properties [4]. In addition, the composition of the filler material fulfills the compositional restrictions to be used in the DEMO reactor.

The simulation of the expected service conditions has been carried out by means of high heat flux (HHF) tests, which is commonly used to reproduce the thermal loads conditions inside the vessel [5, 6]. Norajitra *et al.* also examined the DEMO divertor finger unit under HHF but the results were not completely satisfactory [7, 8]. Later, several papers have been published regarding the impact of the HHF tests on W-CuCrZr, Be-CuCrZr and CFC-CuCrZr joints for their application in the first wall and in the tungsten concept of the ITER divertor obtaining different results [9-11].

The present work aims to evaluate the effect of steady state loads on the quality of the joints by exposing them to different thermal loads. The joints were IR monitored during the tests to detect possible surface overheating and microstructural and mechanical analysis after the tests (shear tests) were made to determine possible changes, diffusion phenomena or phase formation that could degrade the mechanical integrity of the joints.

#### 2. Joint design and HHF tests

The base material used in brazing experiments was tungsten (>99.97 %, *Plansee AG*). The fabrication procedure of the filler consisted in the lamination of a mixture of 86Fe-14Ti powders with an organic binder (powder/binder ratio: 95/5) obtaining flexible tapes of 250  $\mu$ m width [4, 12]. Brazing tests were carried out in a high vacuum furnace (~10<sup>-6</sup> mbar) to avoid oxidation. The brazing temperature was 1350 °C which was kept for 10 min and the heating and cooling rates were 5 °C /min [12].

Afterwards, W-W joints were brazed to a copper cooling structure (heat sink) to ensure the correct refrigeration of the samples during HHF test (Figure 1a). W-W/Cu joints were carried out using a silver base filler tape with a composition Ag-28Cu-2Ge-0.2Ni (wt. %) supplied by Forschungszentrum Jülich (FZJ), applying the following cycle: heating up to 350 °C (5°C/min); holding the temperature for 30 min; heating up to 750 °C (5°C/min); holding the temperature for 15 min; heating up to 815 °C (3 °C/min); holding the temperature for 10 min; cooling down to 550 °C at 5°C/min; and, finally, cooling down to room temperature at 1.5 °C/min. Figure 1b shows a schematic representation and dimensions of the sample and cooling component used for HHF tests.



**Figure 1.** (a) General view of cooling structures with the brazed samples used for the tests. (b) Schematic representation and dimensions of the samples and cooling component used for the tests.

HHF tests were carried out at the electron beam facility JUDITH 1 at FZJ. The samples were individually exposed to the scanning electron beam of approximately 1 mm diameter at full width half maximum and water was used as coolant. The chosen frequencies and all other parameters are shown in Table 1. During the tests the samples were monitored with an IR camera and pyrometers in order to detect surface overheating events caused by a deficient refrigeration or failure of the samples. The heating and cooling times of each cycle were chosen to reach steady state conditions. The vacuum conditions in JUDITH 1 reached an oxygen partial pressure of about  $1 \times 10^{-5}$  mbar.

**Table 1.** HHF testing parameters in JUDITH 1.

A <sub>loading</sub>	64 mm <sup>2</sup>
Scanning	40 × 31
frequency	kHz
Water velocity	14.6 m/s
Water P in	0.5 bar
Water T in	21 °C
Power (on/off)	10/10 s

Samples were exposed to normal operation conditions (steady state loads) because the main interest of the present paper is to analyze the interaction and behavior of the joint interfaces during the normal operation of the reactor. The use of high transient thermal loads corresponding to off-normal operation conditions could lead to partial melting of the material and formation of crack networks along the top surface. However, the influence on the joint interfaces is limited [13, 14].

Samples were tested under different conditions varying the surface temperature (1000 °C and 1250 °C) and the number of cycles applied (100 and 1000). For each condition, four joints were exposed to the electron beam, one for microstructural examination and three to study their mechanical behavior after the test.

The temperature distribution across the joint was studied via FEM simulation with ANSYS software.

Thermal diffusivity tests were carried out at FZJ to analyze the heat transmission properties across the joint using a laser-flash equipment. Thermal diffusivity of three samples with approximately 2-3 mm thickness was measured from room temperature to 1000 °C.

After HHF tests, the microstructure of the joints was analysed by scanning electron microscopy (SEM). The samples were prepared with the standard polishing technique. Some joints were etched by immersion before microscopic observation to develop the tungsten grains and determine the effect of the thermal loads on the grain size of parent material. For it, an etchant solution of 30 ml  $H_2O$ , 10 ml  $H_2O_2$  and 20 ml  $NH_3$  was used.

In order to obtain the mechanical properties of brazed joints, shear tests were carried out using a Universal Testing Machine (*Zwick Z100*). The specimens were tested at room

temperature with a speed of 1 mm/min. For each HHF condition, three samples were sheared and the average shear strength was calculated.

#### **Results and discussion**

The first test was carried out with a W surface temperature of 1000 °C and it was monitored using an IR pyrometer (Figure 2 (a)). The temperature gradient inside the joint from the top surface (heat source) to the heat sink (copper cooling structure) was uniform (Figure 2 (b)).



#### W base material

#### W base material



**Figure 2.** (a) IR image of the samples during the test at 1000 °C surface temperature. (b) Simulation of the temperature distribution across the joint. (c) and (d) SEM micrographs of the joint cross-section after 100 and 1000 cycles, respectively.

At this surface temperature, 100 and 1000 cycles were applied and no surface overheating event was detected during the performance of the test. The micrographs of two specimens after a different number of cycles are shown in Figures 2 (c) and (d), respectively. Both joints showed similar microstructural characteristics, which corresponds to the previous description made of the samples after brazing conditions [4, 15]. The braze was constituted by  $\alpha$ -Fe (dark grey) and Fe<sub>2</sub>Ti (light grey) phases and an interdiffusion layer formed at both interfaces. Therefore, the thermal heat cycles did not produce changes in the microstructure of the joints. Cracks originated at the interface and were also observed across the braze. These cracks had been detected after the brazing cycle and showed a moderate growth caused by the exposure of the joints to thermal fatigue during the cycles. As it was previously reported, the cracks stopped when a more ductile phase was reached. These cracks did not cause a drop of the refrigeration capabilities of the joint because they were parallel to the heat flux direction.

Under these conditions, the braze was in the range of temperatures of 562-599 °C

(Figure 3), which is approximately 700 °C lower than the solidus temperature of the filler alloy ( $T_{sol}$ = 1293 °C [4]), and melting of braze will not occur during the service life of the component. Norajitra *et al.* carried out HHF in thimble-tile mock ups with similar thermal loads (10-12 MW/m<sup>2</sup>) than those applied in the present work, but using He gas at 600 °C as a coolant fluid. The mock ups were tested for a maximum number of 100 cycles using a commercial filler with a solidus temperature of 995 °C [16]. During the test, the temperatures reached by the braze resulted in its melting and subsequently overheating of the top part of the joint (tile). Later, an improved design withstood 1000 cycles under 10 MW/m<sup>2</sup> [17]. However, the filler used for brazing the thimble to the tile contained Ni, and the use of this element in the DEMO reactor is not allowed because it induces swelling when is exposed to a neutron flux [18].





In order to analyze if the selected conditions could lead to recrystallization of tungsten, the specimen subjected to more severe conditions (1000 cycles) was metallographically etched. The general view of the joint showed that the microstructure of the top base material was similar to that of the as-received one and neither evidence of recrystallization nor grain growth of the parent material were observed (Figure 4 (a)). The detailed analysis of the top surface (Figure 4 (b)), which has been exposed to the electron beam, did not show any apparent sign of damage.



**Figure 4.** (a) General view of the etched joint after 1000 cycles at 1000 °C surface temperature. (b) Detail of the top surface.

The 100 cycles specimens at surface temperature of 1250 °C did not report any overheating event during the performance of the test that was satisfactorily finished (Figure 5(a)). Finite element simulation showed a uniform temperature distribution across the joint, similar to the previous case (figure 5 (b)). The temperatures reached by the braze under these conditions were in the range of 686-739 °C, still far from the melting range of the alloy (figure 5 (c)). The refrigeration capabilities of the tested specimens before and after the test were evaluated by analyzing the cooling curve after the stop of the heating source (figure 5 (d)). The results indicated that the refrigeration capabilities remained unaffected after 100 cycles at 1250 °C because the cooling curves of both joints were similar during the first seconds of the cooling stage.





**Figure 5.** (a) IR image of the samples during the test at 1250 °C surface temperature. (b) and (c) Simulation of the temperature distribution across the joint and in the braze, respectively. (d) Cooling curve obtained after the stop of the heating source.

The microstructural characterization of the joint after the test showed a similar microstructure than the one obtained at 1000 °C surface temperature and, therefore, after brazing conditions (figure 6 (a)). However, a detailed analysis of the interface showed cracks propagating between the base material and the diffusion layer (arrow in Figure 6 (b)). The analysis of the cooling curve shown in Figure 5 (d) demonstrated the lack of influence of these cracks in the refrigeration mechanism. Their small size and the low quantity of cracks did not imply a real obstacle for the heat transmission. However, their exposure to longer cycle conditions could cause the formation of new interfacial cracks and the propagation of the already existing ones, which could affect to the refrigeration capabilities of the joint.



**Figure 6.** (a) SEM micrograph of the joint after 100 cycles at 1250 °C. (b) Detail of the W-braze interface.

Different behavior was found for the specimens planned to 1000 cycles at 1250 °C. Samples failed during the screening stage and the tests were stopped. Top W parts were detached from the bottom ones causing a sudden overheating of the surface, detected by the IR camera. The cross section of a failed sample showed that the braze adhered still to the top part and the fracture propagated following the interface formed by the diffusion layer and the bottom base material (Figure 7 (a)). There were some remaining zones with metallic continuity between the braze alloy and the base material. Figure 7 (b) showed that a high interaction of the braze alloy with the base material took place at the interface that was exposed to the more severe conditions (top one). The interaction gave rise to W rich phases and the displacement of the diffusion layer into the braze, both phenomena caused by the partial melting of the braze alloy at the interface. It was caused by limited size of the remaining metallic contact area that was insufficient to remove the thermal load applied over the top part. It occurred during the heating stage of the screening mode due to the thermal stresses induced by the high heating rate. Once the joints failed, surface overheating was detected and the tests were stopped fast enough to produce only partial melting of the braze at the interface.

The fracture surfaces of one of the joints were analyzed by stereoscopic microscope (Figure 7 (c) and (d)). Cracks distributed over the interface of the top part of the joint (Figure 7 (c)) were observed, while the bottom one did not show any evidence of cracks (Figure 7 (d)).





(d)



Figure 7. (a) General view and (b) detail of the cross section of the top part of the joint.(c) and (d) Fracture surfaces of the top and the bottom part of the joint.

The main registered events and microstructural observations associated to W surface and braze temperatures for all tested conditions are summarized in Figure 8. As the W surface temperature increased, the temperature the braze had to withstand also did. According to simulation, the temperature gap between the top and the bottom part of the braze ( $\sim$ 100 µm) was 37 °C for 1000 °C surface temperature condition, while for the 1250 °C condition it was 53 °C. This fact is associated to the thermal barrier effect caused by the lower thermal conductivity of the iron base alloy of the braze with respect to pure tungsten, which could be beneficial to protect inner materials from high temperature exposure. For the most energetic condition, the total temperature reduction in ~4 mm thickness, associated to the top plasma facing part of the joint, and the braze alloy was around 600 °C.



**Figure 8.** Relationship between top W surface and braze temperature versus absorbed power density and main events associated to each condition.

Figure 9 (a) represents the thermal diffusivity of the W-W joints versus temperature. As the temperature increased, the thermal diffusivity decreased. For example, at 21 °C the first W-W joint show a thermal diffusivity of 44 mm<sup>2</sup>/s while at 1000 °C this value decreased down to 37.6 mm<sup>2</sup>/s. As it was previously commented, the thermal diffusivity was lower than that obtained for pure tungsten ( $\alpha = 66.2 \text{ mm}^2/\text{s}$  [19]) due to the thermal barrier effect produced by the iron based alloy of the filler.

Furthermore, a thicker braze layer increases the thermal barrier effect resulting in a lower thermal diffusivity (figure 9 (b)). However, the strong discrepancy in thermal diffusivity of W-W-1 and W-W-2 despite their comparable braze thickness indicates an influence of inhomogeneities within the brazing layer on its thermal properties.



**Figure 9.** Thermal diffusivity of the W-W 86Fe-14Ti 95/5 PPC brazed joints versus (a) temperature and (b) braze thickness at 21 °C.

#### **Mechanical properties**

The shear strength of the joints was evaluated after the HHF tests (Figure 10). The strength values of the joints exposed to a surface temperature of 1000 °C were 25 and 31 MPa for 100 and 1000 cycles, respectively. These are lower values than those obtained by the joints directly after brazing (44 MPa [4]). The drop in strength of the joints was associated with the thermal fatigue caused by the application of fast heating/cooling cycles, which caused their progressive deterioration. However, as it was described in the microstructural characterization of the joint, the increase in the number of cycles (up to 1000) produced moderate growth of cracks across the joint.

Regarding the specimens tested at 1250 °C for 100 cycles, they showed a considerable drop in the strength caused by a higher impact of the test on the joints under these conditions. This significant decrease in the mechanical integrity of the joints (8 MPa) could be attributed to the formation of cracks along the joint, which could play an important role in the initiation and propagation mechanism of fracture, which usually take place through the base material-diffusion layer interface during the shear test.



Figure 10. Shear strength of W-W joints for each HHF tested condition.

#### Conclusions

W-W brazed joints fabricated using 86Fe-14Ti 95/5 PPC as filler alloy were exposed to different conditions under steady state loads. Two different surface temperatures were analyzed (1000 and 1250 °C) for 100 and 1000 cycles.

The results indicated that a surface temperature of 1000 °C represents a limit condition for the joints to be used in the DEMO fusion reactor, which corresponded to a braze

temperature of 562-599 °C obtained by simulation. For this surface temperature, the microstructure of the braze was not affected when the number of cycles increased and the only effect of the thermal fatigue was the moderate growth of the cracks already reported after brazing conditions. After 100 cycles, a drop in the shear strength was observed but, as the number of cycles increased, the mechanical integrity of the joints was no further deteriorated.

Regarding the second tested surface temperature (1250 °C), a different behavior was observed. After 100 cycles, the incipient formation of interfacial cracks between the diffusion layer and the base material was observed. However, it had no influence on the refrigeration capabilities of the joint. Under this condition, the strength of the joints was 8 MPa after 100 cycles showing a fast deterioration of their mechanical integrity. The analysis of the fracture surface indicated that the failure occurred during the heating stage of the cycle due to the thermal stresses, causing a high interaction between the base material and the braze alloy.

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