Journal of Materials Processing Tech. 319 (2023) 118056

Contents lists available at ScienceDirect

Journal of Materials Processing Tech.

journal homepage: www.elsevier.com/locate/jmatprotec

Thermal fatigue response of W-EUROFER brazed joints by the application of High Heat Flux loads

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ARTICLE INFO

Associate Editor: Hui-Ping Wang

Keywords:
Tungsten
Eurofer
Brazing
Fusion reactor
High heat flux
Plasma facing component

ABSTRACT

The thermal fatigue effect on the microstructure and mechanical properties of the joints that form some components of the future fusion reactor is a concern within the scientific community. In this study, we analyze the metallurgical modifications caused by thermal fatigue and their impact on the mechanical properties of tungsten-EUROFER brazed joints (blocks measuring 6 × 6 × 4 mm). We conduct the analysis using an actively cooled mock-up subjected to steady-state thermal loads, which provides valuable information about the operating conditions of the reactor. Three different surface conditions of tungsten were evaluated: 600 °C (2 MW/m^2), 700 °C (2.5 MW/m^2), and 800 °C (3 MW/m^2), with varying numbers of applied cycles ranging from 100 to 1000. Throughout the tests, infrared cameras and pyrometers were used to analyze the thermal behavior of the W-EUROFER joint. At 600 °C and 700 °C target temperatures, no anomalies in the heating and cooling capacity of the W-EUROFER joint were observed. This represents an advancement compared to previous studies that employed Cu20Ti filler, as it demonstrates consistent and efficient cooling capabilities even at surface temperatures of up to 700 °C, without any notable anomalies starting from the previous filler's 500 °C. However, in the case of 800 °C, the test had to be prematurely stopped. Microstructural analysis revealed the formation of cracks in some cases due to the stresses generated by the mismatch in the coefficient of thermal expansion between the materials used. These cracks affected the mechanical integrity of the joint.

1. Introduction

The development of the DEMOnstration fusion reactor (DEMO) is an extraordinary complex engineering project based on the research, development and innovation of the different fields that involve this project. Materials technology for the inner components plays an important role since they have to ensure the necessary stability and conditions for the correct operation of the reactor. Besides, the validation of the developed materials has certain complexity due to the difficulty of reproducing the operation conditions outside a fusion reactor. In addition, materials such as tungsten, for which industrial experience is limited for this application, needs to be validated under the operation requirements for its adequate use in the DEMO reactor. These requirements include resistance to sputtering and high thermal loads for an adequate lifetime during operation, or the possibility of joining to a structural material (i.e., EUROFER) to manufacture the components. In particular, the use of tungsten as sacrificial layer in the first wall involves the research and development of joining technologies to EUROFER to produce part of the First Wall component. The requirements of the joint for this particular application include withstanding temperatures of approximately 500–600 °C, due to the softening effect that Eurofer experiments above this temperature range according to the studies carried out by Rieth et al. (2003), thermal fatigue and generally mechanical stability. According to the studies carried out by (Heuer et al., 2018) who, in order to provide a basis for better understanding of thermally induced stresses and strains in the First Wall, the thermo-mechanical behavior of a water-cooled test component was explored. The contribution provides a simple geometry simulation that allows straightforward comparison of numerical and experimental results. Furthermore, Miyoshi et al. (2020) calculate the heat flows that the different structures of the reactors will have to withstand and found that, in the case of the first wall, it will be subjected up to 8 MW/m^2 in the worst scenery.

The selected joining technology needs to address the qualification of the joint from the operational, metallurgical, and constructive point of view. Thus, it is necessary to study the microstructure, formation

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https://doi.org/10.1016/j.jmatprotec.2023.118056
Received 14 December 2022; Received in revised form 31 May 2023; Accepted 5 June 2023
Available online 8 June 2023

mechanism of the seam/braze, control of the diffusion elements and possible loss of properties during operation conditions.

Copper as fusion reactor material suffers significantly from hydrogen embrittlement under fusion radiation conditions as Fabritsiev and Pokrovsky (1997) have showed, this process is attributed to an effective helium accumulation at the grain boundaries under irradiation. That is why, in this work, only a fine layer of copper is used as a filler material to join tungsten to a reduced activation ferritic/martensitic steel (EUROFER) using the brazing technique. Copper, as filler material, can relieve the residual stresses generated during the cooling stage of the thermal process due to the mismatch in the coefficient of thermal expansion by plastic deformation. To show that Moon et al. (2019) studied the effects of surface and the residual stress of pure copper processed using ultrasonic nanocrystalline surface modification and the low temperature annealing sample showed a decreased in 20% in the tensile strength comparing with other sample without the heat treatment. Besides, copper has an adequate melting point, which allows performing the brazing process with minimal distortion of the EUROFER microstructure. Furthermore, copper achieves good wettability on both base materials ensuring the correct filling of the joint clearance by the molten filler producing low wetting defects and it has been demonstrated as a good option for this application. Li et al. (2017) studied the microstructures and mechanical properties of laser-welded Fe-19Ni-3-Mo-1.5Ti maraging steel joint with copper as a filler, underscoring that the Cu element has a significant effect on wettability because its addition change the diffusion kinetic condition and promote Ni diffusion, resulting in the decrease of the contact angle. Zhang et al. (2014) analyzed the design and fabrication of dissimilar brazed joints between tungsten and fusion relevant materials using a copper based brazing foil studying the effects of diffused elements between filler and based material on the mechanical properties. Liu et al. (2016) investigated brazing of tungsten/steel using Ta and Cu interlayer with Ni-based amorphous foil filler getting a reliable bonding between W and steel. They found that fracture surface occurred in the W/filler interface, near the braze affected zone.

The operational and metallurgical qualification of the W-EUROFER joint using Cu as filler has been performed in previous works, where microstructural and mechanical characterization were carried out by de Prado et al. (2020). Those works described the consecution of high continuity interfaces, where tungsten is involved in the formation of different reaction layers. The mechanical properties of the joint, measured by shear tests, were in the range of 309 MPa, which are highly influenced by the different studied parameters. This work demonstrated its suitability for this type of applications. Izaguirre et al. (2022) completed this study by a TEM characterization of the joint interface, the study revealed the formation of different phases associated to the brazing process, which enhanced the adhesion properties of the materials to be joined.

Furthermore, by the application of this brazing process and the subsequent tempering treatment, necessary to recover the microstructural and hardness properties of the EUROFER material, it is possible to achieve a shear strength higher than 100 MPa and recover the EUROFER hardness in the as received conditions (220 HV) without any modification in the tungsten base material properties according to the studies carried out by de Prado et al. (2016a).

However, it is still needed to characterize the joint under relevant reactor conditions. The tests carried out in this work, High Heat Flux (HHF) tests, simulate the stationary heat fluxes during steady state operation of the reactor, allowing to study how the temperature gradient inside the joint and the thermal fatigue affect the microstructural and mechanical properties of the joint. Therefore, it is a valuable tool to validate and qualify Plasma Facing Materials (PFM) and Components (PFC) for fusion reactor applications.

This test is commonly performed using a mock-up sample configuration for the material and joint characterization. For example, Norajitra et al. (2009) used this test to study the design of the helium-cooled divertor for DEMO of W/W alloy joints and EUROFER steel. Pintsuk et al. (2021) tested the joint W-CuCrZr with a copper interlayer by thermal cycling at 10 MW/m^2 and 20 MW/m^2 with a maximum of 1200 cycles without an obvious damage formation. In the same direction, Bang et al. (2022) designed small mock-ups manufactured by hot radial pressing of W/Cu monoblock and underwent high heat flux of 10 MW/m^2 up to 5000 cycles with no defects on the interfaces. Kim et al. (2021) analyzed how the microstructure and the mechanical properties of tungsten monoblocks in samples containing small defects at the brazing-bonded interface change. The joints failed after 4000 cycles during HHF tests and showing more pronounced damages on the tungsten armor.

In previous works of our research group carried out by de Prado et al. (2018), a similar study was conducted in W-EUROFER joints by using 80Cu-Ti filler material. The joints were subjected to tungsten surface temperatures of 400, 500 and 600 °C, varying the number of applied cycles from 100 to 1000 demonstrating that the braze temperature of 359 ºC was the threshold condition. The increase of the braze temperature caused the loss of the mechanical integrity of the joint failing as a cooling structure and decreasing the shear strength of the joint.

The present work aims to evaluate the effect of the steady state loads on the quality of W-EUROFER joints by exposing them to different thermal loads. Thus, the joints were monitored with IR and pyrometers during the tests to detect possible overheating of the surface associated to the joint or material defects. After testing, the joints were subjected to microstructural and mechanical analysis (shear test) to determine possible changes, diffusion phenomena or phases formation, which could limit the mechanical integrity of the joints, making them unsuitable.

2. Joint design and HHF tests

The base materials used for brazing were: i) tungsten plates (> 99.97%, Plansee) and ii) reduced activation ferritic/martensitic steel (EUROFER) with a chemical composition in wt% of 0.11 C, 8.9Cr, 0.42Mn, 0.19 V, 1.10 W, 0.14Ta balanced Fe and with the following heat treatments 979 °C / 1 h 51 min /air cooled plus 739 °C / 3 h 42 min /air cooled (de Prado et al., 2016a, 2016b). The sizes of the base materials were blocks of 6 × 6 mm^2 exposed surface area and thickness of 4 mm. The intermediate material used as filler was pure Cu (>99.9%) supplied by *Lucas Milhaupt* in strip form of 50 µm thickness. The strip was cut with the exposed base material surface dimensions and placed between both specimens. Prior to the brazing tests, exposed surface of both base materials was ground down with 4000 grit silicon carbide paper. The brazing tests were carried out in a high vacuum furnace to avoid oxidation with a residual pressure of 10^{-4} Pa. The brazing cycle consisted of heating up to 1135 °C for 10 min and cooling to room temperature using heating and cooling ramps of 5 ºC/min. After that, a post brazing heat treatment or tempering treatment consisting of 760 °C during 90 min was applied (de Prado et al., 2020). The purpose of this heat treatment is to recover the tempered martensite characteristic of the EUROFER and its hardness (216 HV (Fernández et al., 2001)) before the brazing process. In addition, prior to HHF tests, samples (with the exception of one sample per batch that acts as a reference, see Table 1) were subjected to a thermal aging treatment of 500 °C for 1000 h. This

Table 1
HHF tests conditions for W-EUROFER joints.

Batch	W Surface target Temperature (°C)	Power density (MW/m^2)	Number of cycles
1	600	2	100
2	600	2	1000
3	700	2,5	100
4	700	2,5	1000
5	800	3	100
6	800	3	1000

simulated the permanence of the joint inside the reactor.

To perform the HHF tests, the samples were joined to an actively cooled copper cooling structure (heat sink) connected to a water pipe. To ensure the metallic continuity between the samples and cooling structure necessary for cooling the samples, brazing was selected as joining technique (Li et al., 2020). The filler material used in this case was a tape-shaped filler supplied by *STELLA WELDINGS ALLOYS* with the following composition (wt%): 56Ag-22Cu-17Zn-5Sn. The applied brazing treatment was heating from room temperature to 775 °C (5 °C / min), holding the temperature for 10 min and then cooling to room temperature (5 °C / min). The high brazing temperature used in this case with respect to the liquidus one respond to the observation of wetting problems of this filler alloy in preliminary tests. This problem was solved by using temperatures of 100 ºC above the liquidus one (655 ºC). The results of the complete mock-ups for the HHF tests can be seen in Fig. 1a. Furthermore, in Fig. 1b it is possible to see a schematic figure of the HHF tests.

The HHF tests were carried out at the JUDITH 2 electron beam facility at Forschungszentrum Jülich (FZJ). Cooling water with an inlet pressure of 2 MPa at 27 °C and a nominal flow of 60 l/min was used to cool the samples. The tests were monitored with an IR camera and pyrometers to determine the surface temperature, adjust the applied power density and analyze the possible overheating of the surface caused by poor cooling of the samples. IR camera temperature values were cross calibrated with a two-color pyrometer, but since samples can have different emissivity, calibration was done on a specific sample and temperatures should be taken with caution. Cross calibration was performed at the beginning of an experiment, so changes in emissivity during loading can cause a deviation between the two-color pyrometer values and the IR camera values. The heating and cooling cycles of the HHF tests (10/12 s, respectively) were chosen to achieve thermal steady state. The vacuum conditions in JUDITH 2 reached an oxygen partial pressure of approximately 5×10^{-3} Pa.

A total number of 30 joints were tested divided in 6 batches. Each batch consisted on 5 samples. One of them acted as reference without thermal aging treatment and within the four aged samples, one was destined to microstructural analysis and 3 for determining the mechanical properties (shear tests). Each batch was subjected to different conditions varying the W surface temperature (600 °C, 700 °C and 800 °C) and the number of applied cycles (100 and 1000). The power densities for these T_{surf} were approximately 2, 2.5, and 3 MW/m^2. The conditions of all experiments are summarized in Table 1.

After HHF testing, the W-EUROFER joints were separated from the copper cooling structure. Microstructural samples were prepared using standard polishing technique until 1 μm diamond particle and examined with a scanning electron microscope (SEM) (*S3400 Hitachi*) with EDS detector and software included. For some samples, a stereoscopic microscope *(Leica DFC320)* was used. The shear strength values were obtained using a compression device specifically designed to ensure the correct alignment of the brazed joint with the applied load, avoiding bending of the specimens during the test, which guaranteed a pure shear stress on the brazed surface, more detailed information of the test could be found in de Prado et al. (2017). The fixture was placed between the compression plates on a Universal Testing Machine (*Zwick Z100*) at a displacement rate of 1 mm/min. The possible effect of the brazing thermal cycle on the mechanical properties of the base materials was evaluated by means of Vickers microhardness. Thus, microhardness profiles from EUROFER side to the tungsten one, across the braze joint were traced with an *MHV-2SHIMADZU* equipment. A 100 g load was applied during 15 s and three indentation tests were made per distance measured from the center of the joint. The separation between neighboring indentations was always kept larger than three times the residual imprint sizes.

The distribution of temperature across the joint and in the braze joint was calculated by Finite Element Modeling (FEM) simulation with *ANSYS* software using the material data described at the beginning of Section 2. A schematic of the samples brazed to the cooling structure used in the simulation is shown in Fig. 2, where the mesh used to calculate the different parameters are also shown. Boundary conditions applied for the FEM simulation are: 1) Eurofer surface temperature in contact with the copper cooling structure was set to 25 ºC, which is the temperature of the cooling fluid used. 2) Exposed tungsten surface to the electron flux was set to 600, 700 and 800 ºC temperature. 3) The thermal conductivity used for the FEM simulations are represented in Fig. 2a. The data were collected from the following sources Lassner and Schubert (1999); Linsmeier et al. (2017); Rieth et al. (2003), where they studied the physical, thermal and mechanical properties of copper, tungsten and EUROFER materials with temperature. Fig. 2c shows the mesh distribution developed for the FEM simulation.

3. Results and discussion

3.1. HHF tests

Table 2 shows a summary of the HHF test results divided by batches and conditions. The tests began with a W surface temperature of 600 ºC. According to the thermal simulation carried out showed in Fig. 3a 1 and 2, this surface temperature corresponds to approximately 498 ºC at the braze area. In order to be more accurate when discussing the effect of the different surface temperature in the joint properties and microstructure, the following nomenclature will be used for each HHF condition: surface temperature/joint temperature/heat flux (600 ºC/498 ºC;2 MW/m^2, in this condition).

Some thermal anomalies were detected during the application of this condition by the IR camera (Fig. 4a). During the calibration of the pyrometer at heat fluxes lower than 1 MW/m^2 sample #3 was detached and removed from the cooling structure (Fig. 4c). Sample #2 showed increasing temperature with every cycle, exceeding 800 °C around cycle 40, but the cool down behavior indicated that the sample did not detach completely from the cooling structure. Although at cycle 85 the

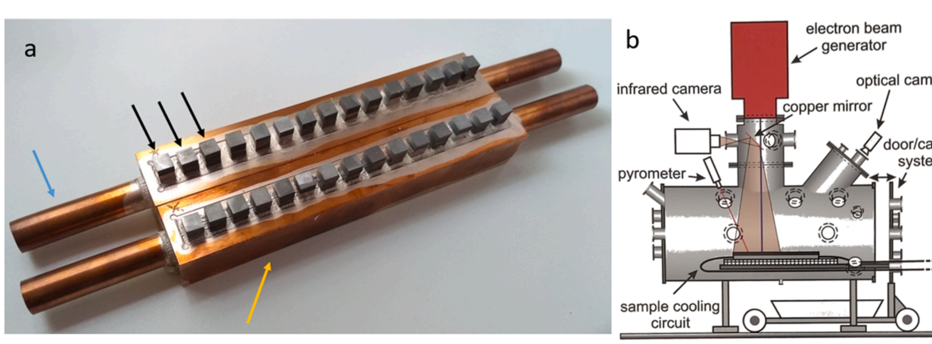

Fig. 1. General view of cooling structure with brazed samples used for HHF test with the water pipe (blue arrow), the cooling structure (yellow arrow) and the samples (black arrows). b) Schematic figure of HHF test.

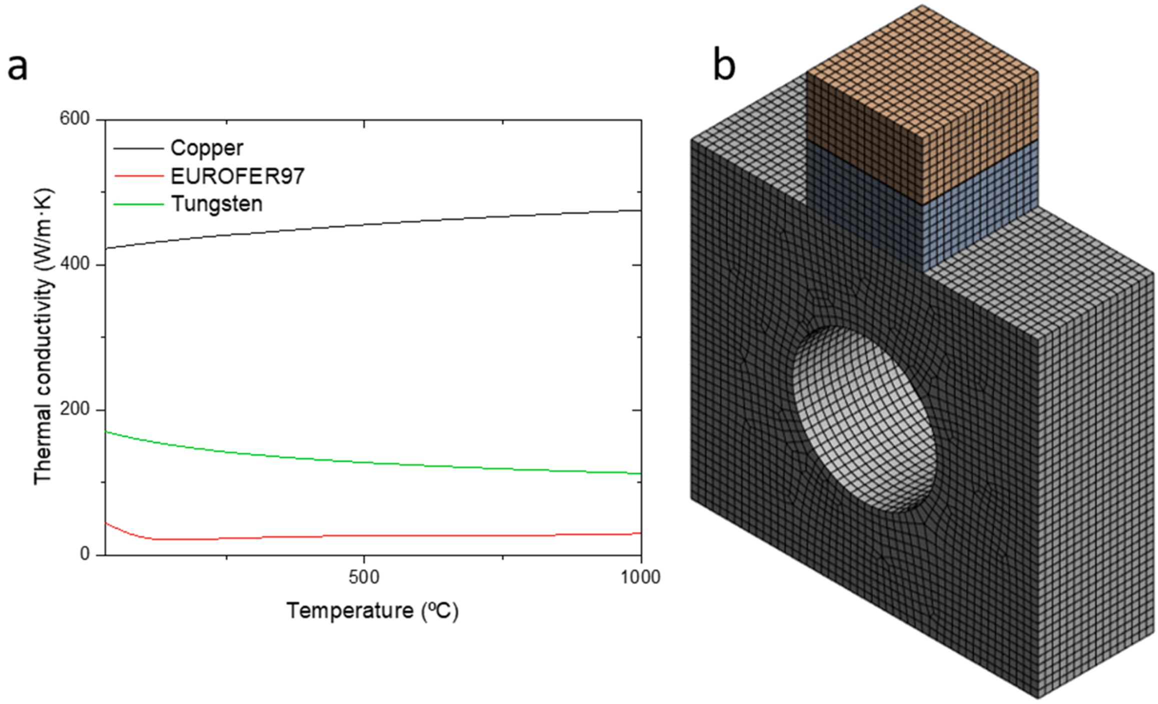

Fig. 2. a) Thermal conductivity used for the FEM simulation of HHF tests. b) Example of the mesh distribution developed for the FEM simulation.

overheating was around 850 ºC, the planned cycles could be applied. After that, the sample could be easily removed from the cooling structure. In both cases, the detachment from the cooling structure occurred, apparently, in the cooling structure/EUROFER joint instead of the evaluated W/EUROFER joint. However, in order to study in detail, the cause of the failure, samples that presented anomalies during the HHF tests were prepared for a metallographic examination. The application of 1000 cycles at the same W surface temperature to the next batch did not report any anomaly.

No anomalies during the test at 700 ºC were reported neither for 100 cycles nor for 1000 cycles. The FEM simulation showed that braze area was subjected, under this condition, to approximately 578 ºC (Fig. 3 b2).

The application of 800 ºC W surface temperature during 100 cycles of batch number 5 did not report special events with the exception of a slight overheating of sample #23, but all cycles could be successfully applied to the whole batch. However, when 1000 cycles were applied to batch number 6, several problems occurred during the test. Sample #29 started to overheat from the beginning of the test and was removed at cycle 25 after reaching 1200 ºC. Sample #26 started overheating at cycle 77, but could not be removed since the sample was still attached to the cooling structure, which could be an indication that, in contrast to the previous cases, the W/EUROFER failed and prevented the proper cooling of the heated W. At cycle 182 the samples #26, #27 and #28 overheated up to 1500 ºC and started to show poor cooling down behavior. At this point the test was stopped. Once samples cooled down to RT, it the fracture of the W-EUROFER joint in samples #26 and #27 was noticed. The EUROFER base material was still attached to the cooling structure, but the tungsten could be removed manually.

The simulation of the temperature distribution in the component carried out at this condition shown in Figure 3c2 indicated that the filler reached approximately 656 ºC. It has to be noted that the temperature distribution on the top of the EUROFER was in the range of 550–650 ºC and according to the studies carried out by Rieth et al. (2021) EUROFER starts to soften at approximately 550 ºC by stress relieving process. Although these high temperatures only affect the nearest area of the EUROFER close to the joint (1–2 mm) they should be avoided during service operation. Their effects should be addressed in future works for a better understanding of their impact on the material properties under undesirable phenomena of the reactor operation such as plasma disruptions. From the point of view of the joint quality, previous studies by Tejado et al. (2020) indicated that the joint can withstand temperatures according to the three-point bending test study from RT to 600 ºC. The results of this study demonstrated that the tempering treatment applied after the brazing cycle involved that the strength of the joint did not significantly change in the studied temperature range.

3.2. *Microstructural analysis*

Fig. 5a-b and 5c-d show the micrograph of the samples corresponding to batch 1 and 2 subjected to a W surface temperature of 600/498 °C, 2 MW/m^2 for 100 and 1000 cycles, respectively. The microstructure of the joints was similar in all cases and no differences between the number of applied cycles were found (Fig. 5b and 5d subjected to 100 and 1000 cycles, respectively). Besides, reference samples (not subjected to aging treatment) did not show microstructural differences in any case (Fig. 5a and 5c for 100 and 1000 applied cycles, respectively).

The braze area was characterized by the formation of a diffusion layer at the W surface with 62W23Fe11Cu3Cr % wt. composition (I in Fig. 5a). Above this layer, the formation of a Fe-rich band has been identified (II in Fig. 5a). The formation of this Fe-rich band between W and copper braze is attributed to the partial solution of iron in the copper filler during the melting stage of this material at the brazing temperature. During the cooling stage of the process, the decrease of iron solubility in copper produces the iron-rich band solidification in contact with tungsten following a heterogeneous solidification mechanism. Copper remained in the braze forming a continuous band above the iron rich band (III in Fig. 5a). This copper band has difference thickness depending on the location in the joint being thicker in the in edges and thinner in the center of the joint (Fig. 5a and b, respectively). Braze exudation process, associated to its high wettability in the system, is the responsible of this phenomenon. During the brazing process, copper also penetrates into the prior austenitic grain boundaries of EUROFER (IV in Fig. 5a).

This microstructure agrees with that obtained after the brazing and

Table 2
Summary of phenomena occurred during HHF tests.

Batch	W surface target T Bond surface T Power density	Sample	Aging	Cycles applied	Additional information
1	600 °C	#1	No	100	-
	498 °C	#2	Yes	100	Overheating above 800 °C around cycle 40
	2 MW/m^2				
		#3	Yes	0	Overheating and extraction of sample
		#4	Yes	100	-
		#5	Yes	100	-
2	600 °C	#6	No	1000	-
	498 °C	#7	Yes	1000	-
	2 MW/m^2	#8	Yes	1000	-
		#9	Yes	1000	-
		#10	Yes	1000	-
3	700 °C	#11	No	100	-
	578 °C	#12	Yes	100	-
	2.5 MW/m^2	#13	Yes	100	-
		#14	Yes	100	-
		#15	Yes	100	-
4	700 °C	#16	No	1000	-
	578 °C	#17	Yes	1000	-
	2.5 MW/m^2	#18	Yes	1000	-
		#19	Yes	1000	-
		#10	Yes	1000	-
5	800 °C	#21	No	100	-
	656 °C	#22	Yes	100	-
	3 MW/m^2	#23	Yes	100	Slight temperature rise above 800 °C
		#24	Yes	100	-
		#25	Yes	100	-
6	800 °C 656 °C 3 MW/m^2	#26	No	182	Overheating at cycle 77 and temperature rise above 1500 °C. Fracture of W-EUROFER joint
		#27	Yes	182	Temperature rise above 1500 °C and fracture of W-EUROFER
		#28	Yes	182	Temperature rise above 1600 °C
		#29	Yes	25	Temperature rise up to 1200 °C (sample removed at cycle 25)
		#30	Yes	182	-

tempering processes and the reference one without aging process as showed the study carried out by de Prado et al. (2020). Therefore, neither the aging treatment nor the application of the HHF loads modified the microstructure with respect to the "as brazed" condition and, most relevant, the metallic continuity is maintained, keeping intact its capacity to conduct the heat loads coming from the simulated plasma reactor chamber.

Micrographs of the samples that showed anomalies during the tests, and were detached from the cooling structures, did not show cracks at the W-EUROFER braze area that could explain the overheating registered by the IR cameras (Fig. 6). Although isolated porosity was detected in the copper braze zone (arrowed in Fig. 6a) it was not the reason that caused the anomalies. Therefore, the overheating should be attributed to the detachment from the cooling structure.

Although the application of 700/578 °C, 2.5 MW/m^2 at the surface temperature did not produce any overheating event during the test detected by the IR camera for any batch, the microstructural characterization of the selected samples showed the presence of cracks, only in the sample subjected to 1000 cycles (Fig. 7c and d), however the samples subjected to 100 cycles did not show any microstructural modification (Fig. 7a and b). The crack was located at the interface between the iron rich region and the copper band.

However, samples did not report any sign of bad cooling down stage according to the information provided by the IR cameras and pyrometers, therefore, it could be assumed that, in samples that cracks have been reported, there were still enough metallic continuity to ensure the heat flux transmission from the hot zone to the coolant.

This achievement contrasts with previous HHF studies, also in W-EUROFER joints, where de de Prado et al., (2018) used a Cu-Ti alloy as filler material and the maximum surface temperature achieved without thermal anomalies was 500 °C. This difference could be associated to different phenomena. The resultant microstructure in this case does not contain brittle phases, which under the thermal fatigue stress could easily propagate cracks and consequently produce the fracture of the joint. In addition, the brazing conditions gave rise to the interaction of the W base material with the braze, which could enhance the adhesion properties of in this zone. This las phenomenon is a key since according to simulation the stress is accumulated at the W-braze interface and progressive interfaces could better distributed the stress obtaining better HHF results. In the case of de Prado et al. (2018) when welding at 960ºC, the thermal, physical and chemical properties of W hindering further interaction with the braze.

Microstructural characterization of samples subjected to 800/656 °C, 3 MW/m^2 showed different behavior depending on the exposed cycles. In the case of the samples of batch 5, subjected to 100 cycles, the microstructure did not show relevant modification (Fig. 8a and b). However, in the case of the 1000 cycles planned for the batch 6, the test was stopped due to the surface overheating after 182 cycles. The preliminary visual examination indicated the fracture of the W-EUROFER joints of two samples (#26 and #27) and the detachment of the W piece from the joint.

The microstructural examination of the exposed joints showed large cracks distributed parallel to the brazed joint at the iron rich region-copper band interface (arrowed in Fig. 8c). The formation of these cracks was associated to the high thermal stresses caused by the test as a consequence of the difference in the Coefficient of Thermal Expansion (CTE) among EUROFER (10–12 $\times10^{-6}$/K) (Mergia and Boukos, 2008), tungsten (4.5 $\times10^{-6}$/K) (Zhong et al., 2010) and copper (16.5 $\times10^{-6}$/K) (Ghovanlou et al., 2011). Each cycle induced residual stresses in the braze area giving rise to a thermal fatigue process. The more the temperature and cycles applied the more the stress is induced, up to some point in which the joint starts to plasticize (residual stress reach the yield point of the softest material) or forming cracks. The formation of cracks parallel to the joint is the logical consequence due to the stresses are maximized at the interface of both materials. This crack did not allow transferring the heat flux from the hot area to the coolant and, therefore, the upper part of the sample overheated.

Fracture surface of the two samples, where tungsten was detached from the joint, (#16 and #17), were exanimated by SEM to analyze the failure. Fig. 9a shows the fracture surface of EUROFER base material of sample #16 analyzed by stereoscopic microscope and Fig. 10b shows a detailed inspection of the inner zone carried out by SEM. In this image, it can be distinguished particles with drop morphology compatible with a partial melting of iron caused by the temperature increase, according to the elemental mapping distribution shown in Fig. 8c. The formation of discontinuities, similar of that studied in previous samples, would be responsible of the initial increase of temperature. The higher temperature resulted in higher thermal stresses until cracks propagated causing the critical failure of the joint and finally its complete fracture. The SEM analysis also shows, in Fig. 9b/c, the presence of tungsten material detached from the base material during the fracture, which indicated the good adhesion properties of the brazed joint.

A deeper understanding of the failure process could be extracted from Fig. 10a and 10b, where a cross section of EUROFER fractured piece of sample #16 is shown. According to the images, fracture occurred between the region rich in Fe and the diffusion layer. However, transversal cracks that connected the fracture surface with the copper band are observed. Those cracks suggest that this iron rich layer could propagate, under certain circumstances, the cracks generated in the

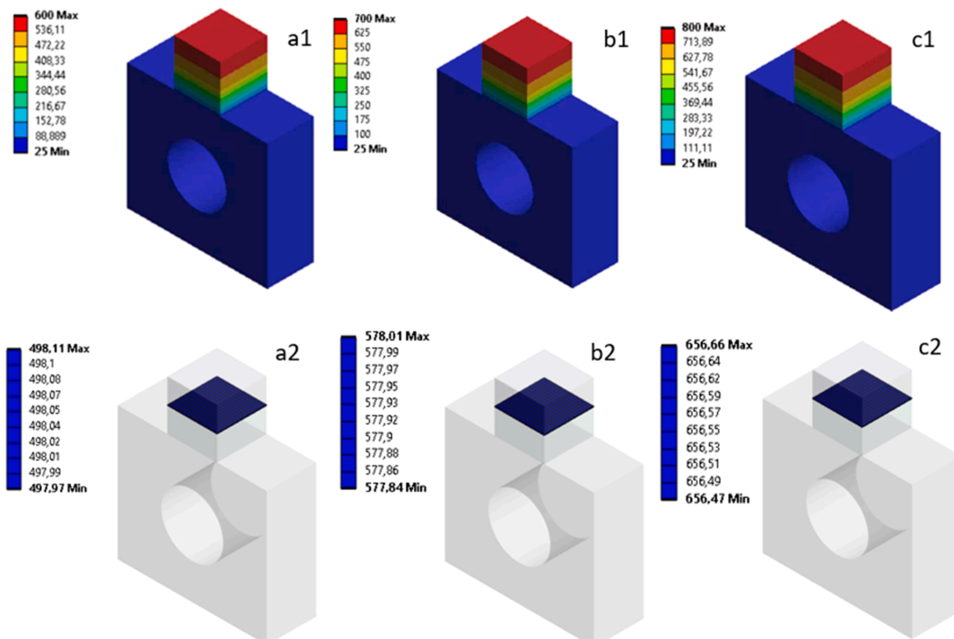

Fig. 3. Simulation of the temperature distribution in the component at the steady state stage at (a) 600 °C, (b) 700 °C and (c) 800 °C of W surface temperature. a2, b2 and c2 are simulation of the temperture of the filler in each condition.

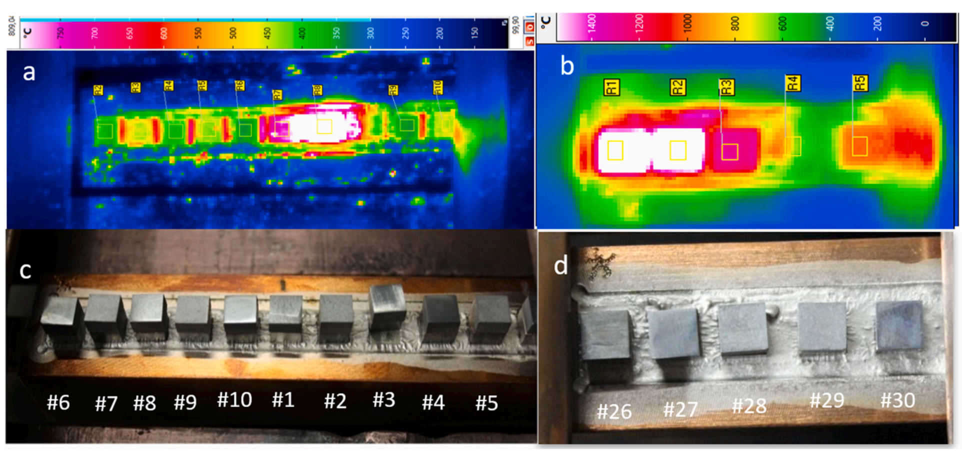

Fig. 4. (a) and (b) IR images showing overheating of samples in batches 1 and 6, respectively. (c) optical images after detachment of #3 (after heat fluxes of < 1 MW/m^2) and (d) before the test.

braze area. Besides, as it was studied, this phase is involved in the generation of longitudinal and transversal cracks, possibly due to its location between the EUROFER and tungsten, that generates the stresses by the mismatch in the CTE. Therefore, future efforts should be focused in avoiding the generation of this zone by controlling the brazing parameters. This should lead to a braze joint with copper in contact to tungsten that, in principle, could avoid the propagation of cracks due to its ductile character.

3.3. Mechanical properties

Shear strength and hardness of the joints were evaluated after the HHF tests to analyze the impact of the thermal fatigue on these mechanical properties.

The shear strength of the joints is shown in Fig. 11. The shear strength of the sample tested at 600/498 °C, 2 MW/m^2 and 100 cycles is around 275 MPa, in this case only one sample survived to this condition, the other were examined by SEM to study the failure mechanisms, as was previously discussed. Only one sample could be tested because the other two were removed from the cooling structure during the cycling test. The shear strength dropped down to 50 MPa after 1000 cycles. This value is lower than that reported for joints at as-brazing conditions (225 MPa) because of the formation of cracks at the edges of the joint between the Cu band and the Fe rich region. This effect was intensified as the W surface temperature and the number of cycles was increased. It was an expected behavior since the temperature gradient between the hot W surface and the cooling copper structure increases with the surface temperature and, therefore, the stresses generated by the mismatch of the CTE also do. These stresses are generated and accumulated in each cycle generating residual stresses and cracks that contribute to the reduction of the strength of the joint.

A representative example obtained by stereoscopic microscopy of the joint fracture surface after shear test is shown in Fig. 12a. All samples showed similar fracture pattern consisting of two concentric rings located approximately at the center of the surface. These rings, which showed different tonalities in the optical images (Fig. 12a), represented

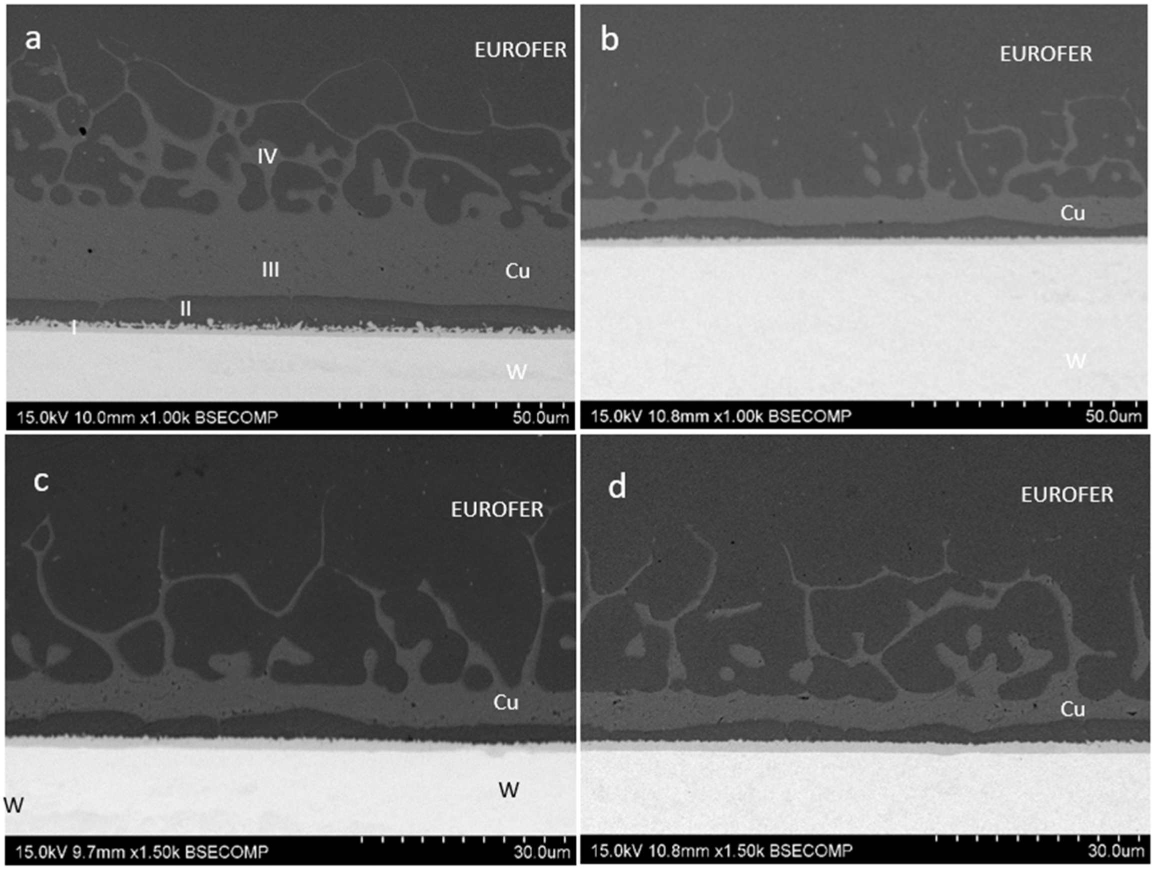

Fig. 5. Micrographs of samples subjected to a W surface temperature of 600 °C with the following conditions: (a) 100 cycles, (b) 100 cycles and aging treatment, (c) 1000 cycles and (d) 1000 cycles and aging treatment.

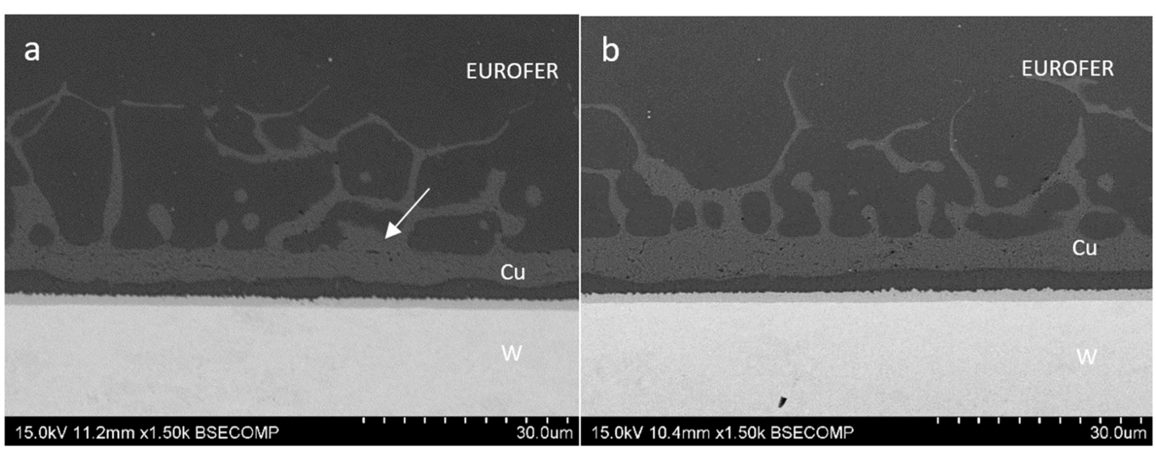

Fig. 6. Micrographs of samples (a) #2 and (b) #3 that showed anomalies during HHF tests.

different propagation mechanism of the crack during the failure propagation.

A detailed analysis of the cross sections fracture surface at different zones following the lines depicted in Fig. 12a shows that in the outermost ring (Fig. 12a and 12c), the fracture propagated following the copper band-Fe rich region interface. In this case, copper remained adhered to the EUROFER base material giving to it this characteristic tonality in the images. Fig. 12a and 12c were taken in the zone where fracture mechanism shifted from the mechanism explained above to the one that dominated in the inner ring (Fig. 12c). In this case, the crack propagated following the diffusion layer – Fe rich region interface. One explanation of this behavior is that cracks nucleated at the copper band-Fe rich region interface, as the microstructural study demonstrated by the observation carried out in samples before the shear tests (Fig. 13a and 13b). Their formation occurred during the HHF tests mainly in the outermost part of the joint, where the presence of the copper band is thicker and continuous. Then, the crack propagated during the early stage of the shear test. However, once the crack reached the inner part of the joint, the copper band was thinner and, at some point, discontinuous. Therefore, the propagation could not follow this interface and shifted to the diffusion layer-Fe rich region interface. The cross-section examination demonstrated that in this zone, the diffusion layer and

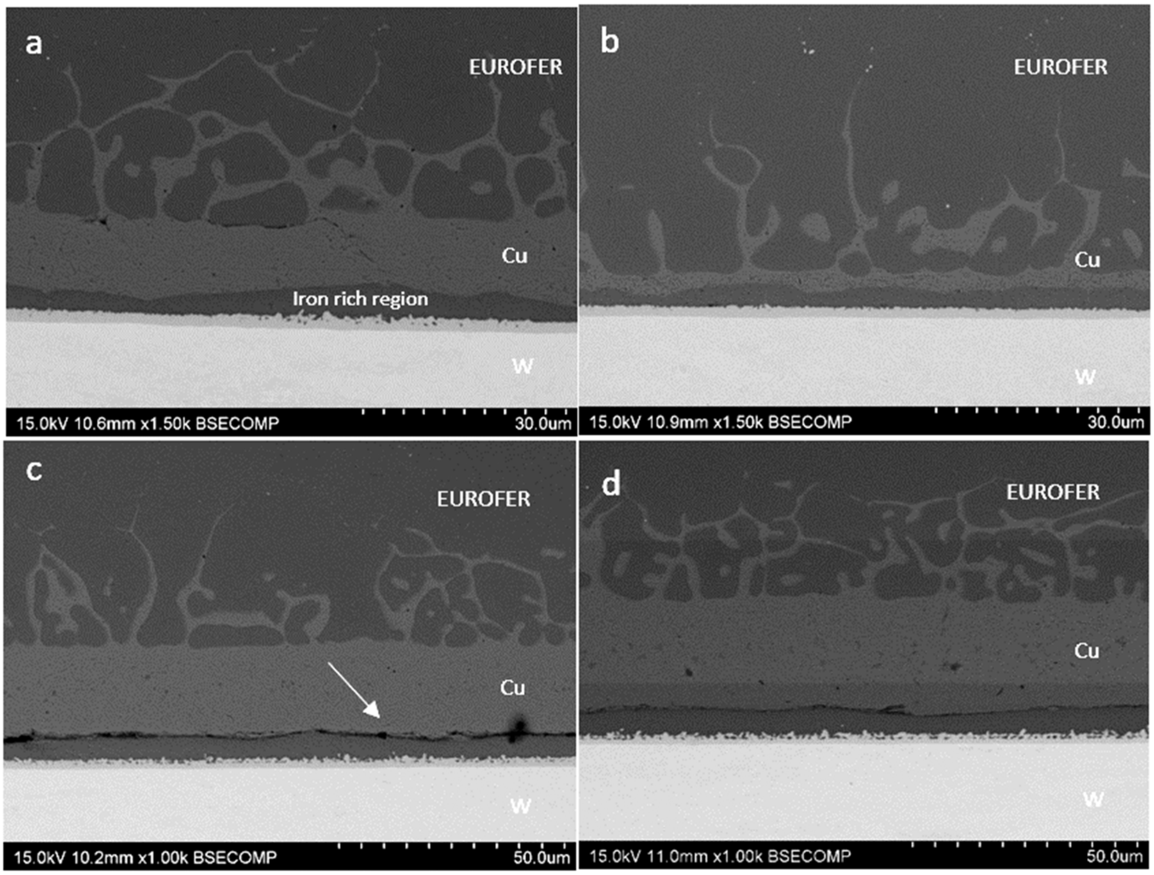

Fig. 7. Micrographs of samples subjected to a W surface temperature of 700 °C with the following conditions: (a) 100 cycles, (b) 100 cycles and aging treatment, (c) 1000 cycles and (d) 1000 cycles and aging treatment.

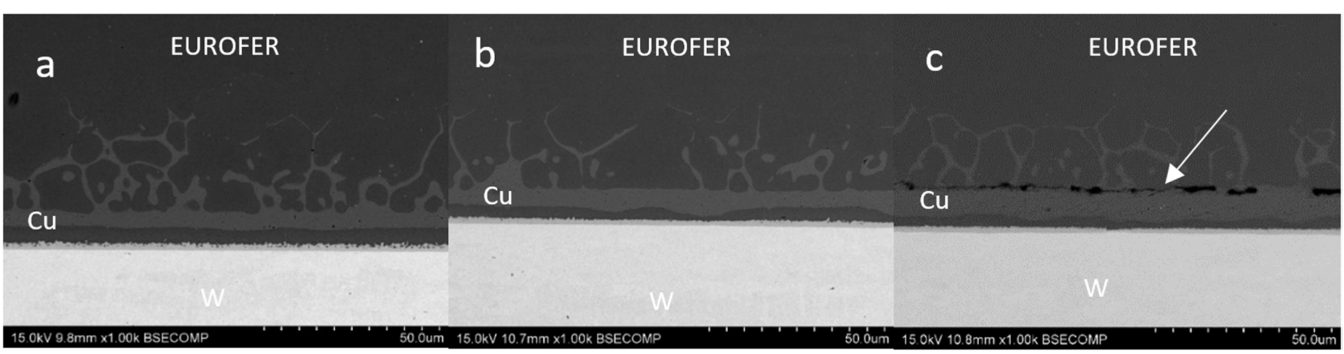

Fig. 8. Micrographs of samples exposed to a W surface temperature of 800 °C of for: (a) 100 cycles, (b) 100 cycles and aging treatment and (c) 182 cycles.

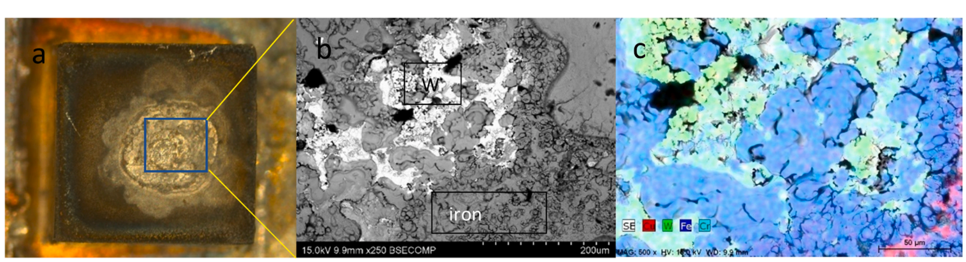

Fig. 9. Fractography images of EUROFER fracture surface of sample #16 obtained by: (a) stereoscopic microscope, (b) SEM and (c) elemental mapping distribution by EDS.

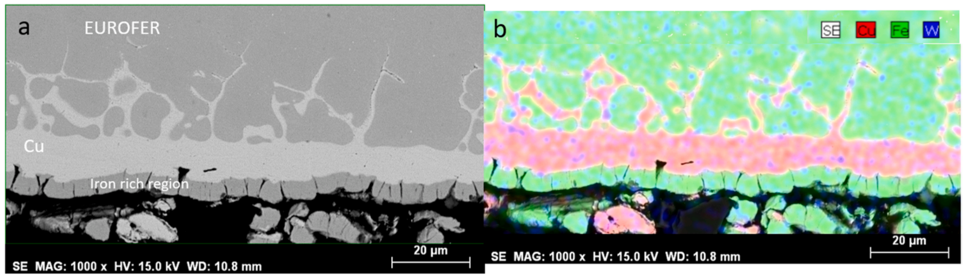

Fig. 10. (a) SEM and (b) EDS map distribution of EUROFER cross section piece of sample #16.

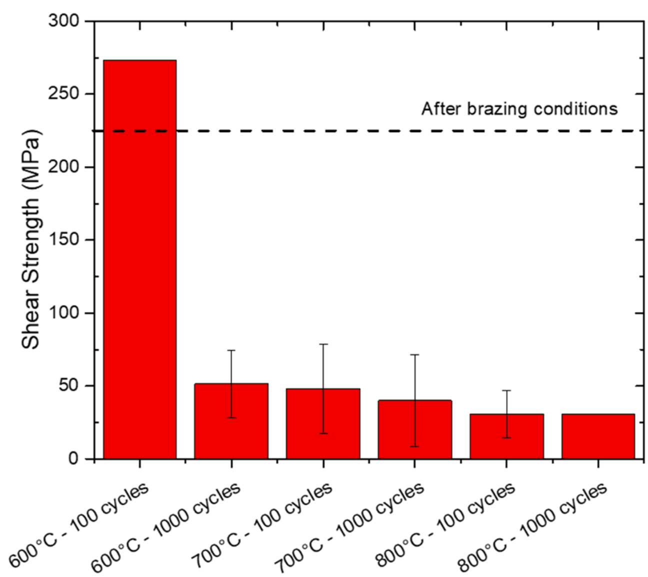

Fig. 11. Shear strength of W-EUROFER joints subjected to different HHF conditions of W surface temperature and number of cycles.

some W base material were detached and remained adhered to the EUROFER base material (arrow in Fig. 12c) demonstrating the higher adherence of this interface.

To evaluate the thermal effects on the base materials caused by the HHF tests, microhardness profiles were traced from one base material to the other through the braze. The results indicated that microhardness of both base materials was not affected neither by the aging process nor by the HHF tests (Fig. 14). In the case of tungsten, microhardness kept constant for all conditions and corresponded to the hardness of base material in as-received conditions (440 $HV_{0.1}$) (Kim et al., 2021).

In the case of the EUROFER, the obtained values were also the expected ones for a material that was not affected, since measured values were around 230 $HV_{0.1}$, typical of this martensitic ferritic steel after applying the brazing process and the subsequent tempering. The high heat flux test did not affect the Eurofer hardness as showed de Prado et al. (2018) in previous works. They tested an EUROFER-W brazing joint with CuTi filler alloy until 600 °C with no variation in the hardness in the EUROFER base material.

Regarding the braze zone, the main difference observed was associated to the formation of a larger copper band in the joints.

4. Conclusions and further work

Eurofer-W joints brazed with pure copper as filler material were exposed to different HHF conditions under steady state loads. Three

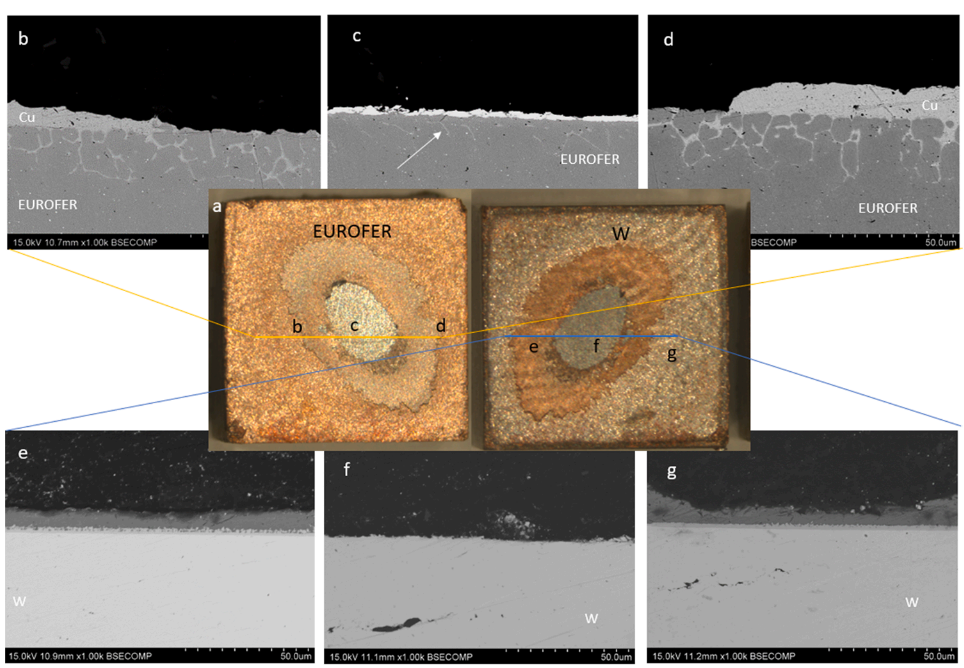

Fig. 12. (a) Stereoscopic image of fracture surfaces of sample #28 subjeted to 700 °C 1000 cycle after the shear test. Cross section images obtained by SEM of the fracture surfaces following the depicted line in (b), (c) and (d) EUROFER base material and (e), (f) and (g) tungsten base material.

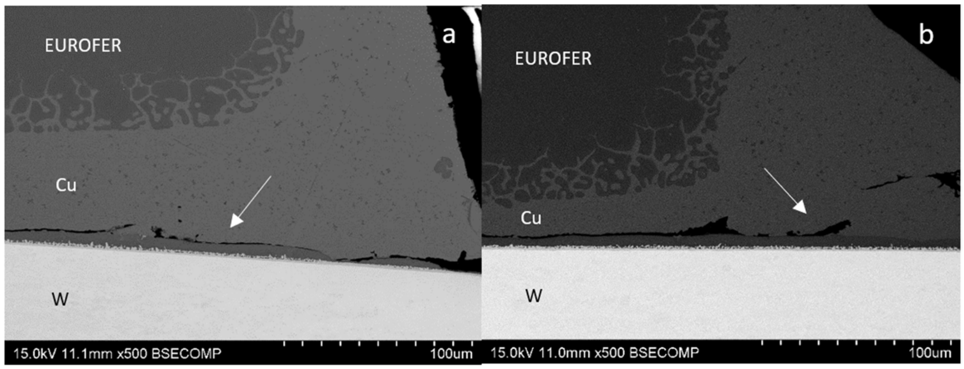

Fig. 13. Detail of nucleated cracks of different samples before shear tests subjected to 600 °C for 100 cycles (#2)– 1000 cycles (#8), (a) and (b), respectively.

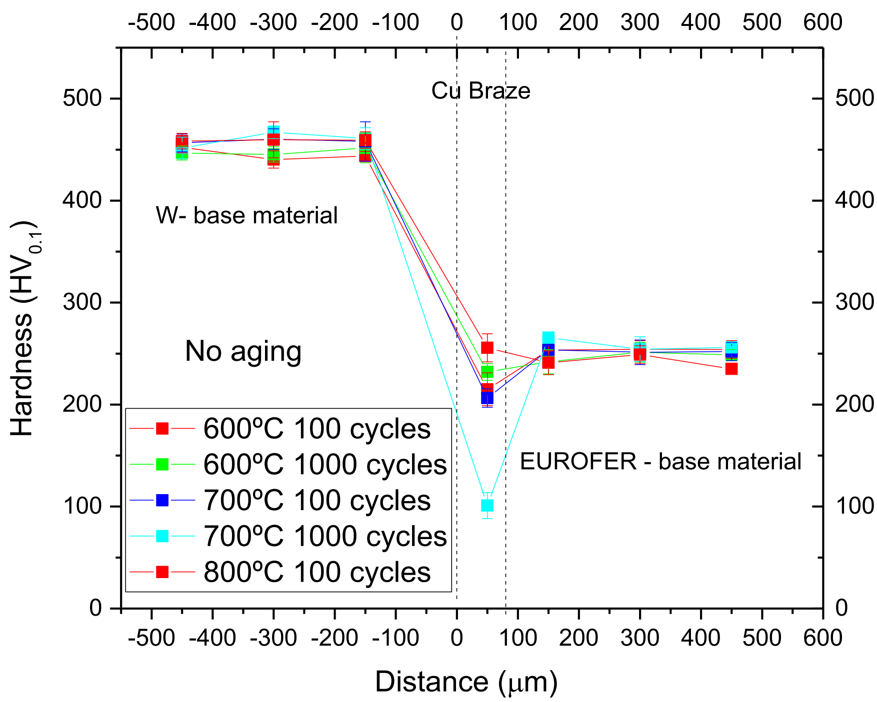

Fig. 14. Vickers microhardness profiles of W-EUROFER joints for the different HHF test conditions.

different W surface temperatures (600, 700 and 800 ºC) and two different numbers of applied cycles (100 and 1000) were studied as variables. Those surface temperatures corresponded, according to the simulation carried out, to 498, 578 and 656 ºC at the braze zone, respectively. Due to the high temperature that the EUROFER had to withstand under this last condition, which is not expected during operation since it involves the softening of the material, future efforts in the characterization of the joints should address the increase of the applied cycles at lower surface temperature.

The analysis of the thermal behavior by IR cameras and pyrometers during the HHF tests indicated that, although some cracks were reported in the metallography examination for 1000 cycles, no anomalies related to the W-EUROFER joint heating and cooling capacity were detected at 600/498 °C, 2 MW/m^2 and 700/578 °C, 2.5 MW/m^2. This reflects an improvement compared to previous work using Cu20Ti filler, which only maintained good refrigeration capacity without anomalies up to 500ºC surface temperature. However, in the case of 800/656 °C, 3 MW/m^2 the test had to be stopped at 182 cycles due to the overheating generated on the tungsten surface, reaching temperatures above 1200–1500 ºC.

The microstructural analysis indicated the formation of cracks in some cases due to the stresses generated by the difference in CTE between the materials used, and small cracks were formed along the joint at both interfaces of the copper band in most of the cases. The cracks hindered the heat flux transmission preventing cooling of the exposed area.

The cracks generated by HHF tests also reduced significantly the shear strength of the joints (to around 50 MPa) compared to the values obtained after brazing. In addition, the microhardness study revealed that the base materials were not thermally affected retaining their hardness close to the as-brazed conditions.

The combination of the fracture mechanism study and the microstructural study suggested that formation of the Fe-rich region played an important role in both the nucleation and propagation of the cracks in the joint. Therefore, future studies should be focused in controlling the brazing parameters to avoid the formation of such phase in the braze area.

In addition, although the remanent copper in braze area is reduced to a 5–10 μm layer its embrittlement under neutron irradiation and its influence in joint properties should be assessed in future works.

CRediT authorship contribution statement

I. Izaguirre: Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **T. Loewenhoff:** Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **J. de Prado:** Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **M. Sánchez:** Conceptualization, Formal analysis, Investigation, Resources, Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition. **M. Wirtz:** Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **V. Díaz-Mena:** Conceptualization, Formal analysis, Investigation, Resources, Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition. **A. Ureña:** Conceptualization, Formal analysis, Investigation, Resources, Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Ignacio Izaguirre reports financial support was provided by Rey Juan Carlos University.

Data Availability

The authors do not have permission to share data.

Acknowledgments

This work has been carried out within the framework of the EUROfusion Consortium, funded by the European Union via the Euratom Research and Training Programme (Grant Agreement No 101052200 — EUROfusion). Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the European Commission. Neither the European Union nor the European Commission can be held responsible for them.

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