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Development of a brazing procedure to join W-2Y_2O_3 and W-1TiC PIM materials to Eurofer

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Abstract
The present work proposes a brazing technique to address both the mechanical and metallurgical requirements of joining two different tungsten powder injection moulding materials (W-2Y_2O_3 and W-1TiC) with Eurofer steel. The joining procedure uses pure copper as a filler material and the brazing parameters are 1135 °C for 10 min in a high vacuum furnace. Metallurgical evaluation indicates the achievement, in both cases, of 100% metallic continuity at the interface. The brazing temperature is high enough to activate diffusion phenomena at the interface, giving rise to strong adhesion properties. The mechanical properties are evaluated by shear strength tests with values of 163 ± 55 and 162 ± 6 MPa for W-2Y_2O_3/Eurofer and W-1TiC/Eurofer, respectively. These values are considerably higher than those obtained in previous works using other interlayer materials. The microhardness of these W-base materials is similar to that in the as-received condition, indicating that it was not affected by the brazing process.

Keywords: brazing, W-PIM, joining, Eurofer, first wall, doped tungsten

(Some figures may appear in colour only in the online journal)

1. Introduction

The high power density of future fusion power plants and their extreme environments, in terms of the irradiation, physical and chemical erosion and sputtering that take place inside the vessel, make it necessary to develop new materials. Since tungsten is the main candidate to act as armour for the first wall and divertor, extensive effort has been made to enhance its mechanical and physical properties to deal with such extreme conditions. One newly developed material is an oxide- and carbide-doped tungsten produced via powder injection moulding (PIM), with a fine dispersion of particles, which enhances the strength of the material and displaces the ductile-to-brittle transition temperature to lower temperatures. However, in order to conform with the plasma facing components, tungsten armour has to be joined to a structural steel (Eurofer) to fulfil the mechanical and metallurgical requirements.

The achievement of high quality Eurofer–W joints has been studied by several authors [1, 2] using different joining techniques [3]; however, most of them use pure tungsten as the base material, which is the reference for this reactor. In contrast, the possibility of joining particle reinforced W-PIM materials with Eurofer remains almost completely unexplored. Preliminary attempts to join both materials using a Cu-20Ti filler material gave rise to high continuity joints with shear strength values of ~105 MPa [4]. The present work proposes a brazing procedure to join two different tungsten PIM materials (W-2Y_2O_3 and W-1TiC) with Eurofer for a DEMO first wall using a pure Cu filler, which has been demonstrated to enhance several aspects of the joint. The brazed joints are investigated by means of scanning electron microscopy (SEM) for the microstructural characterization. Microhardness and shear tests are used for the mechanical characterization.

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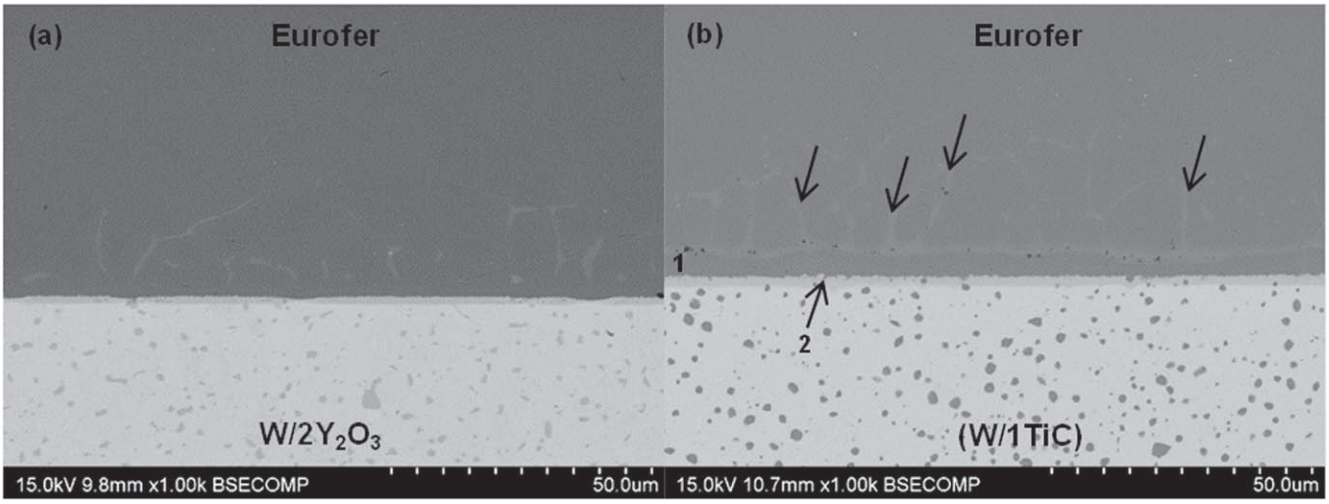

Figure 1. SEM micrographs of W-PIM/Eurofer brazed joints using Cu filler material and fabricated with (a) W-2Y_2O_3 and (b) W-1TiC base materials.

2. Experimental procedure

2.1. Materials

Standard Eurofer 97, along with two different doped tungsten samples, were used as the base materials. The former was supplied by the Karlsruhe Institute of Technology (KIT) and its composition and microstructure are described by Rieth *et al* in [5]. The latter, produced via PIM, were fabricated using two oxide- and carbide-doped tungsten metals at the KIT. PIM is a manufacturing process in powder metallurgy and a promising fabrication method from the large-scale production and near-net-shape precision points of view. The fabrication route consists of a mixture of inorganic tungsten powder with a polymer binder resulting in a so-called feedstock, which can be moulded. The production of the green part is a fast process that only takes ~20 s. After shaping, the green part is subjected to a heat treatment where the polymeric binder is eliminated and the metallic powders are sintered (2400 °C) to the near-theoretical density. This results in isotropic materials with an equiaxed grain orientation, good thermal shock resistance, shape complexity and a high final density (>98% theoretical density) [5].

Two W grades were developed: W-1TiC (99 wt% W + 1 wt% TiC) and W-2Y_2O_3 (98 wt% W + 2 wt% Y_2O_3). The intermediate material used as a filler was pure Cu supplied by Lucas Milhaupt in strip form with a thickness of 50 μm. The strip was cut with the exposed base material surface dimensions (6 × 6 mm^2) and placed between both specimens.

2.2. Brazing tests

A high vacuum furnace was used for the brazing tests at a residual pressure of 10^{-6} mbar. Prior to the brazing tests, samples of both base materials of 6 × 6 mm^2 in area were ground down with 4000 grit silicon carbide paper. Two different thicknesses of base material were used, 2 mm for the microstructural and microhardness tests, and 5 mm for the shear tests, which allowed us to adjust the sample to the grip of the shear fixture to minimise the misalignment of the load with respect to the joint interface. The brazing conditions were 1135 °C for 10 min. The heating and cooling rates were 5 °C min^{-1}.

2.3. Characterization techniques

A scanning electron microscope (S3400 Hitachi) equipped with energy dispersive x-ray analysis was used for the microstructural examinations. The error in the EDS results in most of the analysed phases was lower than 3%. The strengths of the joints were evaluated with a UTM machine Zwick Z100 using a special shear fixture designed for that purpose. The shear tests were carried out at a velocity of 1 mm min^{-1}. Finally, to evaluate the effect of the brazing process on the base materials, microhardness profiles across the joint were obtained with MHV-2SHIMADZU equipment. A load of 100 g was applied for 30 s from steel to W.

3. Results

3.1. Microstructural characterization

Figure 1 shows the micrographs of both the W-2Y_2O_3/Eurofer and W-1TiC/Eurofer joints (figures 1(a) and (b), respectively) after the application of the brazing process. Due to the high chemical and thermal stability of the TiC and Y_2O_3 reinforced particles, their influence on the microstructure of the joint is limited. Both joints are characterized by a high metallic continuity between both base materials, which means that the filler correctly melted at the brazing temperature, thereby filling the whole joint clearance. The high wetting properties of copper in iron, according to other studies carried out in the field [6], favour the spreading of the filler in the liquid phase, giving rise to the above-mentioned feature.

The microstructure of the solidified braze is the result of the interaction of the copper filler during the liquid stage of the

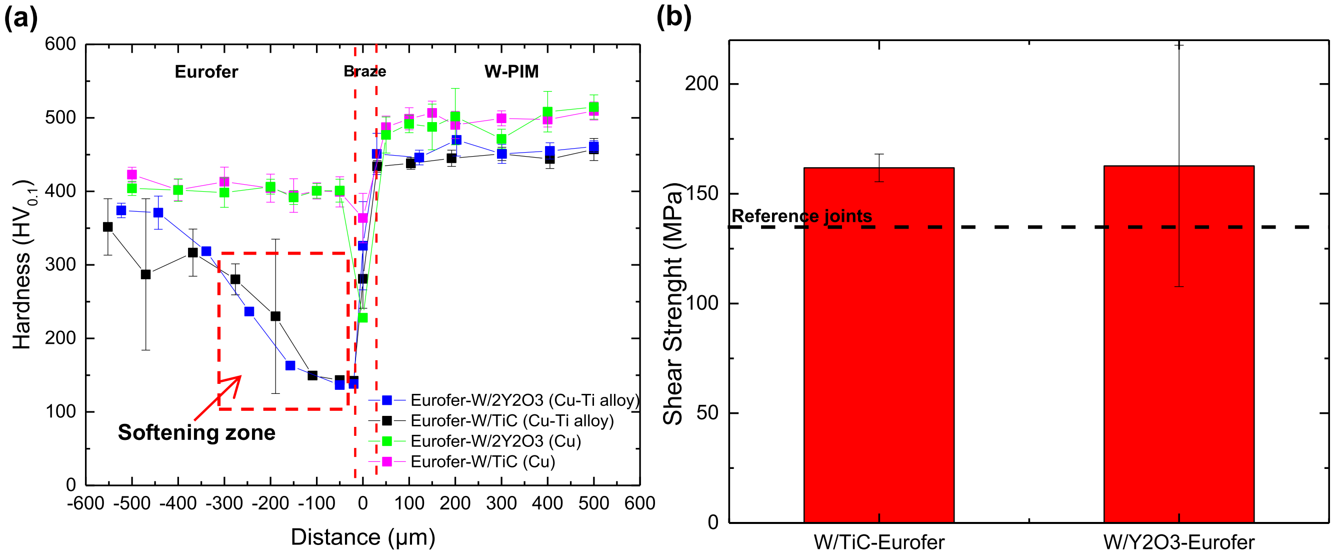

Figure 2. (a) Microhardness profiles across the W-PIM/Eurofer joints. (b) Shear strength of brazed joints. Data for the Cu-Ti alloy were obtained from [4].

filler at the brazing temperature and the reaction of copper with the other elements of the systems. As a consequence, the thickness of the copper filler is reduced compared to its initial thickness by the above-mentioned effect and the partial exudation of copper out of the joint clearance. The interaction, favoured at the Eurofer grain boundaries, promoted its penetration into the base material through the grain boundary giving rise to the columnar microstructures observed in both figures and arrowed in figure 1(b). The phase is constituted by pure copper according to the EDS microanalysis. In the case of the Eurofer/W-1TiC joint, a copper layer at the bottom of the columnar shape structures is also observed, which means that not all copper has penetrated into the Eurofer and remains as a layer. Right under the columnar structure, another layer composed of (at%) 86Fe-4Cu-8Cr-2W was also detected (phase marked as 1 in figure 1(b)). In this case, the EDS error result associated with the W content is 0.7 at%. The presence of W in this layer indicates that the temperatures reached during the brazing process were high enough to activate the diffusion mechanism of W. This phenomenon has not been previously observed and could explain the improved mechanical properties in comparison to the previous studied filler (Cu-Ti alloy), which used a lower brazing temperature of 960 °C [4].

Finally, a second layer constituted in both cases of (at%) 69W-27Fe-4Cr (EDS analysis) and formed at the W-PIM material's interface was also observed (phase marked as 2 in figure 1(b)). The layer is 2 μm thick and its formation is also associated with the interdiffusion mechanism of the system. Finally, it is noticed that the microstructure of both W-PIM materials has not been modified by the application of the brazing process according to the previous characterization of the base material in the as-receive properties.

3.2. Mechanical characterization

The microhardness profile shown in figure 2(a) indicates that the hardness properties of both tungsten base materials were not affected by the brazing process, which agrees with the microstructural examination described above. The hardness values measured under as-received conditions were 465 ± 16 $HV_{0.1}$ for W-2Y_2O_3 and 454 ± 14 $HV_{0.1}$ for W-TiC, which remained constant after the brazing process. However, in the case of the Eurofer base material, the brazing process does affect the hardness properties. The average measured after the process was ~400 $HV_{0.1}$, which means an increase of the hardness from the 220 $HV_{0.1}$ measured in the as-received conditions. This effect has been widely studied and is caused by the austenitisation of Eurofer during the brazing cycle (A_{1c} = 890 °C [7]); however, the application of a tempering treatment to the joint recovered the initial hardness of the steel [8]. One important fact in using pure Cu filler is that the formation of the softening region detected in the Eurofer (observed in figure 2(a)) can be avoided in the proximity of the interface, when the Cu-Ti alloy was used as a filler material, as detected in other studies [4]. The loss of hardness in that zone is associated with the loss of the alloying elements of the steel.

The adhesion properties of the joint, measured in shear mode, gave rise to strengths of 163 ± 55 and 162 ± 6 MPa for W-2Y_2O_3 and W-1TiC/Eurofer, respectively, representing an increment of ~53% with respect to the joints fabricated using the Cu-Ti alloy (106 and 104 MPa, respectively) [4]. The higher dispersion of the data in the W-2Y_2O_3 joints could be associated with difficulties related to the shear tests, such as alignment of the interface with the applied force and bending of the joint during the tests, meaning more statistics should be necessary to have more accurate data. The enhanced adhesion properties are associated with the interdiffusion phenomena observed at the W-braze interface, which gave rise to a better base material-braze interaction, resulting in the formation of stronger bonds at the interface.

The fracture surface is characterized by two different propagation zones, as observed in figure 3(a), namely, a bright zone, which corresponds to the propagation of the fracture throughout the interface, and a darker one, where the

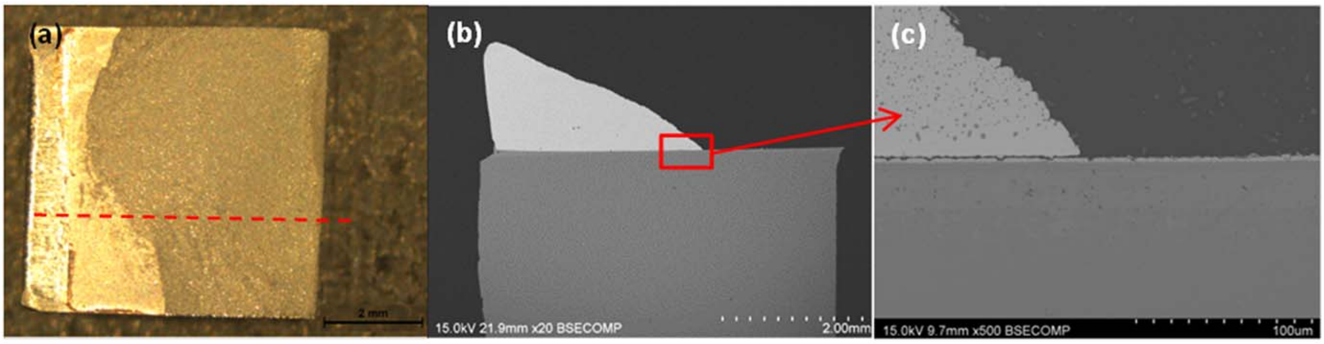

Figure 3. Fracture surfaces of W-TiC/Eurofer joints after the shear test: (a) stereoscopic image; (b) fracture cross section through the dash line in (a); (c) detail of the crack propagation in the two fracture zones.

crack changes its propagation mechanism and starts to penetrate into the W-PIM base material. The analysis of the fracture cross section in the dash line drawing in figure 3(a) shows clearly the two fractures zones. In addition, it can be seen that the propagation of the crack in the first zone occurs following the interface formed by the diffusion layer and the W-PIM base material.

4. Conclusions

High quality W-PIM/Eurofer joints have been achieved using a high vacuum brazing technique and pure Cu as a filler material. The brazing conditions (1135 °C for 10 min) were high enough to melt the filler and activate the diffusion phenomena of tungsten, giving rise to a continuous joint with high adhesion properties.

The microstructure comprises the melted cooper that penetrated into the Eurofer grain boundaries and two diffusion layers formed at the W-PIM/braze interface, as a consequence of the diffusion mechanism activation. The microhardness profile confirms that the W-PIM base material does not suffer any modification in the hardness properties as a consequence of the brazing process. Furthermore, the profile also shows that Eurofer does not develop the softening zone close to the interface observed in previous studies. Finally, the obtained shear strength of the brazed joints produces an increment of almost 53% with respect to the previous studies, which is associated with better base material–braze interactions.

Acknowledgments

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