

WPMAT-CPR(17) 17125

M Sanchez et al.

# Development of W-PIM/EUROFER brazed joints

Preprint of Paper to be submitted for publication in Proceeding of 16th International Conference on Plasma-Facing Materials and Components for Fusion Applications



This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission. This document is intended for publication in the open literature. It is made available on the clear understanding that it may not be further circulated and extracts or references may not be published prior to publication of the original when applicable, or without the consent of the Publications Officer, EUROfusion Programme Management Unit, Culham Science Centre, Abingdon, Oxon, OX14 3DB, UK or e-mail Publications.Officer@euro-fusion.org

Enquiries about Copyright and reproduction should be addressed to the Publications Officer, EUROfusion Programme Management Unit, Culham Science Centre, Abingdon, Oxon, OX14 3DB, UK or e-mail Publications.Officer@euro-fusion.org

The contents of this preprint and all other EUROfusion Preprints, Reports and Conference Papers are available to view online free at http://www.euro-fusionscipub.org. This site has full search facilities and e-mail alert options. In the JET specific papers the diagrams contained within the PDFs on this site are hyperlinked

# DEVELOPMENT OF W-PIM/EUROFER BRAZED JOINTS FOR DEMO FIRST WALL

J. de Prado<sup>a</sup>, M. Sánchez<sup>a\*</sup>, S. Antusch<sup>b</sup>, A. Ureña<sup>a</sup>

<sup>a</sup>Materials Science and Engineering Area. ESCET. Rey Juan Carlos University, C/Tulipán s/n, 28933 Móstoles, Madrid, Spain <sup>b</sup>Karlsruhe Institute of Technology, Institute for Applied Materials, P.O. Box 3640, 76021 Karlsruhe, Germany.

Email: maria.sanchez@urjc.es

**Abstract.** The present work describes a brazing procedure to join two different tungsten PIM materials (W-2Y<sub>2</sub>O<sub>3</sub> and W-1TiC) with Eurofer. The results indicated the achievement, in both cases, of high quality W-PIM/Eurofer joints using 80Cu-20Ti filler material. The braze is constituted by several ternary Cu-Ti-Fe phases distributed along a Cu-matrix, which acts as ductile phase capable to absorb the residual stresses, and may be produced during the service life of the component. Some intergranular cracks have been detected growing from W-braze interface into the base material, caused by the mismatch in the coefficient of thermal expansion (CTE). Regarding to the strength of the joints, similar shear strengths of both joints were obtained (~105 MPa). The values were slightly lower than the one obtained when pure tungsten was used as base metal.

# 1. Introduction

The necessity of finding new materials capable to ensure the correct development during service life of the DEMO fusion reactor makes necessary a special effort in the areas where the selected materials have weaker properties. Tungsten is the most significant one because it has extraordinary physical characteristics as a plasma facing materials (high thermal conductivity, sputtering resistance and melting point). However, it has also two main disadvantages: 1) its high brittleness and 2) its high corrosion rate at high temperatures in an oxygen atmosphere [1]. In order to solve the first inconvenient, oxide and carbide doped tungsten PIM materials, with a fine dispersion of particles, have been developed to enhance the strength of the material and to displace the DBTT to lower temperatures [2]. These new materials have to be joined to other materials (i.e. Eurofer) in order to conform the plasma facing components. Therefore, joining technologies need to be implemented to meet the requirements of the reactor environment.

The present work proposes a brazing procedure to join two different tungsten PIM materials (W- $2Y_2O_3$  and W-1TiC) with Eurofer for DEMO first wall. The brazed joints have been characterized by means of SEM for the microstructural characterization and microhardness and shear tests for the mechanical characterization.

# 2. Experimental procedure

#### 2.1 Materials

The base materials used for the joints were Eurofer and PIM doped tungsten. Eurofer has been supplied by Karlsruhe Institute of Technology (KIT) with the standard composition and microstructure. The two oxide and carbide doped tungsten materials were produced at KIT via W Powder Injection Molding (W-PIM). This is a promising fabrication method

in view of large-scale production and near-net-shape precision. The combination of tungsten powder with a small quantity of a polymer, a so-called feedstock, can be moulded. Depending on the size and shape of the parts, for simple geometries, only 20 seconds are needed to produce a green part. After shaping the green part (consisting of powder and binder), the polymeric binder must be extracted and the powder sintered at 2400 °C to the near-theoretical density. Isotropic materials, equiaxed grain orientation, good thermal shock resistance, shape complexity and high final density (>98% theoretical density) typical properties of powder injection molded tungsten. One of the possibilities for manufacturing parts via W-PIM has been shown by the latest produced series of Langmuir samples for diagnostics for the French tokamak WEST (Tungsten (W) Environment in Steady-state Tokamak) [2-4].

The filler used for brazing cycles was a mixture of pure powders with 80Cu-20Ti composition and an organic binder in 95/5 weight ratio. Copper (*Stream Chemical*, 99.9% purity, 100 mesh) and titanium (*Alfa Aesar*, 99.95% purity, 200 mesh) metallic powders were manually stirred together with the organic binder (polypropylene carbonate, *Empower Materials, QPAC 40*). Finally, the mixture was laminated obtaining flexible tapes of 250 µm thick.

#### Brazing tests

Brazing tests were made in a high vacuum furnace at the residual pressure of  $10^{-6}$  mbar. Samples of both base materials with an area of 8 x 8 mm<sup>2</sup> were ground down to 4000 grain size with silicon carbide paper. The thickness of the samples was 2 mm for the microstructural and microhardness tests, and 5 mm for the shear tests in order to ensure a tight grip from the shear fixture. Brazing conditions were 960 °C (50 °C above the liquidus temperature of the filler alloy) for 10 min. Heating and cooling rates were 5 °C/min.

#### 2.2 Characterization techniques

Microstructural and fractographic examinations were made by SEM (*S3400 Hitachi*) equipped with energy dispersive X-ray analysis (EDX). Mechanical properties of the joints were evaluated by Vickers microhardness and shear tests. The effect caused by the brazing process in the base materials was analyzed by the microhardness variations measured across the joint with *MHV-2SHIMADZU* equipment. A load of 100 g was applied for 30 s from steel to W. The shear tests were carried out at a velocity of  $1 \text{ mm/min}^{-1}$  in a UTM machine *Zwick Z100*.

# 3. Results

#### 3.1 Microstructural characterization

Both W-2Y<sub>2</sub>O<sub>3</sub>/Eurofer and W-1TiC/Eurofer brazed joints showed high continuity interfaces reaching 100 % of metallic contact (Figures 1 (a) and (b), respectively). The microstructure of the solidified braze is similar to that obtained when pure tungsten was used as base material [5]. This fact is associated to the same level of interaction between the base materials and the filler alloy. The microstructure at the Eurofer-braze interface is characterized by the formation of several Cu-Ti-Fe layers as a consequence of the reaction between the Eurofer and the filler during the brazing process. The microstructure at the center of the braze is constituted by a Cu matrix and two different Cu-Ti-Fe phases. The phase richer in Ti showed darker at the backscattering image.

Regarding the W-PIM/braze interface, intergranular cracks, which nucleated at the interface and penetrated into the PIM base materials following the grains boundaries were detected (Figures 1 (c) and (d)). They were caused by the thermal stresses produced during the cooling stage of the brazing cycle associated to the mismatch in the CTEs of both base materials. Their propagation mechanism is associated to the weak grain boundaries, characteristic of tungsten base materials, where the crack tends to propagate between reinforcing particles due to they act as stress concentration points.



**Figure 1.** General view of W-PIM/Eurofer brazed joints fabricated with (a)  $W-2Y_2O_3$  and (b) W-1TiC. Detail of the (c)  $W-2Y_2O_3$ /braze and (d) W-1TiC/braze interfaces.

# 3.2 Mechanical characterization

Figure 2 (a) shows the microhardness profile across the joints fabricated with W-PIM base materials. The results indicated that mechanical properties of both tungsten base materials have not been affected by the brazing process, since the hardness values measured under as received conditions have not been modified ( $455 \pm 16 \text{ HV}_{0.1}$  for W-2Y<sub>2</sub>O<sub>3</sub> and  $444 \pm 14 \text{ HV}_{0.1}$  for W-TiC). However, a hardening process (~ 400 HV<sub>0.1</sub>) is detected in the Eurofer base materials at distances long enough from the braze to be influenced by it. The temperatures reached during the brazing cycle situated the steel in the austenization field ( $A_{1c}$ = 890 °C [6]) and modified the as receive microstructure of Eurofer [5]. Besides, as the indentations approached to the braze a softening process in the Eurofer is detected caused by the loss of the alloying elements of the steel through

solid state diffusion mechanisms. Both, hardening and softening processes have been studied in more detail in previous works [5, 7].

In the case of the joints fabricated using W-2Y<sub>2</sub>O<sub>3</sub> and W-1TiC, similar shear strength values were obtained (106 and 104 MPa, respectively) due to their similar microstructure. The values achieved were slightly lower than those obtained when pure tungsten was used as base metal (133 MPa). The lower values could be associated to the intergranular cracks propagation in the base materials observed at the proximity of the interface. This fact favored that during the shear tests, once the crack is nucleated at the interface, it is easily propagated into the base materials following intergranular paths. Therefore, it can be concluded that the use of these advanced tungsten materials does not provoke a significant detriment in the mechanical behavior of the joint.



Figure 2. (a) Microhardness profiles across the joints. (b) Shear strength of brazed joints.

#### Conclusions

Operational brazeability has been achieved for brazed joints between tungsten PIM materials (W-2Y<sub>2</sub>O<sub>3</sub> and W-1TiC) and Eurofer using 80Cu-20Ti 95/5 PPC filler in a high vacuum furnace at 960 °C for 10 min. The microstructure of the braze is constituted by ternary Cu-Ti-Fe phases distributed along a Cu-matrix. During the brazing cycle, Eurofer reacted with the braze alloy and formed several reactions layers at the Eurofer-braze interface. Intergranular cracks growing from W-braze interface and penetrating into tungsten PIM base materials have been detected; they are caused by the mismatch in the CTE during the cooling stage of the brazing cycle. The mechanical properties of the tungsten PIM base materials have not been modified by the brazing cycle according to the microhardness results. However, hardening and softening processes took place in the Eurofer base material. The shear strength of both joints was similar (~105 MPa), which is slightly lower than the strength reported by joints fabricated with pure tungsten as base material. This fact could be associated to the presence of the intergranular cracks observed at the W-braze interface.

# Acknowledgments

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not

necessarily reflect those of the European Commission. The authors would also like to acknowledge to MULTIMAT-CHALLENGE program (S2013/MIT-2862).

#### References

[1] Rieth M *et al.* 2013 Recent progress in research on tungsten materials for nuclear fusion applications in Europe *J. Nucl. Mater.* 432(1–3) 482-500

[2] Antusch S *et al.* 2015 Mechanical and microstructural investigations of tungsten and doped tungsten materials produced via powder injection molding *Nuclear Materials and Energy* 3–4 22-31

[3] Antusch S *et al.* 2017 Refractory materials for energy applications *Energy Technology* DOI: 10.1002/ente.201600571

[4] Brittle me this, Fusion in Europe, www.euro-fusion.org/newsletter (2016)

[5] de Prado J, Sanchez M and Ureña A 2016 Development of brazing process for W– EUROFER joints using Cu-based fillers *Phys. Scr.* T167 014022

[6] Rieth M, Falkenstein A, Graf P, Heger S, Kempe H, Lindau R and Zimmermann H 2003 EUROFER 97 Tensile, charpy, creep and structural tests, M1 - FZKA-6911

[7] de Prado J, Sanchez M and Ureña A 2016 Wettability study of a Cu-Ti alloy on tungsten and EUROFER substrates for brazing components of DEMO fusion reactor *Mater. Des.* 99 93-101