



Qualification & testing of joining development for DEMO limiter component

Elena V. Rosa Adame^{a,*}, Andrius Aleksa^a, Mark R. Gilbert^a, Martin Cuddy^a, Tristan Calvet^b, Zsolt Vizvary^a, Nicolas Mantel^a, Francesco Maviglia^c, Jeong Ha You^c

^a Central Engineering Department, UKAEA, Abingdon, Oxfordshire OX14 3DB, United Kingdom

^b Imperial College London, London SW7 2AZ, United Kingdom

^c Max-Planck-Institut für Plasmaphysik, Boltzmannstr. 2, Garching 85748, Germany

ARTICLE INFO

Keywords:

Process and Infrastructure
Joining technology
Brazing joint
Plasma facing components
Filler metal
Base material
Wettability
Testing

ABSTRACT

A development plan for validation of functional principles is defined to support the challenges of mock-up manufacturing and testing. It is aimed to develop the process and infrastructure for qualifying fusion components for the limiters in the European DEMO. The limiters are components that define the plasma boundary by direct contact during normal and off normal transient events and thus they protect the First Wall of the Breeding Blanket System from extreme heat fluxes during these events. Within this framework, the joints play an important role for making feasible the combination of dissimilar materials required for the plasma facing components (PFCs) by providing a compliant interlayer. The main limiter PFC functionality is to act as thermal barrier, therefore the materials combination shall be able to support the thermal gradient between the high heat flux (HHF) from the plasma and the heat sink. The primary aim of the PFC is to ensure the structural integrity of the heat sink of the PFC (to prevent in-vessel loss of coolant accident), as it is foreseen that during the off-normal transients the PFC surface may melt or evaporate. Minimizing the large deformation and guaranteeing the strength and fatigue behaviour of the joints is required to achieve this.

Therefore, a testing programme for joining development and qualification based on brazing technology is performed. It is focused on joint assessment between representative filler metals (OB1025TM, OB950TM, PB950TM, NBLMTM & H-BronzeTM) from the different families in the market and the chemical compatibility, capillarity flow and spreadability on the typical base materials used for PFC (laser powder bed fusion additive manufactured W-6%Ta, W, P91, OFHC Copper, CuCrZr). The main results show good wetting of the gold-copper alloy (OB1025TM) with all the base materials. It allows to progress with the integration of a PFC to create the process and infrastructure for optimizing the design of critical joints. NBLMTM seems to be an interesting filler for materials with high melting temperature as tungsten and P91 and OB950TM presents acceptable wetting condition with the base materials. PB950TM is rejected because the excess of wetting on all the base materials.

1. Introduction

The main goal for the development plan is to release the manufacturing and testing programs for limiter mock-up by the identification of short, medium and long-term R&D work packages (WPs). The short-term is focused on developing the process and infrastructure with the suitable and achievable technologies to capture the limitations and the functional requirements coming from the manufacturing and testing during the life cycle for the limiter mock-up.

The development plan defines a methodology for validation of

functional principles and First Wall Limiter Mock-up. This methodology is applied to the testing programme for joining development and qualification based on brazing technology. It consists on the functional analysis and the analysis of failure modes by the root cause-effect during the validation. And it has been applied to the joint design, procedures, process as technologies during the life cycle.

This paper presents the initial results coming from developing the process and infrastructure required for assessing chemical compatibility between dissimilar materials used for PFC (AM W-Ta, W, P91, OFHC Copper, CuCrZr) and representative filler metals from the market:

* Corresponding author.

E-mail address: elena.adame@ukaea.uk (E.V.R. Adame).

<https://doi.org/10.1016/j.fusengdes.2022.113164>

Received 15 November 2021; Received in revised form 5 April 2022; Accepted 3 May 2022

Available online 21 May 2022

0920-3796/© 2022 Published by Elsevier B.V.

OB950TM, OB1025TM, PB950TM, NBLMTM and H-BronzeTM.

2. Joining technology assessment

Brazing, HIPing and Additive Manufacturing (AM) are assessed in [1] as feasible technologies for joining dissimilar materials. Brazing technology is selected at short-term for creating the process and infrastructure required for releasing the manufacturing and testing of an integrated PFC.

Brazing is a well-known technique; it allows the assembly of large components in furnace as well as the assembly of dissimilar materials. It minimizes the residual stresses by the homogeneous temperature in the chamber during the brazing cycle and it provides repeatability of the process. In contrast, it is necessary to develop the manufacturing-test procedures specific for fusion application and trials (section 4 and 5). The effort is focused on developing the process and infrastructure for the testing rather than developing the own technology.

HIPing is a technique that applies temperature and isostatic pressure during the process in inert atmosphere; the assembly of the components are limited by the chamber dimensions, 1 m approx., smaller than brazing, 2-3 m. Besides, there is not a clear advantage between HIPing and brazing in terms of residual stresses; they depend on many coupled parameters during the assembly processes.

The main difference of HIPing regarding to brazing lies in the use or not of filler to create the bounding interface as well as powder and solid metals can be joined in the same process with HIPing.

HIPing can use or not a filler to create bounding interface whilst brazing requires diffusion of the filler into the parent material for it. The main concern about using filler in fusion applications consist on losing the mechanical properties. The transmutation of the alloy chemistry composition (Table 5 and Table 6) produces the creation of inclusions by heavy metals and porosity by outgassing.

Rather than being a disadvantage, a detailed study on [4] shows the development on brazing joints nowadays are focused on the design of the joint itself (gap, applied force, length, surface finish and brazing cycle) to optimize the strength and fatigue behaviour of the joint in a specific application. Additionally, the market can develop a filler for fusion application. So, it is matter of defining the functional requirements in terms of chemical content limitations on the filler for fusion. For that, it is necessary to progress on the integration of a component to define the process and infrastructure during the life cycle that allows to capture those functional requirements by the testing. Furthermore, inclusions can be added to the joint; or cracks and porous could be induced in the joint to emulate different stages of the filler during operation.

As previously mentioned, HIPing can also use powder to create complex geometries minimizing the porosity and to combine powder with solid material in the process minimizing the impact of misalignment due to thermal mismatch or clearance between different materials. However, the strength and fatigue behaviour of the powder materials would be compromised, and the number of joints is not further reduced. So, it is not recommended to use powder material for PFC with this process. Additionally, HIPing could cause cracking of solid tungsten during pressurization process. Solid tungsten is typically used as thermal shield material for PFC due to its thermal resilience; though, it is brittle.

In summary, HIPing is identified as a medium-term technology requiring further development in the process and procedures for fusion components. HIPing could also be used as a post-process method to relax the residual stresses.

Finally, AM is an emerging technology which brings the advantages of using powder as manufacturing of the component without limitation of any chamber. It is able of generate complex geometries that can be brazed to other materials. It is a very flexible process that allows an accurate control by the footprint area, power of the laser, speed, and trajectory of the process. Further, AM process minimize the number of joints in the component by the generation of complex geometries and it

is a repeatable process.

AM admits post-processes as machining to control geometry or surface finish, brazing to other parts and post-treatments with HIPing.

The technique has proved the manufacturing of large components with some materials as aluminium, titanium, etc (more than 1 m), and less than 0.5 m for tungsten [5,6].

The process is not simultaneous, the direction of the laser and subsequent passes of the laser beam re-heats the already solidified material. The evolution of the solidifying liquid as well as the microstructure is dependent on the laser direction and the re-heating due to the passes. So, residual stresses are expected, and they are expected to be directional. However, in contrast to HIPing, AM creates discretional melting and solidification; the powder feedstock is shaped and consolidated layer by layer to arbitrary configurations, normally using a computer-controlled laser.

The advantage of AM for PFC at medium term lies on optimized functional geometries that optimizes the topology of the component, not just minimizing powder material used, but also making driven mechanical properties according to the geometry and functionality of the component. For example, metal matrix composite presents high strength, high stiffness, toughness, damping capacity, etc. AM tungsten-6% tantalum (AM W-6%Ta) is used for FW of DEMO divertor to optimize the contact area with the plasma; tantalum contributes to the corrosion resistance and provides ductility that tungsten lacks [7].

AM components use to present similar strength to solid base material and good fatigue behaviour [10] and [11], though it needs to be proven for the PFC materials.

At longer-term, functional grading materials made with AM is recommended for PFC in which a blended interface exists. The transition to one material to other can be gradual. Powder tungsten can pass gradually to other powder material with ductile properties as copper [12]. The transition from tungsten to the heat sink material can gradually pass from one to other, or even more transitions as required. Other potential transitions could be from tungsten to CuCrZr and P91 to act as thermal boundary or structural integrity. Some of the material combinations already achieved can be checked in [13].

So, as part of the objectives to release the manufacturing and testing, brazing is selected as the achievable technique at short-term to define and optimize the process for a specific component and application over the life cycle. It is a mature technology which will minimize uncertainties in the process definition and infrastructure; the challenge of brazing technique is focused on performing procedures specific for fusion components. Brazing is a flexible technology; HIPing can be used as a post-process of brazing at lower pressure; and complex geometries made with AM can be also brazed. Finally, the brazing method offers the possibility of transferring requirements from the plasma to the filler alloy like, for example, the element transmutation in the filler by inclusions or cracks simulating operational stages. Therefore, the acceptance criteria for the PFC joints at short, medium and long term seem to be a strategic WP for assessing the component.

Fig. 1 summarizes the discussion on technology development for PFC joints. This paper pursues the definition of the process and procedures, the creation of infrastructure and the capture of functional requirements for wetting and capillarity tests on dissimilar material combinations in order to assess the chemical compatibility with different fillers. The next phase would be to progress with the manufacturing and testing of an integrated component. With the first iteration ($t=0$), the process and infrastructure would be defined and it could be optimized for achieving a component verified and validated. It will be called "pattern", the achievable solution at short-term. The pattern will be used as point of reference for comparing with any other development that pursues the long-term objectives.

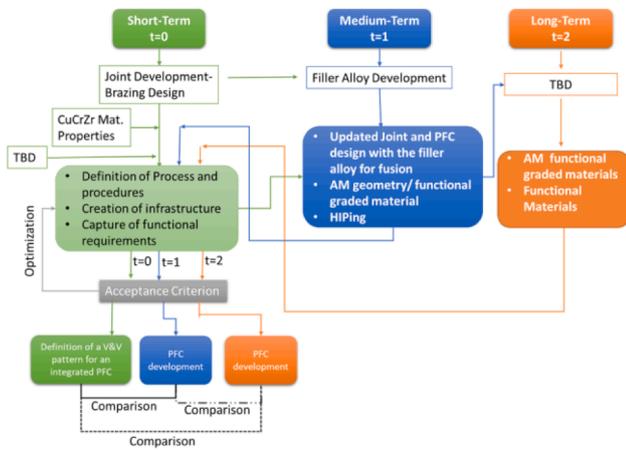


Fig. 1. Flow chart for Joining Development R&D WP's.

3. Material selection

3.1. Base materials for PFC

The materials potentially used for PFC are tungsten, AM W-6%Ta, OFHC Copper, EUROFER97/P91 and CuCrZr. They are combined to achieve a functional grading material effect in the assembly.

The specific combination of materials for PFC is still under development and it depends on the main function of the component, thereby, several designs for PFC are conceived with the combination of these materials. It is not the objective of this paper to assess the PFC designs. The objective is to assess all the possible joint combinations with the base materials for the PFC designs.

Limiters require to resist the erosion due to the direct contact with the plasma, to act as thermal shield for avoiding boiling in the coolant during the severe transients and, at the same time, to transfer the thermal load to cool down the plasma and to protect the FW during regular operation keeping the integrity of the heat sink. These requirements are directly transfer to the joints in the assembly. The joints are critical within PFC; they must keep the functionality of the base materials to allow a smooth transition from one base material to another and to contribute to the functional grading material assembly.

This paper studies the possible joints for the base material combination of Table 1, and following the configuration from

Fig. 6 with the selected filler metals (section 3.2). In total, 39 material combination and tests to assess material compatibility (Table 9).

Tungsten is typically selected as armour of PFCs. This material can be eroded by the plasma particles, mostly during short pulses of high heat loads, associated with ELM or plasma disruptions. It is also able to withstand high heat flux.

Besides tungsten is a material with high thermal inertia, and it presents high melting temperature (Table 2). In contrast to the rest of the base materials used for PFC, tungsten has a low thermal expansion coefficient. The difference of thermal expansion between tungsten and the rest of base materials creates residual stresses during operation and manufacturing process where the temperatures are high.

Table 1
Base Material Combination.

Base Mat.1	Base Mat.2	Filler Metals
AM W-6%Ta	OFHC Copper, P91	OB1025, NBLM
Tungsten	OFHC Copper, P91, CuCrZr	OB950, OB1025, PB950, NBLM, H-Bronze
OFHC Copper	P91, CuCrZr	OB950, OB1025, PB950, NBLM, H-Bronze
P91	P91, CuCrZr	OB950, OB1025, PB950, NBLM, H-Bronze

Table 2
Thermal Properties at 20 °C ([16] and [17]).

Material	Melting Point, °C	λ W/mk	CTE $10^{-6}/^{\circ}\text{C}$
AM W-6%Ta	≈ 3410	-	-
Tungsten	3410	173	4.5
OFHC Copper	1085	401	16.7
P91	1420	23.1	10.3
CuCrZr	1081	318	16.7

For that, some PFC designs use OFHC Copper as interlayer between tungsten and the heat sink material to take the advantage of elastic-plastic properties of copper absorbing the difference of dilatation between different materials. Additionally, the good thermal conductivity of copper helps to transfer the thermal load in the assembly, as for example Fig. 3.

Alternatively, AM W-6%Ta is a material under development and specifically defined for armour in PFC. Its characterization is still in progress [8]. Though, melting point is expected to be close to pure tungsten [9]. AM brings the advantage of passing from solid material to metal matrix progressively. Several lattice structures are assessed to optimize the thermo-physical characteristic in [14] for AM W. The solid material is used as armour and the lattice is optimized to transfer the thermal load taking the advantage of high stiffness of density ratios and damping capacity of the matrix.

Fig. 2 is an example of this concept which additionally is joined to a CuCrZr part.

Currently, AM W-6%Ta solid material and metal matrix are under development and a concept as

Fig. 2 is not still performed. This paper assesses the joints of solid AM W-6%Ta in order to progress on the integration of a PFC concept with this material as performed with AM W.

Such as tungsten as AM W-6%Ta have low thermal expansion coefficient (Table 2). Though, AM W-6%Ta adds ductility to the material, it is considered as pure tungsten fragile materials (Table 3) and the main function is to act as armour and transfer the thermal load. So, materials to keep structural integrity are needed in the assembly. CuCrZr and EUROFER are selected as possible structural materials for most of PFC designs.

CuCrZr is a precipitation hardened alloy. Copper is the dissolute and Chromium and Zirconium are the solutes. Chromium brings the benefit of higher strength and Zirconium improves the fatigue properties, improving the ductility at elevated temperatures with a thermal expansion coefficient similar to copper (Table 2). Furthermore, CuCrZr has similar thermal conductivity to copper. So, it is a good material for transferring the thermal load to the working fluid during normal operation.

EUROFER97 is a ferritic martensitic heat resisting steel with good irradiation resistance. It is a specific material developed for Fusion Reactor Power Plants. And it is a modified version of 8-12%CrMoVNB steels. However, it is not fully standardized and qualified, and its availability is limited. For that, it is substitute by x10CrMoVNB9-1,

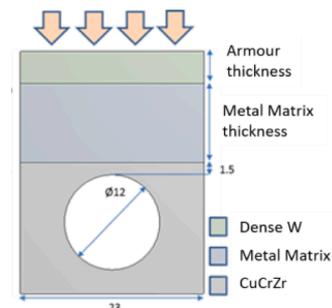


Fig. 2. Design proposed within WPDIV in [14] using AM W lattice structure.

Table 3
Mechanical Properties at 20 °C ([16] and [17]).

Material	Sy _{0.2%} MPa	Su MPa
AM W-6%Ta	–	–
Tungsten	1360	1432
OFHC Copper	55	200
P91	400	450
CuCrZr	407	452

commonly called P91 according to ASME SA 387 Gr 91. EUROFER97/P91 is a material with good mechanical properties at high temperature (Table 3). However, the thermal expansion coefficient and thermal conductivity are low (Table 2). For that, some PFC designs uses the EUROFER97/P91 as thermal barrier between tungsten and the fluid (Fig. 3). An interlayer of OFHC Copper with good ductile properties is used to absorb the mismatch between tungsten and P91 thermal expansion.

In summary, these are the main base materials used for PFC designs. The objective is to assess the multiple joints with the dissimilar materials typically used for PFC in order to assess the suitability of configurations and the fillers. The combination of P91 with P91 is also assessed due to its relevance as a structural material for fusion components.

Table 2 and Table 3 summarize the mechanical and thermal properties for the base materials used in these trials according to the standards.

3.2. Filler metal selection

A detailed assessment of fillers in the market and their different applications is performed in [4]. This paper also reviews the better combination of elements for offering a good behavior of the joint under the different operational temperatures.

Pre-selection of different family filler metals available in the market for limiter application is carried out in [2]. It corresponds to the fillers compatible with the brazing temperature of the base materials (Table 2). The brazing temperature should be around 950 °C to avoid any change in the microstructure of the softer base materials during the brazing (OFHC copper and CuCrZr).

The initial preselection of fillers is given by the market classification according to the main features of the different families, the compatible brazing temperature with the base materials and prior experiences that proves the metallurgical compatibility for applications under challenge environment as high temperature, corrosion, or radiation.

Family of Ni-alloys (NB51TM, NB50TM, NB130TM and NBLMTM) provide exceptional resistance to chemical corrosion and oxidation coupled with high strength at elevated temperatures (1000 °C). Besides some of Ni-alloys are vacuum compatible. They are especially attractive for base materials with high melting point such as tungsten and P91.

Family of Au-alloys, OrobrazesTM (OB950TM, OB1025TM and OB1030TM), are supplied to vacuum grade purity standards. It is also good corrosion resistant. Gold-copper filler metals show good wetting on

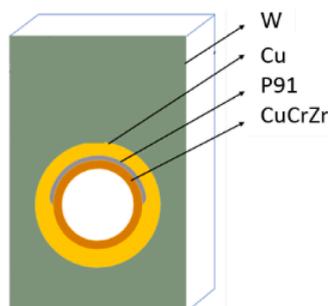


Fig. 3. PFC design proposed for upper limiter within WPBB in [15].

base materials of Table 1 and gold-nickel filler metals show high temperature strength (up to 600 °C).

Family of Ag-alloys, PallabrazesTM (PB950TM), provide similar features to OrobrazesTM. Though, generally, they present lower services temperature than gold-base alloys. PallabrazesTM based on Nickel-Palladium-Chromium (PB960TM, PB977TM) are also assessed as substitute of OB950TM according to [18].

Others such as BronzesTM (C-BronzeTM, H-BronzeTM and J-BronzeTM) are a range of special products designed for high temperature brazing of steel and carbide components. They are copper content improving wetting and molten metal flow characteristics. These products contain nickel or manganese, and it is suitable for elevated service temperature applications up to 400C.

Further from this initial preselection, the fillers shall comply with the service requirements which implies a service temperature of approx. 350 °C and to withstand the environmental conditions in terms of radiation and transmutation.

A neutron transport analysis is performed for the suitable filler metals from the market and under the 5-year pulsed operation scenario for plasma “phase-1” in DEMO. The inventory simulation code FISPACT-II [19] was used to evolve the composition of the filler material (Table 4) according to the possible nuclear reactions that each nuclide/isotope in the material can experience during neutron irradiation. Since the DEMO operational scenario has not been definitively planned, the chemical composition limits for the fillers cannot be absolutely assessed using this approximate prediction. So, the neutronic analysis performs a comparative analysis between the filler metals selecting the lowest values for each filler family.

Fig. 4 shows the activation level for the shortlisted fillers. Comparatively, it can be checked nickel-base alloys present higher activation level after a month of operation. However, other factors must be considered for assessing the integrity of the joint as the inclusions due to the transmutation or the outgassing and the joint design parameters.

Specific concentration for relevant elements that can affect the integrity of the joints are quantified (Table 5). The out-gassing due to helium and hydrogen production creates porosity in the joint affecting to the integrity. Production and precipitation of chromium might affect to joints with P91 and the low melting temperature of mercury in golden alloys compromises the integrity of the joint during operation.

Additionally, all the pre-selected filler metals transmute into heavy metals creating inclusions in the joint. Therefore, this analysis is not able of discriminate what filler metal is better than other, it is just able to do a comparative-qualitative analysis between filler metals.

Table 4 collects the chemical composition of the fillers selected from each family and compatible for this application.

Table 5 summarizes the values of quantitative production of elements that impact on integrity from the neutronic analysis for the selected filler metals. It has been selected the lowest concentration production from each filler family.

Additionally, Table 6 shows the main transmutant heavy metals produced by the selected fillers during operation. It is needed to progress on the integration and testing for the application to define the limits on transmuted elements and, therefore, on chemical composition for the fillers.

Other factors to consider are the form of the filler metal, foil, and the joint design considerations: gap, clamping force (weight), length of the

Table 4
Filler metals. Chemical composition.

Filler Metal	Wt%	Sol/liq T
OB950 TM	82% Au, 18%Ni	950 °C
PB950 TM	25% Pd, 54% Ag, 21% Cu	901/950 °C
OB1025 TM	20% Au, 78% Cu, 2% In	975/1025 °C
NBLM TM	7% Cr, 3.1% B, 4.5% Si, 3.0% Fe, 0.06%<C, Ni Bal.	970/1000 °C
H-Bronze TM	52.5% Cu, 9.5% Ni, 38% Mn	880/920 °C

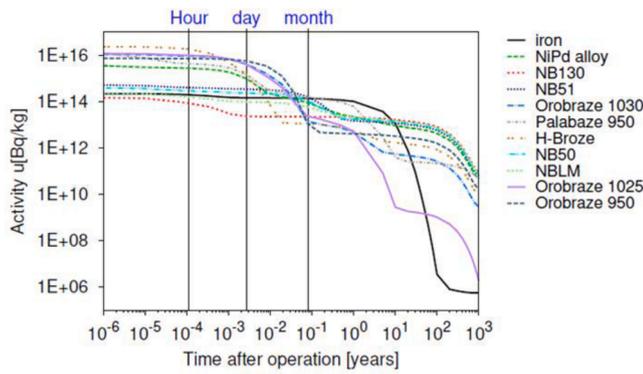


Fig. 4. Activation level for the shortlisted fillers [2].

Table 5

Neutron-induced transmutation analysis. Predicted concentration in atomic parts per million (appm) of helium, hydrogen, chromium and mercury in the 5 fillers after DEMO operation. An empty entry implies that Cr/Hg cannot be created from transmutation in that filler.

Filler Metal	He (appm)	H (appm)	Cr (appm)	Hg (appm)
OB950 TM	2.46E+3	2.68E+2	6.61E-6	5.33E+5
PB950 TM	5.77E-1	8.77E+0	-	-
OB1025 TM	1.41E+0	2.53E+1	-	8.58E+4
NBLM TM	3.09E+4	4.23E+2	6.30E+4	-
H-Bronze TM	5.62E+2	7.50E+1	3.04E-6	-

Table 6

Neutronic transmutation analysis. Main heavy metals produced under operation. Some could reach concentrations of more than 1 atomic % during operation, while others might be less than 1 appm.

Filler Metal	Heavy metals
OB950 TM	Hg, Co, Zn, Pb, Mn
PB950 TM	Cd, Zn, Sn
OB1025 TM	Hg, Zn, Sn, Cd, Pb
NBLM TM	Cr, Co
H-Bronze TM	Mn, Zn, Co

joint, surface finish and lay marks. Section 5 collects these parameters for wetting tests of multi-material for PFC. With these tests, it is initiated the optimization process to assess the metallurgical compatibility with the base materials (section 6).

4. Test configuration description

The test configuration corresponds to multi-material combination of dissimilar brazed materials, Base Mat.1 and Base Mat.2 joined by a Filler Alloy (Figs. 5 and 6). The main objective to define the test configuration is the wettability assessment, that is, the chemical compatibility between the filler and the base materials, the capillarity in “y” direction and the spreadability in “x” direction (Fig. 6 and Fig. 7). The specimens are also defined to be pulled allowing the inspection of sheared-joint and

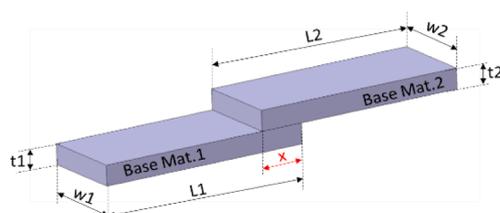


Fig. 5. Specimen dimensions.

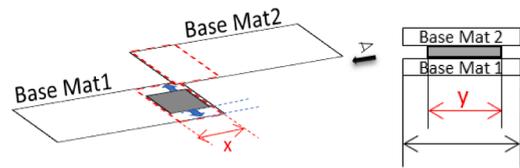


Fig. 6. Test configuration: filler alloy and base materials.



Fig. 7. Chemical compatibility: spreadability for/against the gravity (left) and capillarity in the gap (right).

capturing the ultimate sheared force. Or, alternatively, to perform optical microscopy inspection of the joint cross-section. Concretely, the microscopy has been performed in those assemblies with H-Bronze instead of sheared test to rationale the oxidation of the base materials with chromium content during the brazing process (section 6).

The geometry dimensions for the specimens are $20 \times 35 \times 5$ mm for base material 1 and 2 ($W1 \times L1 \times t1$ & $W2 \times L2 \times t2$) and the overlapped length (x) is 10 mm (2 times the thickness of the weakest item). Filler alloy area is 10×15 mm ($x \times y$), width is smaller than base material width to assess the flow in the gap (Fig. 6, left).

5. Manufacturing-test procedure

All the parts, fillers and auxiliary tools are cleaned with acetone and isopropanol. Finish surface (R_a) is measured for each part (base material) obtaining values between $0.3-0.4 \mu\text{m}$ except for the raw AM W-6% Ta which surface finish is very rough and irregular and R_a is not measurable (Fig. 9, left). Surface finish is grounded till achieving values between $0.4-0.7 \mu\text{m}$ (Fig. 9, right).

Lay marks are collected per base material. Tungsten is ground in flowing direction as Fig. 8, left. The rest of base materials are milled, Fig. 8, right. AM W-6%Ta does not present marks despite it is machined to improve the surface. It presents a surface with pores (Fig. 9, right).

The assembly of the specimens to achieve the test configuration from section 4 is performed as shown in

Fig. 10. The clamping force used for the trials is of 224.4 gr for all the material combinations of Table 1, except for H-Bronze that uses 22gr., to check the free movement of the filler due to the lack of prior experience on it.

CuCrZr parts require a pre-heat treatment, it is performed prior to the mounting of specimens. As discussed in section 3.1, CuCrZr is a precipitation hardened alloy and, during the brazing process, the temperature is close to annealing conditions ($800 \text{ }^\circ\text{C}-1,000 \text{ }^\circ\text{C}$) where nucleation occurs. The kinetic barrier of surface energy can be easier overcome allowing precipitation on the surface that affects the joint strength by the reaction with the filler metal or the creation of flaws. The pre-heat treatment is performed on the CuCrZr parts to zirconium migrates to the surface. Then, the parts are sanded to remove the zirconium (Fig. 11).

The pre-heat treatment applied is defined by a ramp up rate of $10 \text{ }^\circ\text{C/}$



Fig. 8. Lay marks of the base materials. Milled in flowing direction (right) and ground in flowing direction (left).



Fig. 9. AM W-6%Ta as built (left) and grounded (right).

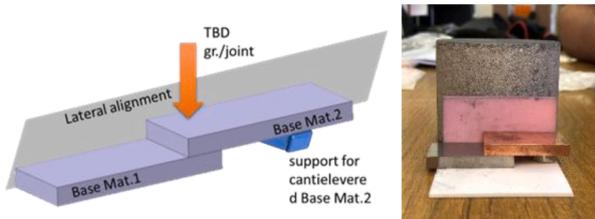


Fig. 10. Mounting of the specimen according to test configuration.

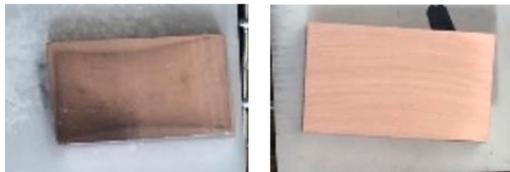


Fig. 11. CuCrZr after pre-heat treatment (left) and after sanded (right).

min till 750 ± 5 °C, followed of dwell at 750 °C for 2 hours and a cool down with nitrogen gas fan quench of approximately 50-54 °C/min.

Furthermore, the brazing cycle modifies the annealing and aging treatments of CuCrZr in which the single-phase supersaturated solid solution is created due to diffusion and the grain grown. So, the material properties are modified. Too little diffusion and the particles will be too small to impede dislocations effectively; too much diffusion and they will be too large and dispersed to interact with most of dislocations.

The material combinations of Table 1 with CuCrZr require a post-heat treatment to recover the mechanical properties in the CuCrZr. The post-heat treatment is defined by a ramp up of 10 °C/min till 475 ± 5 °C, followed of dwell at 475 °C for 3 hours and a natural cool down to room temperature with nitrogen partial pressure atmosphere.

After the pre and post heat treatments, the CuCrZr is over-annealed and over-aged. However, these treatments are sufficient for performing joint assessment.

Fig. 12 shows microscopy of CuCrZr after pre and post heat treatments. It can be seen the structure has evenly distributed grains with no signal of precipitation.

It is recommended to study the pre and post heat treatments for CuCrZr in future phases to self-correct the over-aging and annealing from the procurement phase until the operation for an integrated



Fig. 12. SEM of CuCrZr after pre/post-heat treatment. Tungsten-CuCrZr specimen brazed with H-Bronze.

component [16,20].

Several furnace runs are planned according to the different requirements for the material combinations (Table 1) and brazed temperatures (Table 7). In Fig. 13, it is shown some of these runs and the location of thermocouples.

The brazing cycles (Fig. 14) for each run are defined by:

- (1) Ramp up of 10 °C/min until achieving dwell temperature (2).
- (2) Dwell temperature at 30 °C lower than solidus temperature of the filler (Table 4) for 30 minutes for allowing quality of the furnace atmosphere and stabilization of temperature in the thermocouples.
- (3) Ramp up of 10 °C/min until achieving dwell temperature (4).
- (4) Dwell temperature at Table 7 for 10 minutes and for performing the brazing.
- (5) Cool down to room temperature. Natural cool down for all the material combination except for those that contains CuCrZr which is performed with nitrogen gas fan quench of approximately 50-54 °C/min.

All the brazing cycles are performed in vacuum atmosphere except those specimens with H-Bronze which require dry-hydrogen atmosphere (Table 7 and Table 8).

6. Test results

The total number of tests performed are: 39 wetting tests, 27 shear tests and 11 microscopy tests ([2] and [3]). After the specimens are brazed (wetting test), they are visually inspected (Fig. 7) to analyze the metallurgical compatibility between the material combination. Secondary tests (shear tests and microscopy) have been conducted to support the main conclusions from the visual inspection using magnifying glass.

The information provided for the shear tests is focused on inspecting the sheared area rather than the strength of the joint since the brazed cycle is focused on wetting tests (dwell of 10 min) and the specimen geometry is not standardized. It is recommended to increase the length of the parts to 60 mm since most of the specimen has bended due to slipping in the grips. Further, this would allow to track the curve elongation-load and ultimate strength could be compared between specimens.

This section summarizes the main results for the tests (Table 9) and analyze possible improvements on the process.

The joints are classified as good wetting, exceed wetting and poor wetting and in turn they can be acceptable or rejected. This classification has been created attending to factors that would allow to optimize the joint or the test and the main cause that drives that classification as follow:

Good wetting is defined as those joints which the optimizations should be focused on the strength and the fillet sides and shapes since the flow has been completed (y direction, Fig. 6) and the fillets are even and visible without large surface migration (Fig. 7, left) as for material combinations with OB1025TM (Fig. 15). The next step should be focused on optimizing the brazing cycle (dwell 30 min) to assess the integrity of the joint by tensile and shear tests. OB1025TM is a good filler that presents good integration between all the materials tested (Table 9).

Table 7

Brazing cycles. Dwell temperature.

Filler	(4) Brazing Temp. (10 min)	Atmosphere
OB950 TM	990 °C	Vacuum
PB950 TM	990 °C	Vacuum
OB1025 TM	1057 °C	Vacuum
NBLM TM	1057 °C	Vacuum
H-Bronze TM	960 °C	Dry hydrogen (Table 8)

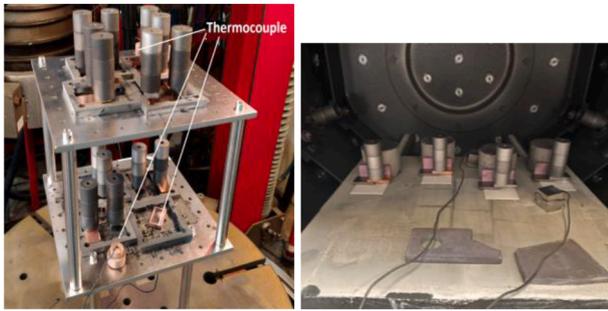


Fig. 13. Preparation of specimens to perform the tests.

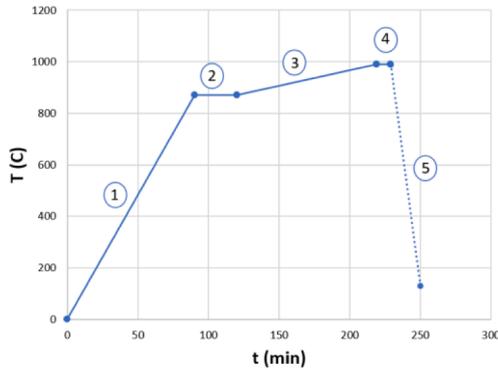


Fig. 14. Brazing cycle.

Table 8
Dry hydrogen atmosphere parameters for H-Bronze™.

Parameter	Specified during test	Proposed after test
Pressure rate	1 mbar/g	10 ⁵ to 1mbar/g
Dew Point	-51 °C of gas inlet pipe to furnace before brazing	-65 °C of gas into vacuum chamber before brazing

Good-acceptable wetting is defined as those joints that are fair in terms of homogeneous velocity between base materials. The flow to fill out the gap to the sides is homogeneous in both base materials and it is completed or not (Fig. 7, right), but it has flowed; the fillets are enough with small surface migration (Fig. 7, left) as Fig. 16 and Fig. 17. So, the improvements of the joint should be focused on factors to control the capillarity and/or chemical compatibility. In the end, control and assessment of the joint design by the surface finish, lay marks, gap, clamping force, brazing cycle, length, fit up, etc.

All these parameters have been collected during the process definition. It is recommended, for example, to increase the brazing temperature for joining AM W-6%Ta, tungsten and P91 with NBLM™ to 1065 °C. However, combinations of NBLM™ with copper or CuCrZr are limited by the grain growth of these materials. Other parameters could be modified to assess the wetting with these materials as increment of the residence time at lower brazing temperatures, increment of the applied force or further assessments of surface finishes. Generally, NBLM™ is suitable for materials with higher melting temperatures where the grain growth is not modified. The sheared tests show the softer materials as copper and CuCrZr are mainly deformed in the grips and in the center area (Fig. 18 and Fig. 20) after higher brazing temperatures. Despite NBLM™ seems to be acceptable for all the materials the brazing temperature is too high for softer materials, specially, for copper. It is recommended to perform microscopy of the base material in future test with NBLM™ to confirm the microstructure is not modified.

Poor-acceptable wetting is defined as those joints that are poor in

Table 9
Summary wetting results.

BaseMat1- BaseMat2- FillerMetal (Test Part Number)	H-Bronze™	PB950™ (BAg-32)	NBLM™ (BNi-2)	OB1025™	OB950™ (BAu-4)
AM W-Ta- OFHC Copper	–	–	Good- Accept.	Good	–
AM W-Ta- P91	–	–	Poor- Accept.	Good	–
Tungsten- CuCrZr	Repeat test (Correct dewpoint as Table 8)	Exceed- Rejected	Good- Accept.	Repeat test (Zr migration)	Good- Accept.
Tungsten- P91	Repeat test (Correct dewpoint as Table 8)	Exceed- Rejected	Good- Accept.	Good	Good
Tungsten- OFHC Copper	Repeat test (insufficient weight)	Exceed- Rejected	Good- Accept.	Good	Poor- Accept.
CuCrZr-P91	Repeat test (Correct dewpoint as Table 8)	Exceed- Rejected	Poor- Accept.	Good	Poor- Accept.
CuCrZr- OFHC Copper	Repeat test (Correct dewpoint as Table 8)	Exceed- Rejected	Good- Accept	Good	Poor- Rejected
P91-P91	Repeat test (Correct dewpoint as Table 8)	Exceed- Rejected	Good- Accept.	Good	Poor- Rejected
P91-OFHC Copper	Repeat test (Correct dewpoint as Table 8)	Exceed- Rejected	Poor- Accept.	Good	Good- Accept.

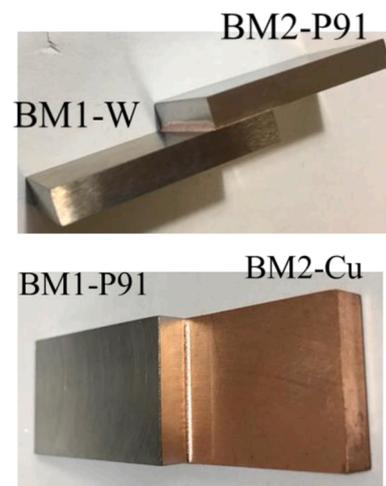


Fig. 15. Good wetting of OB1025™ on several base materials.

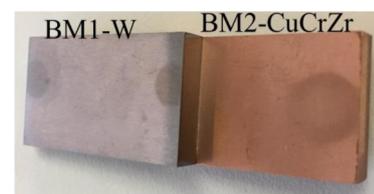


Fig. 16. Good- Acceptable wetting on tungsten-CuCrZr brazed with OB950™.

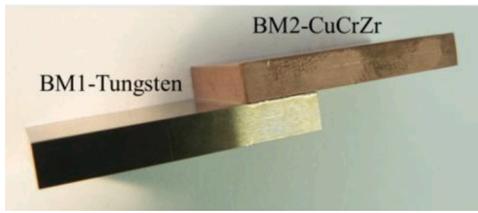


Fig. 17. Good-Acceptable wetting on tungsten-CuCrZr specimen brazed with NBLM™.



Fig. 20. Sheared CuCrZr-Cu specimen brazed with OB1025™.



Fig. 18. Sheared test on CuCrZr-Cu specimen brazed with NBLM™.

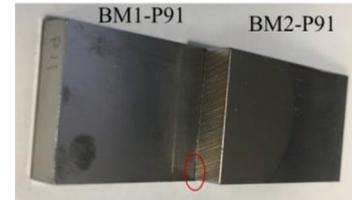


Fig. 21. Poor-Rejected wetting on P91-P91 with OB950™.

terms of non-homogeneous velocity between base materials (Fig. 7, right) as Fig. 19. The flow to fill out the gap to the sides, has not been completed and/or the fillets are small or uneven with no surface migration (Fig. 7, left). So, the improvements of the joint should be focused on factors to control the capillarity and/or chemical compatibility. In the end, control and assessment of the joint design by assessing surface finish, lay marks, gap, clamping force, brazing cycle, length, fit up, etc.

OB950™ (Fig. 16, Fig. 19 and Fig. 21) is generally acceptable for most of the combination with tungsten (Table 9). Wettability on copper and CuCrZr is poor, but the alloy is melted. It is recommended to increase the clamping force or dwell time to improve the wetting and before further studies on the surface.

In contrast to OB1025™, OB950™ has high content of gold (Table 4) which make the brazing temperature lower than OB1025™ and supposedly better compatible with softer materials as copper in terms of grain grown. However, the high content of copper in OB1025™ improves the wetting and molten metal flow characteristic as well as lower gold content minimizes the transmutation into mercury (Table 5).

OB950™ contains nickel which is very compatible with other alloying elements and offers desirable chemical and physical properties, but it is pastier. Further assessment on OB1025™ should be focused on increasing the dwell time to 30 minutes to assess the strength of the joint since the tests performed are wetting tests, but also it could be explored lower brazing temperatures depending on the base materials used in the PFC integration, specially, if copper is used. For these initial trials, the material properties of the copper seem not to be further modified for the brazing temperatures used; despite the specimen has bended during the pulling (Fig. 20), the ultimate strength on copper has been 182.93 MPa close to the theoretical one of 200MPa. Microscopy inspection should be required for further assessments.

Poor-rejected wetting is defined as those joints without flow capillarity and non-visible fillets (Fig. 21).

Exceed-rejected wetting is defined as those joints with excess of

surface migration and large spreadability, creating gaps and without fillets (Fig. 7, left) as the combination of base materials with PB950™ (Fig. 22). The silvered surfaces seem to indicate over-reaction of base materials.

Some of the tests need to be repeated due to failures in the process (Table 9). And they are collected as part of the main goal of this paper; to define the process and to start the optimization process.

For example, Zirconium was not removed properly on the CuCrZr brazed to tungsten with OB1025™ (Table 9, Fig. 23). However, the molten of OB1025™ on CuCrZr and on tungsten in other specimens was good (Table 9). So, it is expected this combination will achieve good wetting.

All the chromium content base materials brazed with H-Bronze have oxidized (Table 9, Fig. 24). Poor atmosphere is rejected as possible cause since the leak rate in the furnace is measured and it is lower than 10 microns per hour as recommended for base materials containing chromium or manganese (filler).

Scanning Electron Microscopy (SEM) with Energy Dispersive Spectrometer (EDS) on joint (Figure. 25, Figure. 26 and Figure. 27) is conducted to check any precipitation of the H-Bronze alloy (52.5%Cu, 9.5% Ni, 38%Mn) into the chromium content base materials without obtaining any signal.

The analysis reveals that the elements of the H-Bronze (Cu, Mn, Ni) diffuse favorably in the OFHC copper parent material, creating a progressive interface over approximately 10 to 20 μm. This is expected because the closer brazing temperature to liquidus temperature of copper allowing more diffusion on the side of copper than on the side of P91 where the diffusion of elements is limited to several μm for Mn or Ni and negligible diffusion of Cu.

The main cause of oxidation is due to the dew point in the atmosphere. It is recommended to reduce the dewpoint below -65°C (Table 8) to guarantee a fully control of the atmosphere in terms of oxygen reduction. The dewpoint shall be measured into vacuum chamber not at the gas source to have a right control of the atmosphere and minimize the moisture.



Fig. 19. Poor-Acceptable wetting on CuCrZr-P91 specimen brazed with OB950™.

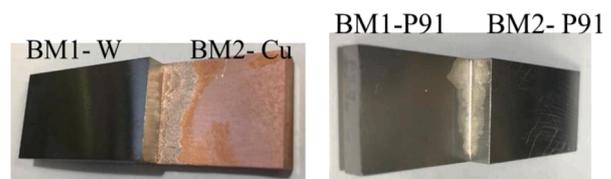


Fig. 22. Exceed of wettability of PB950™ on several base materials.

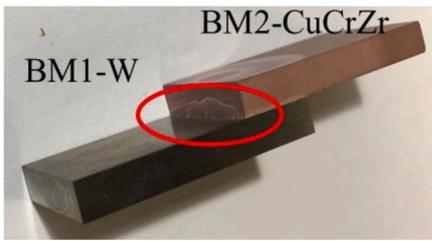


Fig. 23. Excess of wetting on tungsten-CuCrZr specimen brazed with OB1025TM.

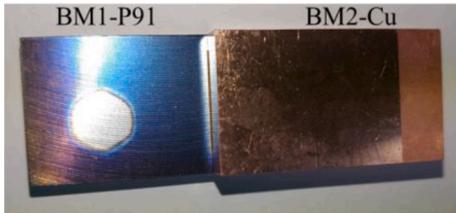


Fig. 24. Oxidized P91 during the brazing cycle with H-Bronze in dry hydrogen atmosphere.

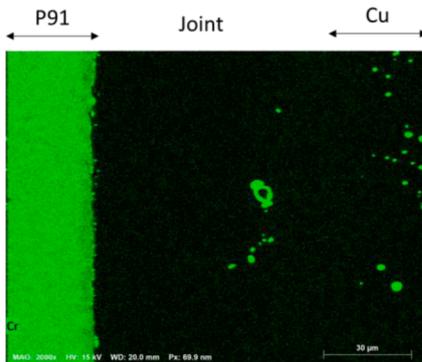


Fig. 25. SEM-EDX technique on the P91-Cu specimen brazed with H-Bronze. Chromium content in green.

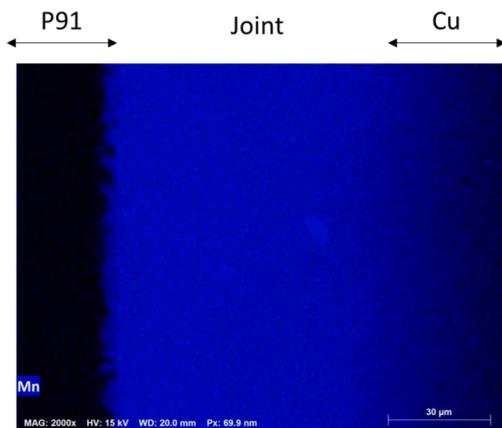


Fig. 26. SEM-EDX technique on the P91-Cu specimen brazed with H-Bronze. Manganese content in blue.

In summary, gold-copper alloy seems to be a good filler for the integration of PFC base materials in the early phases of the joint development. However, OB950TM and NBLMTM are also acceptable and they raise other advantages as better compatible brazing temperature with softer materials as copper for OB950TM or the high temperature

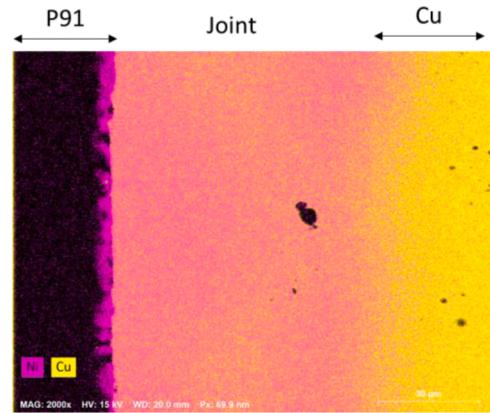


Fig. 27. SEM-EDX technique on the P91-Cu specimen brazed with H-Bronze. Nickel content in pink.

operation resistance for the NBLMTM. It is required to progress in the integration of a PFC and its testing in order to assess chemical composition limitations by the failure analysis. Therefore, the fillers can be compared between themselves.

PB950TM is rejected for the material combination of this paper (Table 1). It is recommended to perform microscopy to rationale the failure. The tests with H-BronzeTM require to be repeated with the proposed atmosphere.

General analysis of the filler metal alloys in terms of element composition shows the market covers a wide range of combination of elements to achieve specific properties in joints. So, it might be necessary to do some minor modifications on already existing filler and to make more effort on the joint definition and its optimization.

The priority is to define the process and the infrastructure to progress and to reduce the uncertainties based on an optimized joint design for PFC rather than select an optimum filler metal. The progress and the optimization on the process and joint design along the life cycle will allow to capture the functional requirements for the market to develop the filler alloys for fusion applications or to modify already existing ones.

Conclusions

The main priority of the qualification and testing of joining development for PFC is to progress with the process definition and infrastructure and therefore with the integration of a PFC. At short-term, brazing method is the most suitable technology since it is a mature technology and it allows to be focused on defining on specific manufacturing and testing procedures for PFC. Additionally, it allows to develop the joint design rather than the method. The functional requirements coming from the manufacturing and testing can be captured.

This paper presents the definition of the procedures and infrastructure for assessing wettability and capillarity of dissimilar material combination.

As part of the optimization process the following actions should be taken to update the process and infrastructure:

- Two manufacturing tests procedures should be implemented. One for wetting and capillarity tests as developed in the paper in which the main parameters of the joint design are just collected (surface finish, clamping force, brazing cycle, etc). And another for critical joints, in which these parameters can be controlled and modified; for that, it is necessary to upgrade the jig to control alignment, gap, clamping force and overlap as well as it is needed accurate control on part machining for the surface finish. This second manufacturing test procedure is focused on the joint design optimization process.

- The manufacturing test procedure allows a secondary test that support the wetting and the visual inspection. After the joint is visually inspected, it is selected the kind of secondary tests, shear test or microscopy.
- It is recommended to increase the specimen length to 60 mm per part instead of 35 mm to be able of track the complete load-elongation curve. Additionally, standardized geometry could be obtained by machining after brazing allowing standardized shear tests.
- H-Bronze requires dry hydrogen atmosphere with a dew point of -65 °C measured in the vacuum chamber not at the gas source to have a right control of the atmosphere and minimize the moisture. If a good dewpoint-meter is used and even so the dewpoint is not acceptable, it might be possible to add a desiccant-drier to dry the atmosphere prior to entering the furnace.
- The pre and post heat treatment performed for CuCrZr are the same than required for procurement specifications [16,20]. It is required further assessment on the pre and post heat treatment to bring the phase of operation to the manufacturing and therefore self-correct the procurement specifications to take into account the zirconium precipitation and the modification of mechanical properties modification during the life cycle for DEMO PFC.

The initial material testing combination for the PFC (AM W-6%Ta, W, P91, Cu, CuCrZr) are conducted with several fillers from the market (OB1025TM, OB950TM, PB950TM, NBLMTM & H-BronzeTM). The preliminary results show gold-copper alloys as a good filler for integrating all the PFC materials studied. However, other fillers as NBLMTM seems to be an attractive alloy for materials with high melting point as AM W-6% Ta, tungsten or EUROFER97/P91 since nickel-based alloys present high service temperature resistance. OB950TM presents the advantage of lower brazing temperature than OB1025TM and NLBMTM; more compatible with softer materials as copper.

Finally, H-BronzeTM is an attractive alloy with low experience in fusion and bringing the advantage of high service temperatures from the typically nickel alloys and the good wetting of copper. However, the tests require to be repeated correcting the atmosphere parameters and the weight applied to the joint. PB950TM is rejected by exceed of wetting.

Further from the wetting trials, the neutronic analysis shows the transmutation of all the filler alloys into heavy metals and it is difficult to know how these inclusions are going to affect in the life of the joint. For that, it is recommended to progress with the integration using gold-copper alloys at short-term to develop the optimization process for the joint design. Once the process and infrastructure are defined, a relative comparison between fillers through the life cycle can be performed. That is, to progress on optimizing the joint design attending to parameters as gap, clamping force, brazing cycle, surface finish, joint length, etc.

In summary, it is challenging to select the optimum filler alloy in this early phase. Progress is needed in the integration and testing on the life cycle to capture the functional requirements in terms of chemical content limitation for the fillers as well as to optimize the joint design. Consequently, the market could develop a filler for PFC.

CRediT authorship contribution statement

Main author and leader: Elena V. Rosa Adame performs the development of the methodology and testing procedure and infrastructure

Co-authors:

Andrius Aleksa junior engineer helping with procurement and test

procedures

Mark Gilbert senior engineer in charge of the neutronic analysis
 Martin Cuddy senior engineer in charge of the brazing tests
 Tristan Calver PhD in charge of microscopy for Hbronze specimens
 Zsolt Vizvary, Lead engineer for WPBB- Limiter
 Nicolas Mantel, Lead engineer for WPDIV
 Francesco Maviglia, Responsible Officer for WPBB Limiter
 Jeong Ha You, Responsible Officer for WPDIV

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This work has been carried out within the framework of the EURO-fusion Consortium and has received funding from the Euratom research and training programme 2014-2018 and 2019-2020 under grant agreement No 633053 and from the RCUK Energy Programme [grant number EP/T012250/1]. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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