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Interfacial metallurgy study of brazed joints between tungsten and fusion related materials for divertor design

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Abstract

In the developing DEMO divertor, the design of joints between tungsten to other fusion related materials is a significant challenge as a result of the dissimilar physical metallurgy of the materials to be joined. This paper focuses on the design and fabrication of dissimilar brazed joints between tungsten and fusion relevant materials such as EUROFER 97, oxygen-free high thermal conductivity (OFHC) Cu and SS316L using a gold based brazing foil. The main objectives are to develop acceptable brazing procedures for dissimilar joining of tungsten to other fusion compliant materials and to advance the metallurgical understanding within the interfacial region of the brazed joint. Four different butt-type brazed joints were created and characterised, each of which were joined with the aid of a thin brazing foil (Au80Cu19Fe1, in wt.%). Microstructural characterisation and elemental mapping in the transition region of the joint was undertaken and, thereafter, the results were analysed as was the interfacial diffusion characteristics of each material combination produced. Nano-indentation tests are performed at the joint regions and correlated with element composition information in order to understand the effects of diffused elements on mechanical properties. The experimental procedures of specimen fabrication and material characterisation methods are presented. The results of elemental transitions after brazing are reported. Elastic modulus and nano-hardness of each brazed joints are reported.

Key words: Brazed joints, Tungsten, EUROFER 97, Cu, SS316L, Au based filler, Nano-indentation

Introduction

Tungsten and tungsten alloys have been considered as primary candidate materials for helium cooled DEMO divertor and possibly for protection of helium cooled first wall in DEMO applications. [1-4] This is directly related to their attractive physical properties, namely, high melting point, high thermal conductivity, high ultimate tensile strength, high yield and shear strength and relatively low coefficient of thermal expansion [1][2]. Joining tungsten divertor components to other suitable structural materials is critical to the success of DEMO and high temperature brazing has been chosen as one of suitable joining technologies [1][3][4]. In the developing HEMJ divertor design [2], each of the cooling fingers consists of a W tile for shielding and a W-1%La_2O_3 (WL10) thimble for heat sinking. The fingers are connected to a reduced activation ferritic-martensitic (RAFM) EUROFER steel body by brazing. Considering the large mismatch in thermal expansion coefficients of W ($4.2 \times 10{-}6$ 1/K at RT) and RAFM steel (ca. $12 \times 10{-}6$ 1/K at RT)[5] under the severe DEMO divertor working conditions, the brazed joints are critical as a result of them being exposed to thermal cyclic loads in both water-cooled and he-cooled divertor applications. Furthermore, other dissimilar material properties such as the Young's moduli and yield stress, in combination with the mismatch in thermal expansion coefficient, results in high residual stresses in the joint regions as a result of the joining process[2]. Kalin [6] developed a brazing process to join W to a ferritic/martensitic steel for use on Helium-cooled divertors and other plasma facing components. It was found that cracks initiated in the tungsten a small distance away from the brazed layer due to significant residual stresses. This phenomenon raises significant challenges in relation to brazed joints between W and other dissimilar metals, either during service conditions or as a result of the fabrication process.

In previous research [1-3], Pd60Ni40 (liquidus temperature T_{lq}=1238°C) was used for brazing W-WL10 and Pd18Cu82 (T_{lq}=1100°C) was used for WL10-steel using the vacuum furnace brazing method. In both cases successful brazed joints were achieved. In the W-WL10 joint with PdNi filler, significant diffusion of tungsten was observed in the brazed layer. In Munez's [7] work, Ni55Ti45 alloy filler wire was used for joining W-Ti-Y_2O_3 alloy and EUROFER steel by means of laser brazing and it was found that NiTi filler showed low brazeability. Cracks caused by residual stresses initiated from the brazed layer and extended to the parent materials. Energy dispersive X-ray spectroscopy (EDS) analysis showed elements of tungsten alloy and NiTi filler diffused into each other after brazing. However Ehrlich [8] detected nickel alloys with significant embrittlement effects after neutron loading testing (c. 150dpa) and indicted a reduction of performance. Reiser [5] noted that the brazed joint of W to structural materials is a critical area when exposed to thermal cyclic load and reported that brittle intermetallic compounds should be avoided under all circumstances and W solid solution should be avoided if possible. It was also noted that producing W laminates, the joining of the foils is also an essential issue [9]

In the present study commercial quality Au80Cu19Fe1 brazing filler was used as gold-based alloy foils are recognized as providing good wettability on tungsten, good resistance to oxidation and corrosion at high temperatures and can create ductile joints without excessive inter-alloying / erosion of the parent metals. Fig. 1 showed the phase diagram of the Au-Cu system, modified from [10]. In this case, solidification started from a disordered face-centred cubic (fcc) structure (Au,Cu). This structure transferred into a long period ordered structure AuCu **II**. AuCu **II** then transferred to a face-centred tetragonal structure AuCu **I**, which is stable at low temperatures. [11][12]

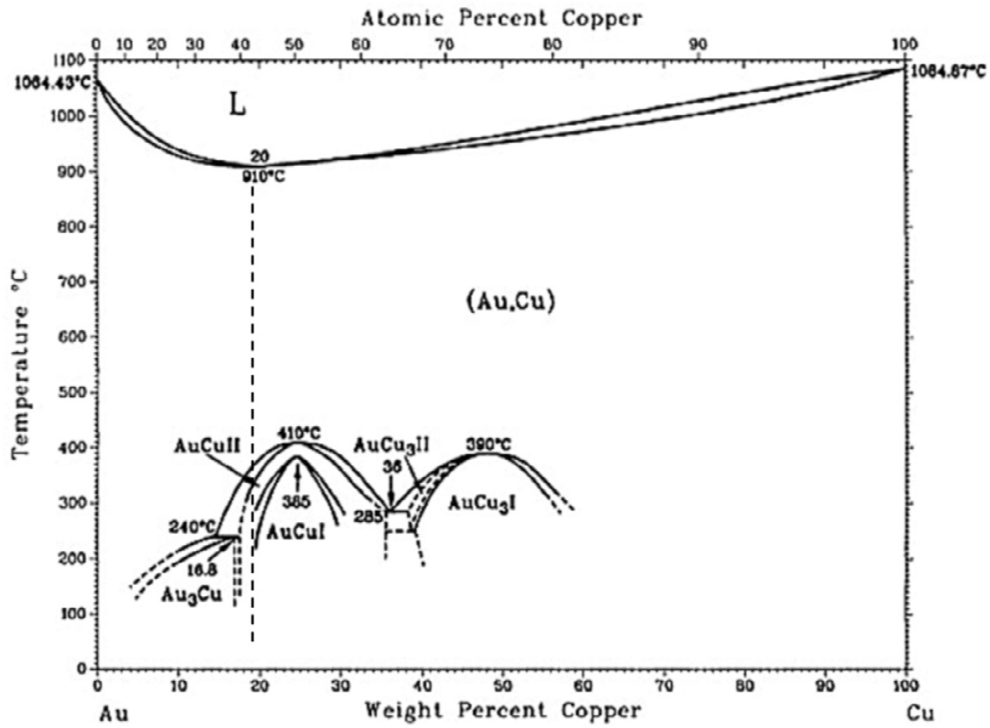


Fig. 1. Au-Cu phase diagram [10]

The study focuses on brazing of pure W with other fusion related materials i.e. pure W itself, EUROFER 97, AISI SS316L and oxygen-free high thermal conductivity (OFHC) Cu, to assess the brazing quality and applicability of this gold-based filler. Reliable joints between W and EUROFER steel are required for both He-cooled and improved water-cooled divertor mockup design [2][5]. The ITER divertor is using CuCrZr pipe [13], but in DEMO conditions, CuCrZr shows a shortened lifetime for the water-cooled divertors. However, Cu has excellent thermal conductivity and heat removal capacity compared with EUROFER steel. Thus, there are still possibilities of using Cu as a heat sink material in the future improved water-cooled divertor. Comparing with EUROFER, SS316L has been successfully used in fission reactor technology and has been well used in industry [8]. SS316L was selected as the primary structural materials for ITER [14] and also a candidate to be used in divertor and blanket design. [15] [16].

A comprehensive microstructural analysis of the interfacial region of brazed joints between the aforementioned materials has been undertaken. Flaws and imperfections were accessed for each brazed joint using the International Standard [17] and the braze quality showed a degree of uniformity in different material combinations. No cracks were detected after brazing, however varies defects were detected: (1) cavities and pores in the brazed layers, (2) filling imperfections, incomplete filling of brazing gap, (3) excess braze metal solidified onto parent materials, (4) recessed braze joints, the surface of the braze filler material in the brazed joint is below the required dimension. Varying degrees of these defects were observed in the joints of all materials combinations.

The microstructures of brazed joints, in different combinations of materials brazed to pure tungsten, were characterised. Additionally, interfacial reactions and elemental diffusion behavior of each material combination have been analysed and discussed. Nano-indentation measurements were also undertaken to generate local mechanical properties correlated to the interfacial reaction and diffusion phenomena due to the brazing procedures. The tests were performed at the interfaces of each material combination. In this study the continuous stiffness measurement (CSM) technique was used to study average elastic modulus and hardness values over an indentation range as discussed in previous works [7][18].

Experimental procedures

Prior to brazing, all the experimental materials were prepared using the same conditions in order to maintain consistency. The parent materials used for characterisation work were commercially pure tungsten, OFHC copper, AISI316L in the cylindrical butt form with dimensions of Φ12.7mm x 10mm and EUROFER97 with dimensions of Φ10mm x 10mm, as shown in Fig. 2. The samples were machined on a CNC lathe to ensure a consistent surface finish.

The dissimilar samples were brazed in butt joint form with the filler 'Orobraze 910', supplied by Johnson Matthey. The chemical composition in weight percentage is 80% Au, 19% Cu and 1% Fe and the working temperature range is 908-910°C. Braze alloy foils of 0.0508 mm (0.002 inch) were utilised throughout the experiments. Specimens were set up and aligned by means of a jig. Some pressure was applied on the specimens by added weight and no spacers were used.

Vacuum furnace brazing was performed at 5×10^{-6} millibar. The heat cycle of brazing was set to heat up by 10°C/min to approximately 900°C then dwell for 5 minutes then heated up by 10°C/min to approximately 1000°C followed by a dwell of 5 minutes. On completion the furnace was switched off and kept under vacuum so that samples could cool down very slowly to avoid thermal shock. The butt brazed specimens are shown in Fig. 2.

The post brazed specimens were cut by a low speed diamond disk on a cut-off machine and prepared following a standard metallographic procedure. The etchant used for W was Murakami's reagent and for Cu was the $FeCl_3$ solution. Joining quality was assessed by use of optical microscopy. A Hitachi 3700W-filament SEM was used for imaging and energy dispersive X-ray spectroscopy (EDS) was used to confirm the compositional variation. The analysis X-ray point has a minimum diameter of 1~2µm. An Agilent nanoindenter G200 fitted with a Berkovich indenter was used for determining the hardness and elastic modulus values of the phases observed in the brazed joints. Utilising the CSM module of the nanoindenter, average hardness and elastic modulus values over an indentation depth between 100 to 1000nm were analysed.

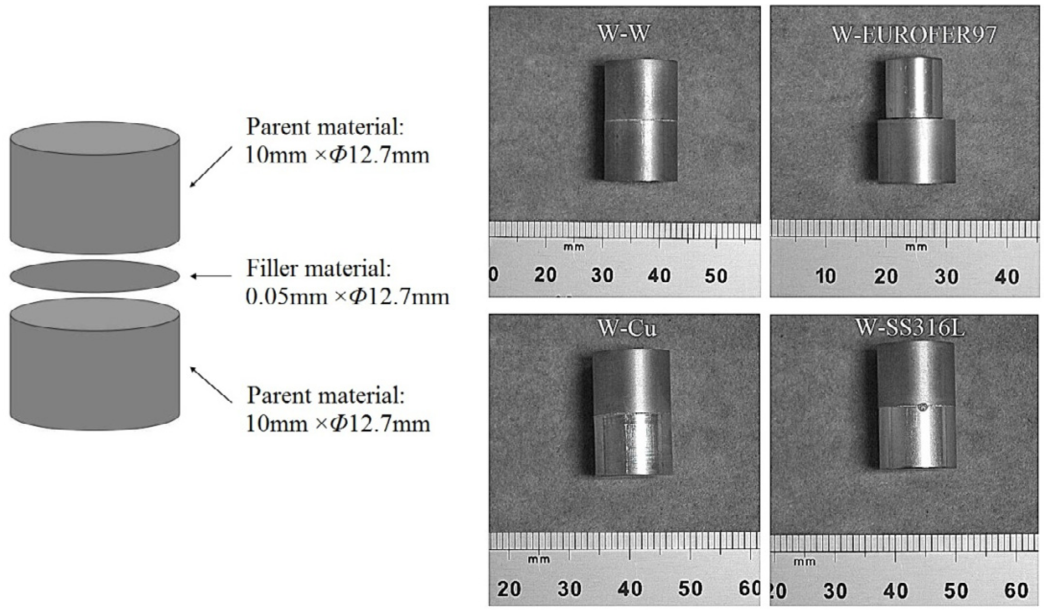


Fig. 2. Material dimensions and butt joined brazed specimens

Results and discussion

W-W joint

Fig. 3a is a SEM backscatter image of the brazed joint. As the W has been etched, elongated grains of W can be observed. The nano-indents in Fig.3a are used for benchmarking the EDS analysis regions. A line scan analysis was developed by crossing the braze joint to generate elemental transition information and the results are shown in the Fig. 3b. The analysis makes 150 measurements through a distance of c. 115µm. The EDS analysis highlights that there are no diffusion regions created at the AuCuFe/W interfaces and the elements from W and AuCuFe filler do not diffuse into each other. However, in Fig.3c, an optical microscope image at the AuCuFe/W interface, filler material penetrations can be observed. This phenomenon is likely to be grain boundary diffusions or micro cracks on the W surface that have been filled with braze material. A further EDS element analysis was performed at the AuCuFe/W interface. Spectrums at 4 different locations were analysed and shown in Fig. 3d and results are shown in Table 1. Spectrum 1 and spectrum 3 are both about 1μm away from brazed interface. Spectrum 2 and spectrum 4 are approx.. 5μm away from interface. The compositions shown in Table 1 confirmed that no diffused filler materials were detected on W side. This is because the amount of grain boundary diffused materials is very small and randomly distributed and as a result was not picked up by the EDS scan.

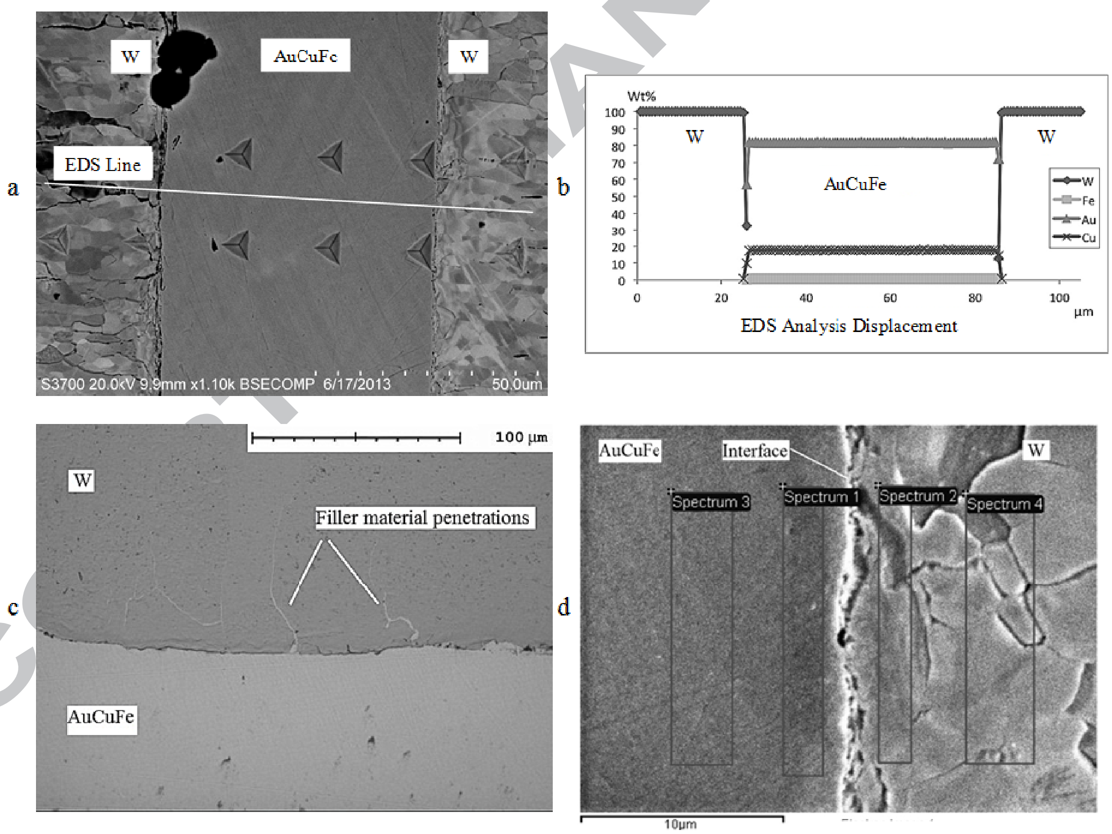


Fig. 3. (a) Backscatter SEM image of W/AuCuFe/W joint; (b) EDS analysis across the joint; (c) Optical image shows interface of W/AuCuFe; (d) SEM image with EDS analysis at the interface of AuCuFe/W.

Table 1:

Elemental compositions measured at AuCuFe/W interface

	Au	Cu	Fe	W	Total Wt%
Spectrum 1	81.68	17.49	0.83		100
Spectrum 2				100	100
Spectrum 3	81.53	17.59	0.88		100
Spectrum 4				100	100

Nano-indentation tests, shown in Fig. 4a, were performed at the interface between AuCuFe filler and W. The maximum indentation depth was 1000nm. The indentations across the interface were numbered from 1 to 12. Element compositions at each indent were analyzed by EDS point scan and the results are shown in Fig. 4b. Reading from Fig. 4a and Fig 4b, indents 1-4 were located at the AuCuFe filler region and 5-12 were located at the W region. The values of elastic modulus and hardness measured by nano-indentation are shown in Fig. 4c respectively. For the indents 1-4 at filler region, the mean value and standard deviation of E = 155.47 ±3.73 GPa, and H = 4.65 ± 0.13 GPa. For the indents 5-12 at W region, E = 405.55 ± 27.88 GPa and H = 7.21 ± 1.13 GPa. The E and H at AuCuFe were constant while those measured at W showed larger standard deviations, as a result of the nano-indentation being affected by sample surface conditions. Because in this joint W was polished and etched while the braze filler was only polished.

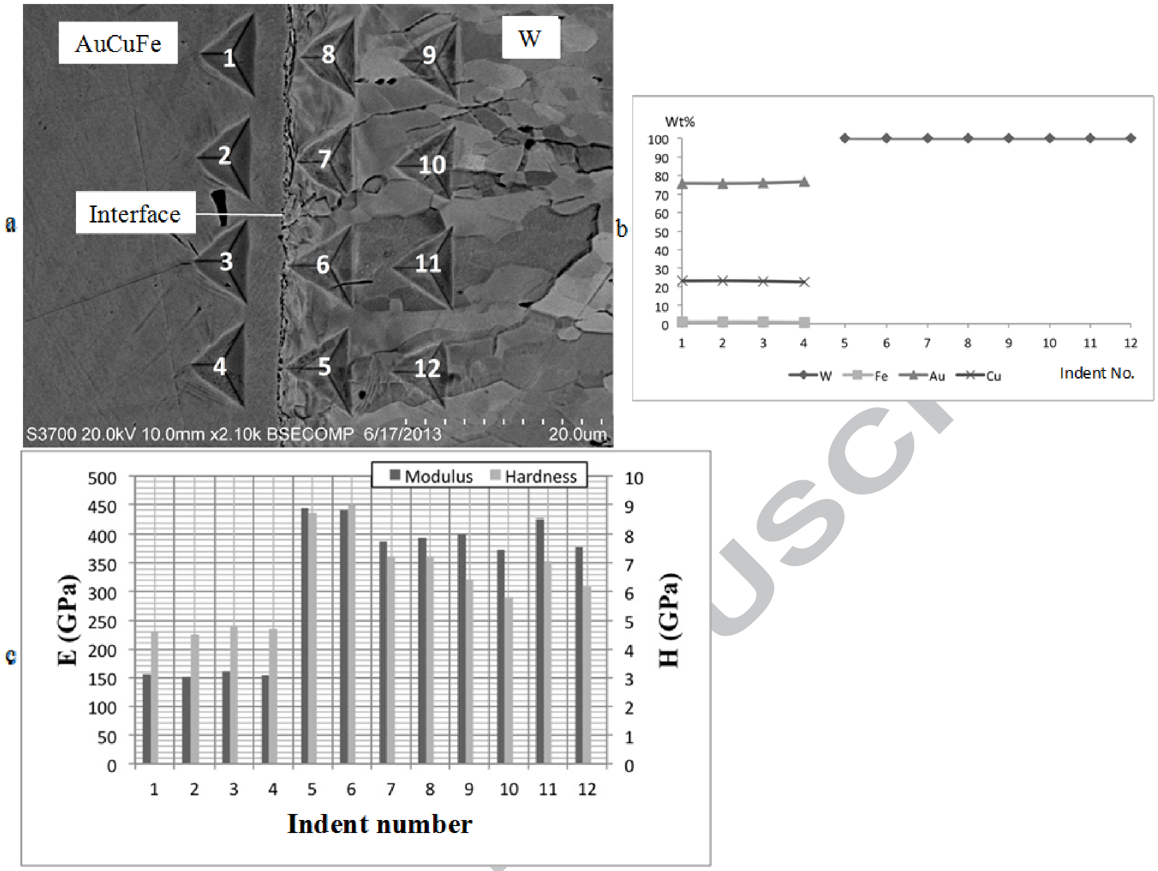
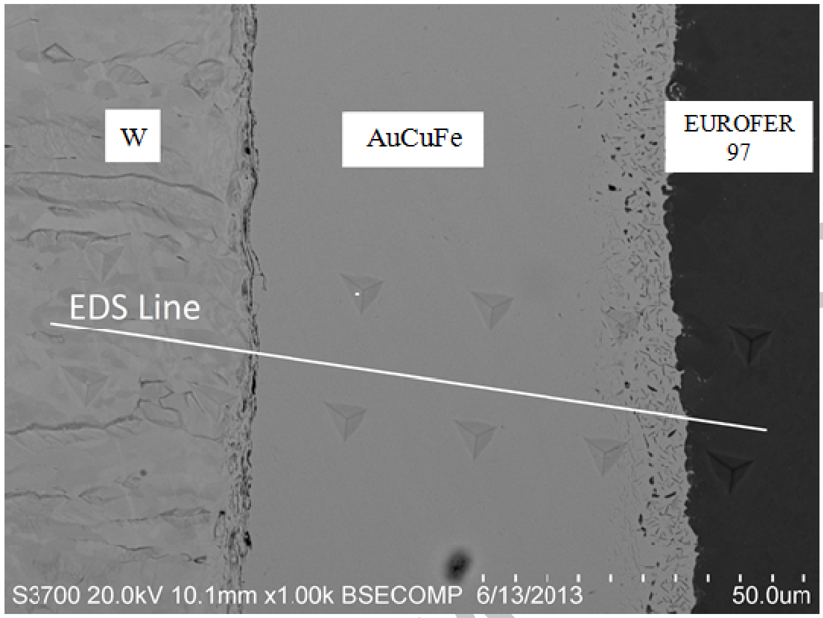


Fig.4. (a) SEM image shows indentation locations; (b) Element compositions of indentations; (c) Elastic modulus and Hardness, measured from nano-indentations

W-EUROFER 97 joint

The experiment setups for analysing W-EUROFER 97 were the same as those used in W-W joint. Fig. 5a shows the backscatter SEM image of the brazed joint. A diffusion region with elemental diffusion (mainly Fe) on the interface between AuCuFe filler and EUROFER 97 can be observed. An EDS analysis was done along the line shown in Fig. 5a and the results are shown in Fig. 5b. The line analysis measured composition at 150 points along a distance of 110µm. Reading from the measurements, the diffusion region contains diffused Fe and Cr. Diffusion of Fe reduced with the distance from the interface. Furthermore, Fe diffused further distance into brazed joint than Cr but did not penetrate beyond at the interface of W/AuCuFe.

a



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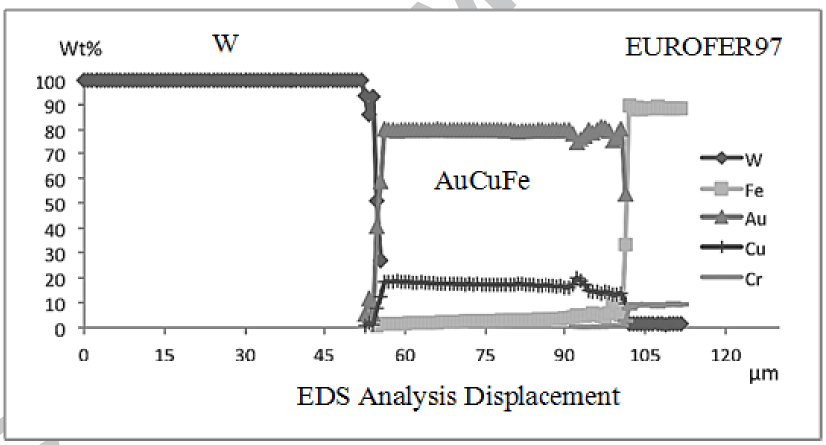

Fig.5. (a) Backscatter SEM image of W/AuCuFe/EUROFER joint; (b) EDS analysis across the joint

An elemental mapping analysis was generated at the interface of AuCuFe/EUROFER 97 in order to understand the diffusion behaviour. The results are shown in Fig. 6. Fig. 6a is an SEM image showing the microstructure of the diffusion region at x5000. Fig. 6b shows elemental maps of the primary elements Au, Cu, Fe and Cr. The elemental maps demonstrate the dispersion behaviour of these elements after brazing. The iron dispersion map showed that the dark microstructures at diffusion region were majorly formed by diffused iron.

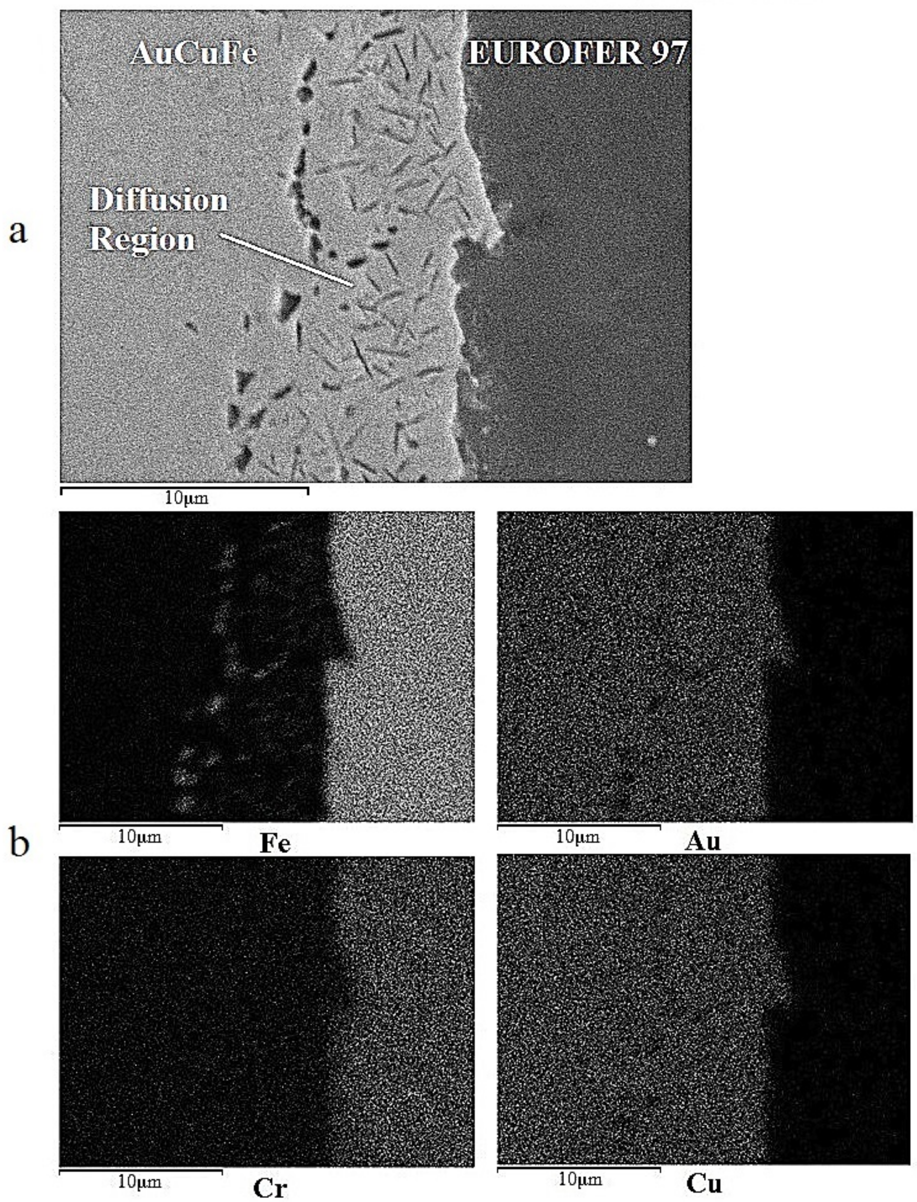

Fig. 6 (a) SEM image at interface of AuCuFe/EUROFER 97; (b) elemental maps of primary elements in the brazed joint

Nano-indentation tests were conducted at the interface of the AuCuFe/EUROFER 97, with a maximum indent depth of 1000nm. The indents are numbered from 1 to 12 as shown Fig. 7a and correlated to the composition measurements shown in Fig. 7b.

Indents 1 to 4 were located in the AuCuFe, 5 to 8 were located in the diffusion region and 9 to 12 were located at the edge of the EUROFER 97. The results of elastic modulus and hardness are shown in Fig. 7c. The values of modulus and hardness were calculated from 400-900nm to avoid the influence of the diffusion region. For the indents 1-4 at filler region, E = 136.98 ± 6.81 GPa, and H = 4.54 ± 0.28 GPa. For the indents 5-8 at diffusion region, E = 140.71 ± 8.78 GPa, and H = 4.11 ± 0.26 GPa. For the indents 5-12 at EUROFER region, E = 235.22 ± 15.11 GPa and H = 3.2 ± 0.77 GPa. The results at diffusion region had similar E and H to those in the AuCuFe filler and no embrittlement due to the diffusion and interface alloying was apparent.

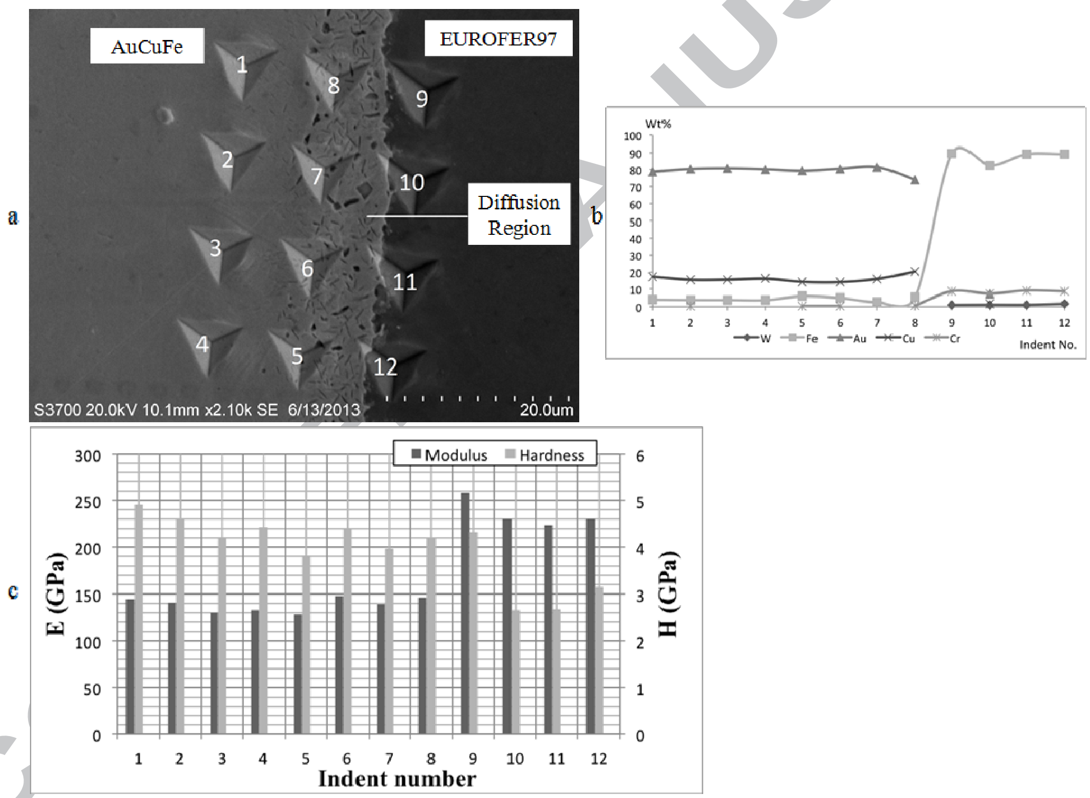

Fig. 7 (a) SEM image shows indentation locations; (b) Element compositions of indentations; (c) Elastic modulus and Hardness, measured from nano-indentations

<u>W-Cu joint</u>

The backscatter SEM image of the joint in Fig. 8a shows a very smooth diffusion region at the interface between the AuCuFe and Cu. The EDS line analysis location shown in Fig. 8a used 150 points over 200µm across the brazed layer. The result clearly shows that in the brazed joint, composition of Cu was increased from 19% to about 30% as result of diffusion. A smooth transition region around 40 microns thick was created by diffused Au and Cu was observed at the interface between the braze filler and the Cu. Results at the W interface were as reported earlier in W-W joint.

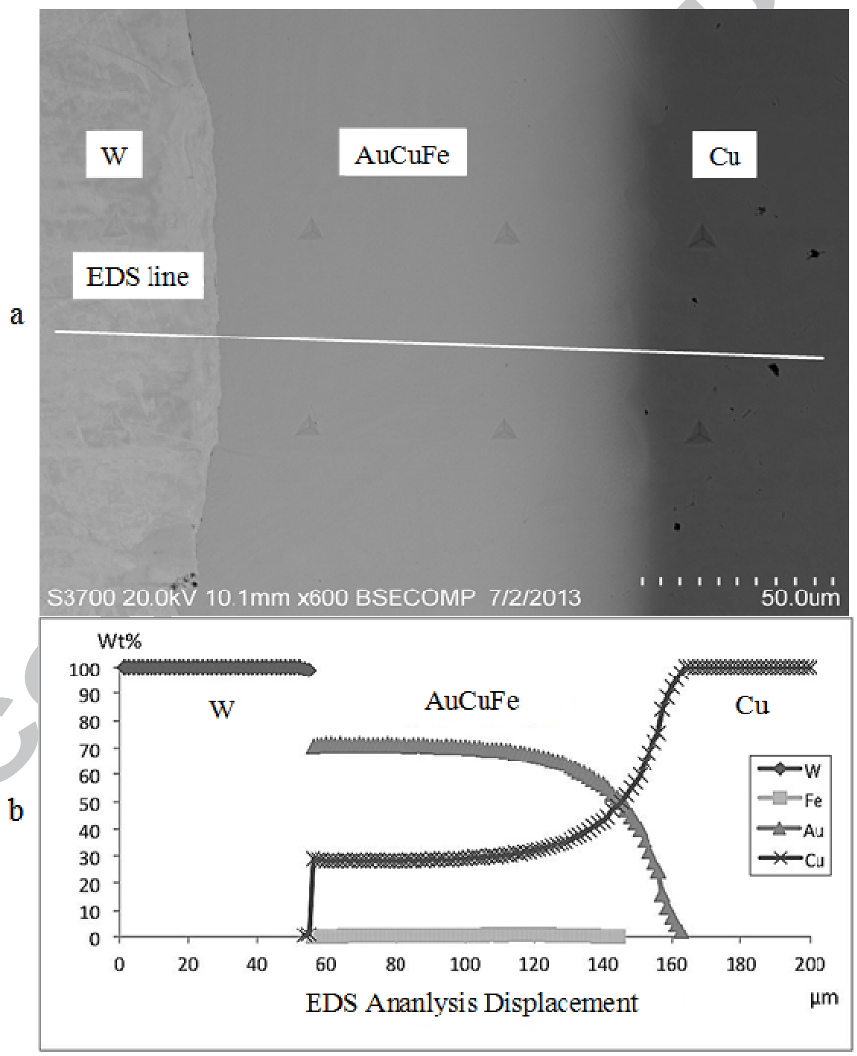

Fig. 8: (a) Backscatter SEM image of W-Cu brazed joint; (b) EDS analysis across the

joint

Nano-indentations were performed at the interface between the AuCuFe and Cu, with a maximum indent depth of 800nm. The indents were numbered from 1 to 12 as shown in Fig. 9a and elemental composition is shown in Fig. 9b. The results for elastic modulus and hardness are also shown in Fig. 9c. Values of E and H measured at filler region were E = 135.04 ± 4.22 GPa and H = 2.39 ± 0.23 GPa. At diffusion region, E = 149.76 ± 4.92 GPa, and H = 1.94 ± 0.27 GPa. At Cu region, E = 145.57 ± 1.1 GPa and H = 1.57 ± 0.27 GPa. Comparing with other types of brazed joints, the hardness values measured at the filler region in this joint was distinct smaller. This is likely to be due to the diffusion of Cu from the parent material. The values measured at the diffusion region were similar as those measured in the brazed interlayer (AuCuFe). This shows that the homogeneous conditions produced in this region have resulted in uniform properties across the interface of the brazed joint on the pure copper side whereas, considering the EDS results showed in Fig. 8b, a more heterogeneous and non uniform property distribution was identified on the pure tungsten side of the brazed joint.

a b c

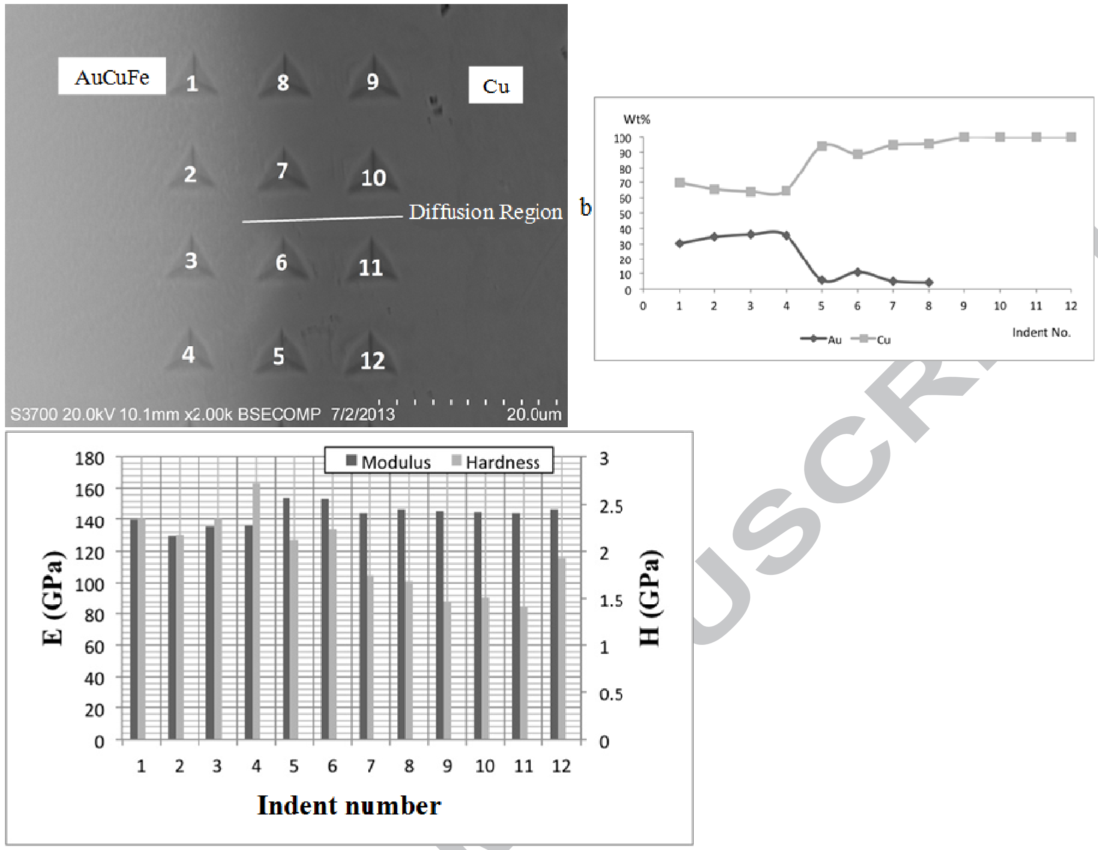

Fig. 9 (a) SEM image shows indentation locations; (b) Element compositions of indentations; (c) Elastic modulus and Hardness, measured from nano-indentations

<u>W-SS316L joint</u>

A backscatter SEM image of the W-SS316L joint is shown in Fig. 10a. An EDS line analysis, Fig. 10a, was performed across the joint from the W to the SS316L. The analysis took 150 measurements within 125µm distance across the brazed joint and elemental transition results are shown in Fig. 10b. Both Fe and Cr diffuse across the filler region and a region with diffused Fe, Cr and Ni is apparent in the AuCuFe adjacent to the SS316L.

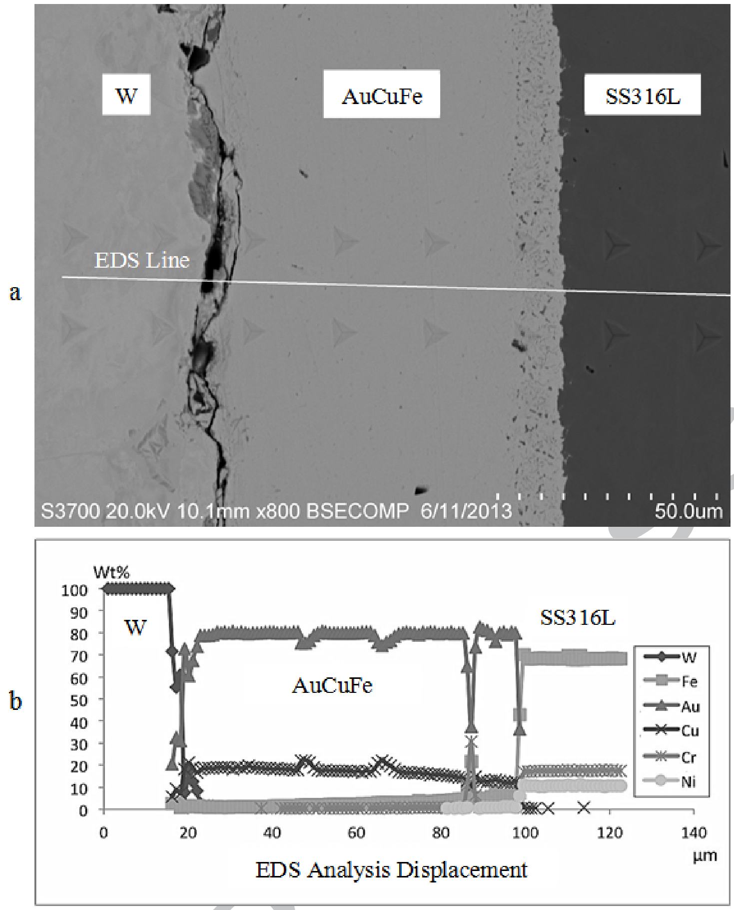

Fig. 10 (a) Backscatter SEM image of W-Cu brazed joint; (b) EDS analysis across the joint

Nano-indentation tests were also performed at the interface of the AuCuFe/SS316L with a maximum load of 10μN and maximum indent depth of 1000nm. The indents were numbered from 1 to 12 as shown Fig. 11a and correlated to the composition measurements shown in Fig. 11b. Indents 1 to 4 were located in the AuCuFe, 5 to 8 were located in the diffusion region and 9 to 12 were located in the SS316L. The results of elastic modulus and hardness are shown in Fig. 11c. Values measured at filler region were E = 135.27 ± 1.34 GPa and H = 4.12 ± 0.06 GPa. At diffusion region, E = 141.58 ± 2.82 GPa, and H = 3.75 ± 0.0.09 GPa. At Cu region, E = 207.7 ± 1.93 GPa and H = 3.45 ± 0.27 GPa. No embrittlement effects relating in high hardness due to diffusion are detected at the interface and the measurements at diffusion region show similar mechanical properties to those in the AuCuFe filler.

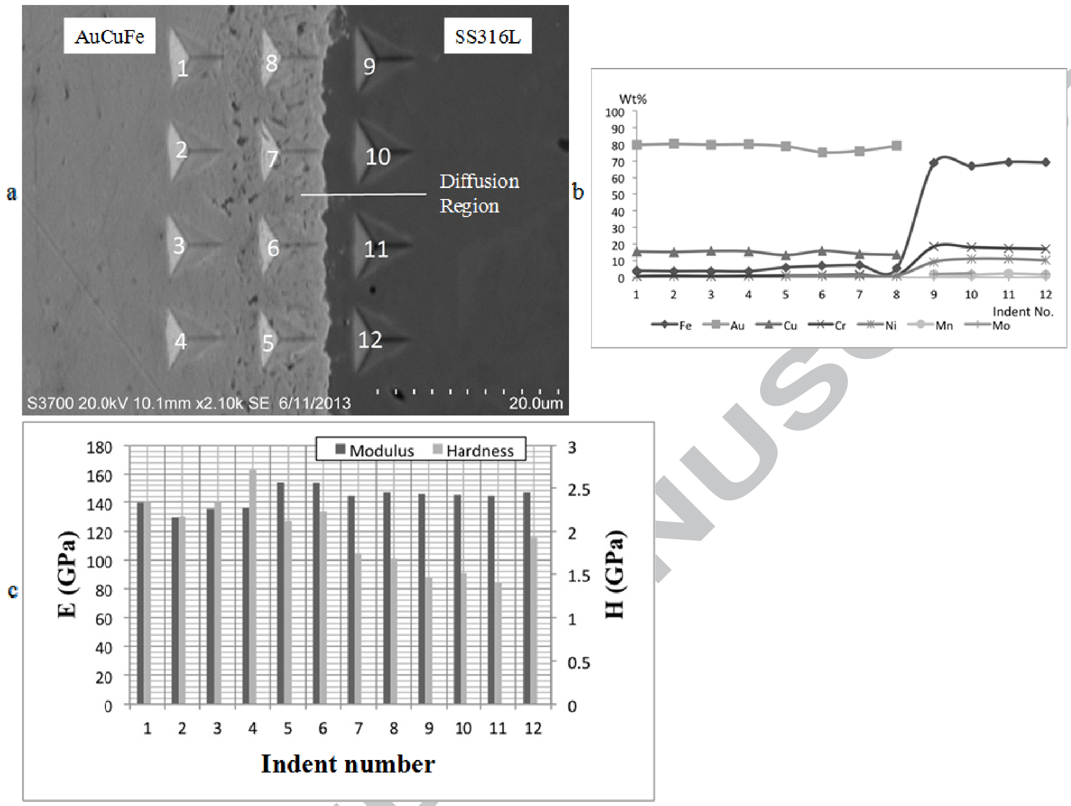

Fig. 11 (a) SEM image shows indentation locations; (b) Element compositions of indentations; (c) Elastic modulus and Hardness values measured from nano-indentations

Conclusions

This work is part of a project aimed at designing and fabricating reliable brazed joints between W and other dissimilar materials. The samples were furnace brazed in butt joint form under vacuum with Au80Cu19Fe1 filler. EDS analysis and nano-indentation was performed at the joint to understand interfacial metallurgy and generate elastic modulus and nano-hardness values. The results of this study can be concluded as following:

W-W

AuCuFe filler creates a uniform joint between W butts. The EDS analysis did not detect elemental transition at the brazed interface between the W and AuCuFe filler. However, melted filler material penetrating into the W was observed by optical microscopy. This is likely to be due to grain boundary diffusions or micro cracks on the W surface that have been filled with braze material. A further analysis at the interface did not detect filler material penetration and elemental transitions. Nano-indentations were performed at the interface between the AuCuFe and W. The elastic modulus and hardness values measured in the AuCuFe were constant while the modulus and hardness measured in the W side adjacent to the braze shows larger variations.

W-EUROFER 97

An EDS line analysis performed across the brazed layer showed no elemental transitions either from W to AuCuFe or from AuCuFe to W. A transition region with complex microstructures was observed at the interface between the AuCuFe filler and the EUROFER 97 after brazing. Elemental mapping analysis confirmed that the transition region consisted of diffused Fe microstructures. Nano-indentations were performed at the adjacent region between the AuCuFe and the EUROFER 97 and the transition region showed similar mechanical properties to the AuCuFe filler. No embrittlement effects due to diffusion were detected at the interface of AuCuFe and EUROFER 97.

W-Cu

No elemental transitions were detected at the W and AuCuFe interface. A very smooth elemental transition was detected at the adjacent region between the AuCuFe and Cu. The smooth transition of elements indicated that the material properties are changing smoothly. The mechanical properties of diffusion region were similar to the braze layer. The homogenous conditions produced in this region resulted in uniform properties across the interface of the brazed joint on the pure copper side.

W-SS316L

The EDS line analysis performed across the brazed layer found no elemental transitions at the W and AuCuFe interface. A transition region created at the adjacent region between the AuCuFe and SS316L was detected. No embrittlement effects due to diffusion were detected at the interface.

Some general conclusions can be made at this stage:

- Joining of W is an important point for the development of a DEMO reactor but is also an important point for the development of a W laminate used as a structural part for a DEMO divertor. [9]
- AuCuFe filler can be used to fabricate brazed joint between W and the dissimilar materials considered, EUROFER 97, Cu and SS316L, and create a uniform brazed layer.
- Parent materials showed no evidence of erosion under these brazing conditions.
- No elemental transitions were detected between the W and the AuCuFe filler in either direction.

- No W solid solutions or intermetallic compounds were found in the joint.
- No evidence of oxidations was detected.
- Transition regions between the AuCuFe filler to EUROFER97/316L showed similar elastic modulus and hardness to the braze filler.
- A very smooth elemental transition was detected between the AuCuFe filler and Cu. This would indicate that the material properties were changing smoothly from filler to Cu.

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Highlights

- We created brazed joints between tungsten and EUROFER 97, Cu and SS316L with Au80Cu19Fe1 filler.
- No elemental transitions were detected between the W and the AuCuFe filler in either direction.
- Transition regions between filler to EUROFER97/316L showed similar elastic modulus and hardness to the filler.
- Smooth elemental and mechanical properties transition were detected between the filler and Cu.