



# Effects of heat treatments on microstructure changes in the interface of Cu/SS316L joint materials

Q. Xu <sup>a,\*</sup>, D.J. Edwards <sup>b</sup>, T. Yoshiie <sup>a</sup>

<sup>a</sup> *Research Reactor Institute, Kyoto University, Kumatori-cho, Sennan-gun, Osaka-fu, 590-0494 Japan*

<sup>b</sup> *Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA 99352, USA*

## Abstract

Precipitation and dispersion strengthened copper alloys joined with 316L austenitic stainless steel are expected to be heat sink materials in the first wall and divertor of International Thermonuclear Experimental Reactor (ITER) owing to the good thermal conductivity of Cu alloys. In the present study, the effects of heat treatment on microstructural stability in the interface of CuNiBe/SS316L and CuAl25/SS316L have been investigated. In the as-received CuNiBe/SS316L joints, voids were observed at the interface, and in the stainless steel side near the interface. But in the CuAl25/SS316L joints, voids were observed only in the Cu side near the interface. These voids would have a significant effect on the mechanical properties of joints. The results of annealing experiments showed that the microstructures in the interface of both types of joints were thermally stable during annealing at 573 and 673 K for 100 h. © 2000 Elsevier Science B.V. All rights reserved.

## 1. Introduction

The structures of the first wall and divertor in the International Thermonuclear Experimental Reactor (ITER) device are composed of three bonded materials: Be (or W)/Cu/SS316L, where Be or W is the plasma facing armor, the copper alloy acts as a heat sink and the SS316L provides the load bearing support [1]. During operation, the components of first wall and divertor may experience temperatures in the range of about 423 and 603 K [2]. It is therefore important to investigate the effects of heat treatment on stability of the joint material microstructure and their interfaces since poor properties of the interface can affect the entire structure. The objective of the present study was aimed to examine the thermal stability of the interface and the adjacent material in GlidCop CuAl25/SS316L and CuNiBe/SS316L joints.

## 2. Experimental procedure

GlidCop CuAl25/SS316L and CuNiBe/SS316L joints were examined in this study. Both kinds of coupling were made utilizing the technique of hot isostatic pressing (HIP). The GlidCop CuAl25 alloy was produced by SCM Metals. CuAl25 alloy is dispersion strengthened by a fine dispersion of Al<sub>2</sub>O<sub>3</sub> particles. It contains 0.25 wt% aluminum in the form of Al<sub>2</sub>O<sub>3</sub>. CuNiBe alloy was produced by Brush Wellman, alloying element amounts are 1.98 wt% Ni and 0.32 wt% Be. The joints were fabricated at a bonding temperature of 1255 K for CuAl25/SS316L and at 1245 K for CuNiBe/SS316L. The pressure and the holding time for the HIP process were fixed at 101 MPa and 2 h [3].

The joints were thinned to 0.1 mm and cut into 3 mm discs for transmission electron microscope (TEM) observation. Two kinds of annealing experiments were carried out to investigate the microstructural stability of the joint. Isochronal annealing of thinned TEM specimens of CuAl25/SS316L was conducted in situ using a high temperature heating stage in a JEOL 2000FX TEM. The specimens were annealed over the temperature range of 298–973 K at 100 K intervals, and the temperature was maintained for 30 min. The bulk

\* Corresponding author. Tel.: +81-724 512 404; fax: +81-724 512 620.

E-mail address: xu@rri.kyoto-u.ac.jp (Q. Xu).

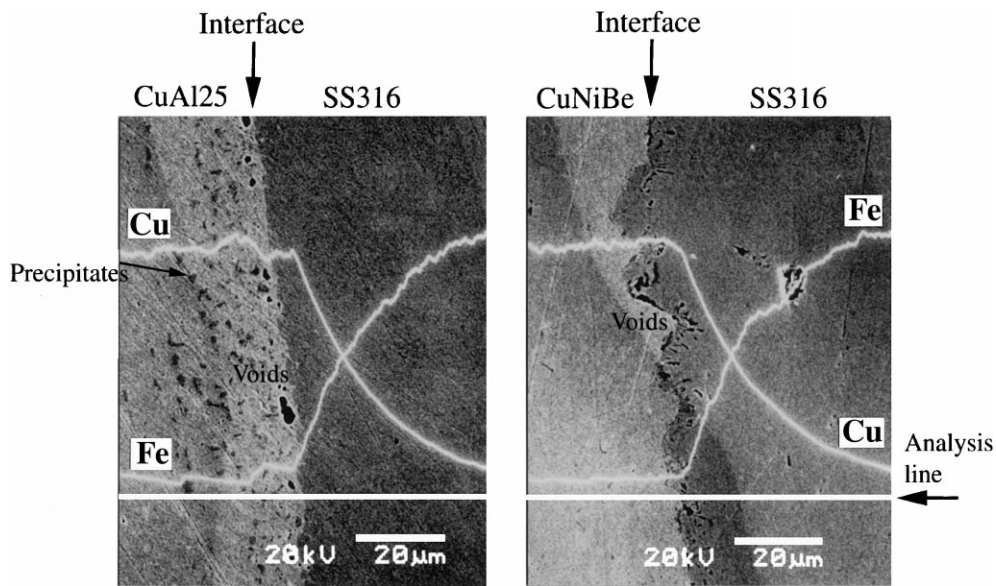


Fig. 1. SEM micrograph of joint materials of CuAl25/SS316L and CuNiBe/SS316L, and typical results of EDX line analyses.

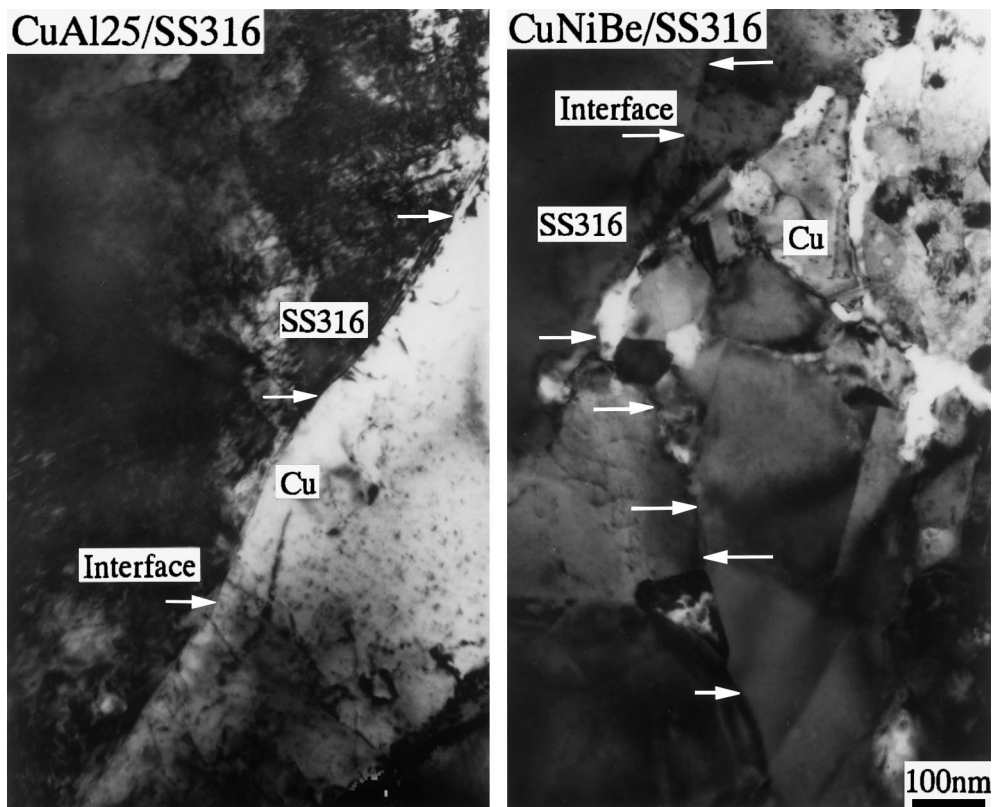


Fig. 2. Microstructure in the interface of as-received joint materials.

specimens of the two types of joints were annealed at 573 and 673 K for 100 h, respectively, and then analyzing TEM specimens were prepared by ion milling. The interfaces were examined using TEM and scanning electron microscopy (SEM, JEOL JSM-5800LV) to characterize the interfacial and the overall microstructures.

### 3. Results

#### 3.1. Microstructures in the interfaces of as-received joints

Fig. 1 shows the SEM micrograph of CuAl25/SS316L and CuNiBe/SS316L joints. Energy dispersive X-ray line analyses were conducted for Fe, Ni, Cr and Cu. Typical results of EDX line analyses (Fe and Cu) are also shown in the figure. It was found that there was interdiffusion of the two base materials. The concentration of Fe, Cr and Ni decreased and that of Cu increased in the region of stainless steel adjacent Cu alloy. Many precipitates, mainly composed of Cr and Fe, were found in the Cu alloy side of interfaces. In the CuAl25/SS316L joints, small voids, about 2  $\mu\text{m}$  in diameter, were

observed in the Cu alloy side near the interface, whereas in the CuNiBe/SS316L joints, voids, about 5  $\mu\text{m}$  in diameter, were observed in the interface, and small voids, about 1  $\mu\text{m}$  in diameter, were observed in the SS316L near the interface.

Fig. 2 shows the TEM micrograph of CuAl25/SS316L and CuNiBe/SS316L interfaces. As in the microstructures observed by SEM, the precipitates were not found at the interface of both joints. Also, voids were only found at the interface of CuNiBe/SS316L, and they were not observed in the Cu matrix and stainless steel far from interface.

#### 3.2. Microstructures in the interfaces of annealed joints

During the isochronal annealing from 298 to 973 K of CuAl25/SS316L joints, no precipitates or voids were formed at the interface. The small Al precipitates and the defect clusters induced by ion milling in the Cu alloy side disappeared. Typical microstructures in the joints before and after the annealing experiment are shown in Fig. 3. Fig. 4 shows that the microstructures at the interfaces of the joint materials were thermally stable even after annealing at 573 or 673 K for 100 h. The

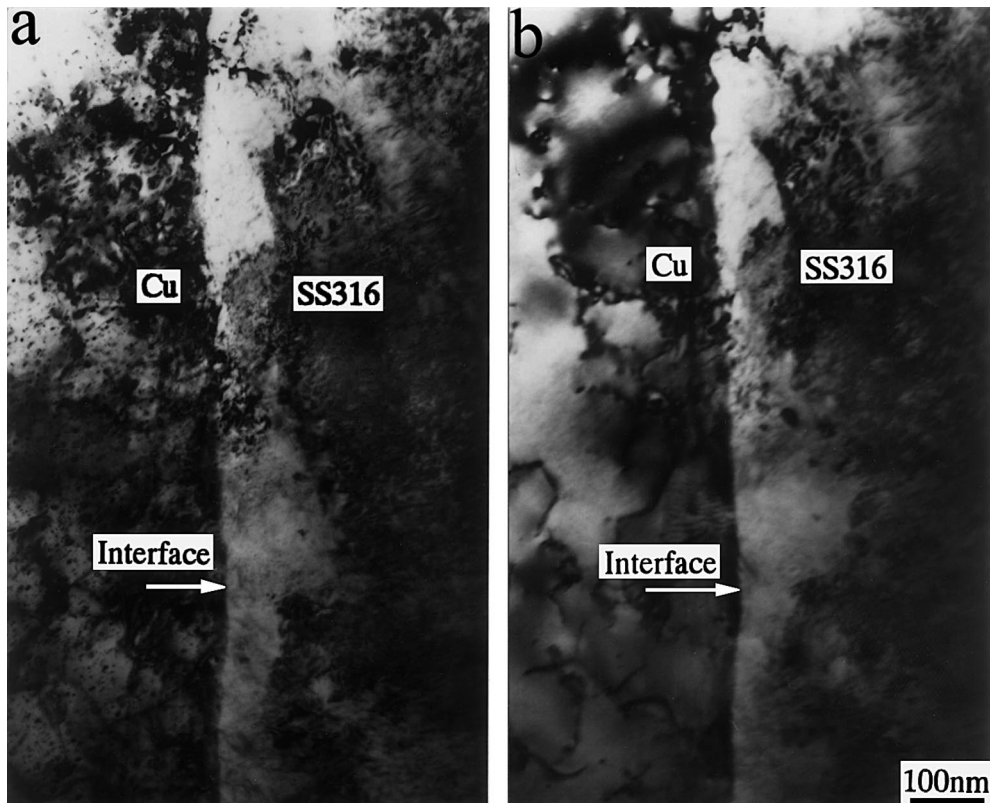


Fig. 3. Typical microstructure in the bonded joint of CuAl25/SS316L before (a) and after (b) the isochronal annealing.

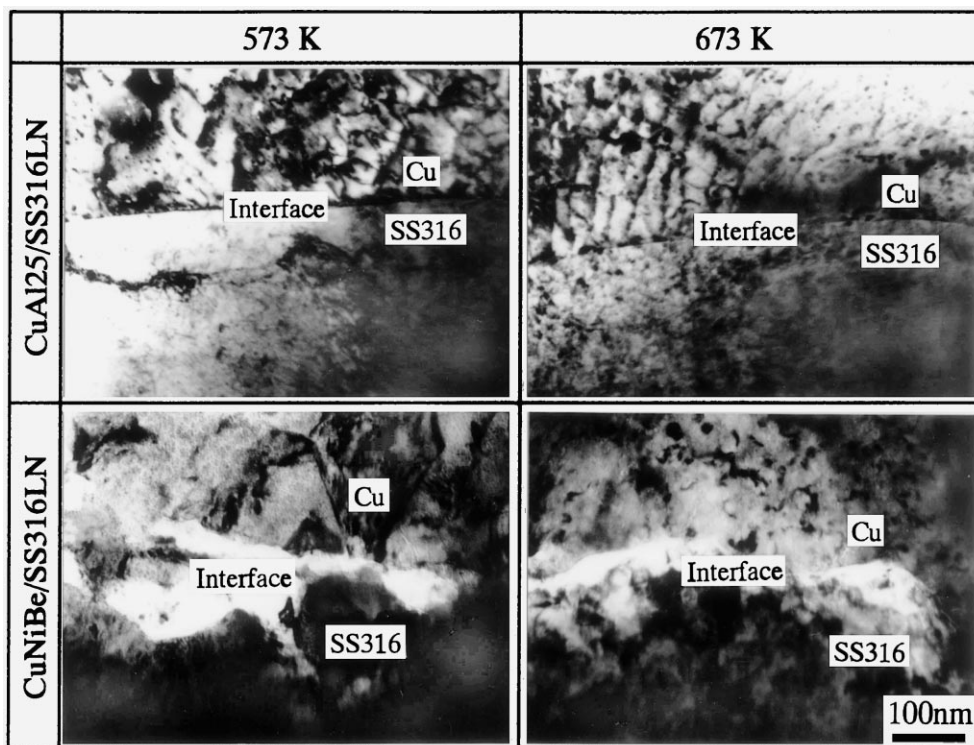


Fig. 4. Microstructure in the interface of joint materials annealed at 573 and 673 K for 100 h.

microstructures in the matrix of both joints and the interdiffusion within joints were also not affected by the annealing.

#### 4. Discussion

Bonded materials of Be (or W), Cu and SS316L have been taken into consideration for use as first wall and divertor materials in the ITER. The joint is the weak part of bonded material. In general, most bonded materials experience failure near the interface [3–7]. Therefore, microstructure changes near the interface would influence the mechanical properties of bonded materials.

In the as-received joints, microporosity, precipitates and interdiffusion were observed near or at the interface. Cu and alloying elements of stainless steel diffused through the interface: concentration of Fe, Cr and Ni decreased, and that of Cu increased in the stainless steel side. Fe and Cr, which are insoluble in Cu, formed the precipitates. The voids and interdiffusion were caused by the HIP process. The formation of voids and precipitates and the composition changes near the interface would directly decrease the static and dynamic properties of materials.

The results of annealing experiments showed that temperatures lower than 673 K did not change the initial microstructure or composition of CuAl25/SS316L and CuNiBe/SS316L joints. Although there are no data from annealing experiments longer than 100 h, it is expected that the microstructure and composition of CuAl25/SS316L and CuNiBe/SS316L are stable under the thermal operating conditions of ITER. However, irradiation may lead to significant changes because of radiation-enhanced segregation, precipitation or dissolution near and at the interface that could alter the properties. In addition, the preexisting voids near the interface of the joints may coarsen under irradiation and enhance the sensitivity of joints to failure. Given the uncertainties in the response to irradiation, neutron irradiation experiments should be performed at appropriate temperatures to investigate the response of the different materials.

#### 5. Conclusion

The microstructure and the composition in the interface of CuAl25/SS316L and CuNiBe/SS316L joints were investigated by TEM and SEM. It was found that voids were formed and that Cu and elements in stainless steel diffused through the interface to the adjacent

material during the HIP process. Also, it was found that the annealing at 573 and 673 K for 100 h did not cause the microstructure to change and interdiffusion in CuAl25/SS316L and CuNiBe/SS316L joints.

#### Acknowledgements

The authors wish to thank Professor Muroga of National Institute for Fusion Science for providing the ion milling instrument to make TEM samples, and Professor Yoshida of Kyushu University for providing TEM to observe the samples. This work was supported by Japan–US collaborative JUPITER program on fusion materials research. Edward's participation was supported by the US Department of Energy under contract DE-AC06-76RLO 1830 with the Battelle Memorial Institute at the Pacific Northwest National Laboratory.

#### References

- [1] R. Aymar, J. Nucl. Mater. 258–263 (1998) 56.
- [2] G. Kalinin, W. Gauster, R. Matera, A.-A.F. Tavassoli, A. Rowcliffe, S. Fabritsiev, H. Kawamura, J. Nucl. Mater. 233–237 (1996) 9.
- [3] J.F. Stubbins, Amanullah, M. James, J. Heuer, P. Kurath, K.D. Leedy, G. Li, G.D. Morgan, K.T. Statterly, B.G. Thomas, G.W. Wille, ITER/US/97/IV-BL-1, Final Report for US ITER Task T212 (CY1996).
- [4] K. Leedy, PhD thesis, Department of Math. Science and Eng., University of Illinois at Urbana-Champaign, July 1997.
- [5] A.F. Rowcliffe, S.J. Zinkle, J.F. Stubbins, D.J. Edwards, D.J. Alexander, J. Nucl. Mater. 258–263 (1998) 183.
- [6] S. Sato, T. Hatano, T. Kuroda, K. Furuya, S. Hara, M. Enoda, H. Takatsu, J. Nucl. Mater. 258–263 (1998) 265.
- [7] S.A. Fabritsiev, A.S. Pokrovsky, D.J. Edwards, S.J. Zinkle, A.F. Rowcliffe, J. Nucl. Mater. 258–263 (1998) 2069.