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Microstructure of neutron irradiated SS316L/DS-Cu joint

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Abstract

To investigate the thermal effects on joint interfaces, hot isostatic pressed (HIPed) and explosively bonded dispersion strengthened copper (GlidCop CuAl25)/316L stainless steel joint samples were irradiated in high flux isotope reactor at 573 K to a dose of about 4.6 dpa. After irradiation, the interface between CuAl25 and SS316L was observed by transmission electron microscopy. The microstructure of neutron-irradiated CuAl25 was strongly affected by the thermal effects during the fabrication. In the HIPed sample, a high density of precipitates composed of Fe (Cr) and voids were observed in coarsened Cu grains. In the explosively bonded sample, on the other hand, the growth of the Cu grains were very limited, which resulted in less void formation in GlidCop CuAl25.

1. Introduction

Hot isostatic pressing (HIP) is considered to be a promising technology to fabricate the international thermonuclear experimental reactor shielding blanket/first wall. The blanket is composed of austenitic stainless steels (SS316L) circular tubes, an oxide dispersion strengthened copper (ODS-Cu) alloy heat sink, and a SS316 backing plate as a structural layer. Characterization of hot isostatic pressed (HIPed) joints has revealed that the mechanical properties (i.e. ultimate tensile strength, yield strength, total elongation, Vickers hardness) of the joints strongly depend on the Hiping parameters (i.e. time, pressure and temperature). But the most important factor is the HIP temperature during the procedure [1–4]. The proposed and optimal temperature for the process is about 1323 K [1] which is very close to the ODS-Cu melting temperature of 1356 K. In this temperature range, an accumulation of alumina might occur, especially in the vicinity of the joint interface. Recovery of dislocation and recrystallization will also occur in the Cu alloys. Different elements (i.e. Cu, Cr, Fe) diffuse across the joint interface and may cause phase transformations (precipitation) and migration of the interface. It is also well known that the microchemical evolution during irradiation strongly affects the microstructure.

In the present study, a HIPed joint of ODS-Cu/SS316L sample was compared to a joint that was produced by explosive bonding. The objective of the present study is to understand the effects of thermal treatments during the fabrication on the neutron irradiated microstructure of the near interface region.

2. Experimental procedure

Two test blocks of HIPed and explosively bonded ODS-Cu (GlidCop CuAl25)/316L stainless steels joint samples were used in this study. The GlidCop CuAl25 alloy was produced by SCM metals and was strengthened by dispersion of 3–12 nm size alumina particles. The details of the joining procedure can be found in Ref. [5]. The average ODS-Cu grain size of the HIPed and explosively bonded sample were 60 and 2 μm, respectively. The samples were thinned to 0.25 mm thick and cut into 3 mm transmission electron microscopy (TEM)

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disks. Neutron irradiation was performed in high flux isotope reactor (HFIR)-11J experiment at 566 ± 19 K to the dose of 4.6 dpa (in stainless steels). The detailed operation history during HFIR-11J experiment is summarized in Ref. [6]. After irradiation, the interface between ODS-Cu and SS316L was observed by scanning electron microscopy (SEM) and TEM.

3. Results

Fig. 1 show SEM images of two joint interfaces. The interface between ODS-Cu and SS316L in an explosively bonded sample was wavy compared to HIPed sample. In both samples, many fine sphere-like precipitates (named as type 1), enriched in Fe and Cr element, were detected in the region close to the interface. In addition, relatively large precipitates (shown by an arrow in Fig. 1(a), type 2) which were also enriched in Fe and Cr were detected in the HIPed samples. The type 2 precipitates formed in HIPed samples, appeared on the ODS-Cu side within a distance of about 100 μm from the interface. This is due to an interdiffusion of the element of stainless steel and copper across the interface [7]. Copper enrichment on the SS316L side is also prominent. An EDS spectrum, which was measured in the dark portion of Fig. 1(b), is shown in Fig. 2 and compared to the matrix. The enriched layer was about 3 μm thick in both joints.

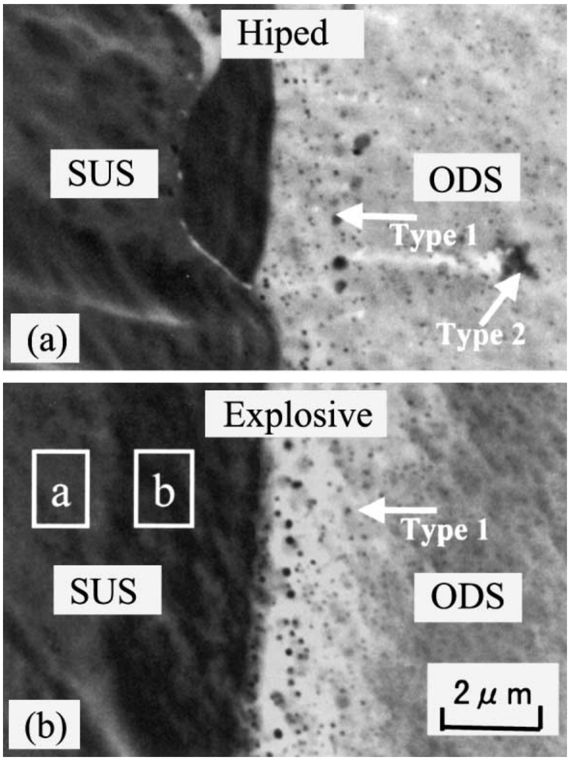

Fig. 1. SEM image of the interface: (a) HIPed sample; (b) explosively joined sample. EDS measurements were conducted in the regions a and b in an explosively joined sample.

Typical microstructure of neutron irradiated ODS-Cu near the interface between ODS-Cu and SS316L is shown in Fig. 3. Fig. 3(a)–(c) show microstructures of the HIPed and the explosively bonded CuAl25 samples, respectively. In the HIPed sample, Fe and Cr enriched

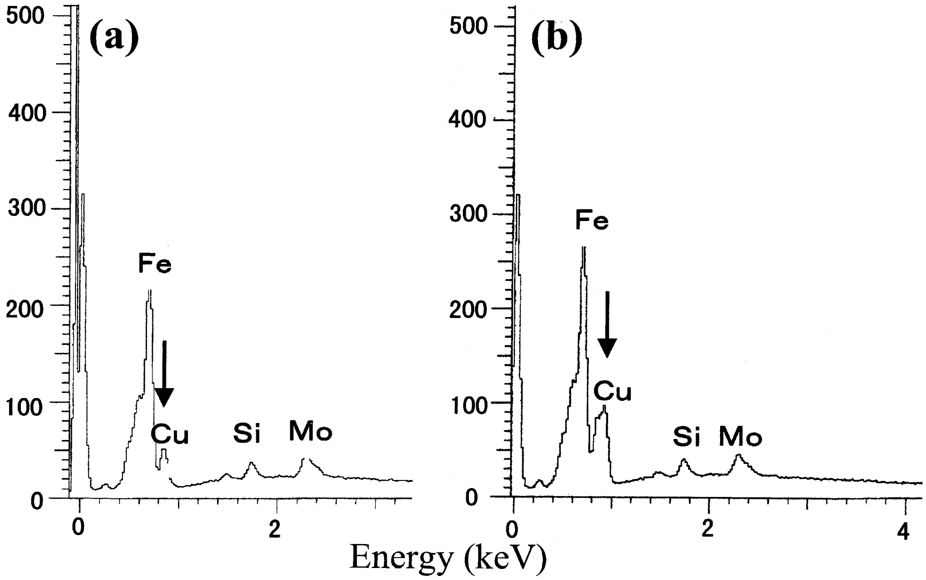

Fig. 2. EDS spectrum: (a) from the matrix and (b) from the copper diffused region.

type 1 precipitates (shown by arrows in Fig. 3(a)) and voids of about 70 nm were observed. A high density of dislocations, which are commonly observed in an unirradiated sample, was not observed. Dislocation structure was only detected in the regions close to voids (shown in Fig. 3(a)). On the other hand, in the explosively bonded sample, dislocation density and void formation depended very much on the distance from the interface. In the region close to the interface (∼1 μm from the interface), as shown in Fig. 3(b), a lower dislocation density and small voids of about 10 nm in diameter were detected. At distances more than 1 μm from the interface, a higher dislocation density and voids of about 35 nm in diameter were observed (Fig. 3(c)).

Fig. 4 shows typical microstructures of annealed (a) and (b) 10% cold worked pure copper irradiated in the same irradiation capsule of the HFIR-11J

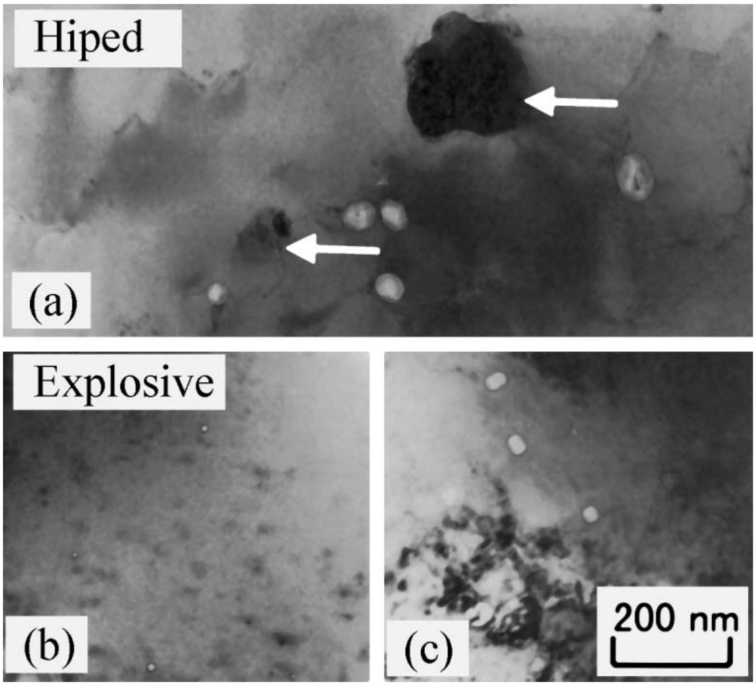

Fig. 3. Microstructure of neutron irradiated ODS-Cu at 573 K to the dose of 4.6 dpa: (a) HIPed sample; (b) near interface region in explosively jointed sample (∼1 μm from the interface); (c) distance more than 1 μm.

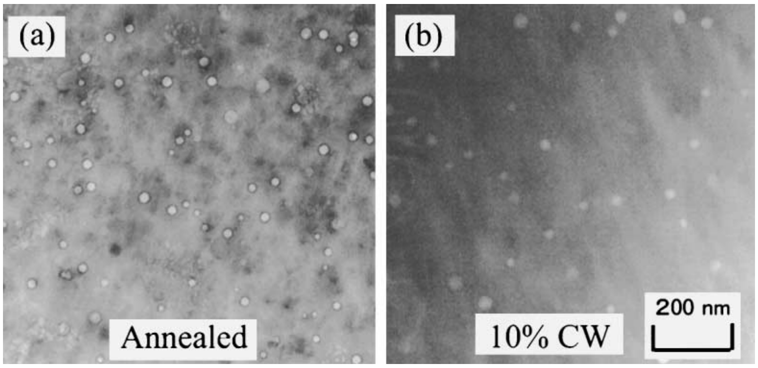

Fig. 4. Microstructure of neutron irradiated pure copper: (a) annealed (1073 K, 2 h, in vacuum); (b) 10% cold worked prior to irradiation.

experiment. Only larger voids were observed in cold worked sample.

4. Discussions

A study of fast neutron irradiation of GlidCop CuAl25 revealed that the alloy exhibits strong void swelling resistance in this temperature range [4]. This swelling resistance is due to very high density of dislocations and homogenously dispersed alumina particles. It is also important that there is no recovery of dislocations and recrystallization during joining procedure and irradiation. But in this experiment, the average ODS-Cu grain size of the HIPed and the explosively bonded samples were 60 and 2 μm, respectively. In addition, a large number of dislocation recovery occurred during fabrication. Cu-ion irradiation on an ODS-Cu sample cut from the HIPed sample used in this study was also performed by Hatakeyama et al. They reported that dislocations were also recovered and void swelling of the sample was close to 3.0% at 673 K and at a dose level of 30 dpa [8].

The optimal HIP temperature for ODS-Cu/SS316L is about 1323 K [1]. This temperature is close to the ODS-Cu melting temperature of 1356 K. This higher HIP temperature is the main reason for the recovery of dislocations and also recrystallization of the ODS-Cu. In the explosively bonded sample, recovery of dislocations was also observed in the region close to the interface. But recrystallization of the sample was very limited, which resulted in less void swelling in this region. As shown in Figs. 3 and 4, the size and density of voids in the region close to the interface and at distances more than 1 μm from the interface are comparable to those of pure copper in annealed and 10% cold worked conditions, respectively. This means that the microstructure is mainly controlled by dislocation density and dispersed alumina particles are not essential in the region close to the interface for suppressing void swelling. The study suggests that the development of lower temperature bonding technique is needed to reduce the void swelling of ODS-Cu alloys in ODS-Cu/SS316L joints.

5. Conclusions

Two test blocks of HIPed and explosive bonding GlidCop CuAl25/SS316L joint were irradiated in the HFIR at ORNL (573 K, 4.6 dpa) and the main results are summarized as follows:

1. The microstructure of ODS-Cu was strongly affected by the thermal effects during the fabrication (bonding) steps.
2. In the HIPed sample, dislocations had recovered, a high density of precipitates composed of Fe (Cr), and voids were observed in coarsened Cu grains.
3. In the explosively bonded sample, on the other hand, dislocation recovery and the growth of Cu grains were very limited which resulted in less void swelling.

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References

[1] S. Sato, T. Kuroda, T. Kurosawa, K. Fukuya, I. Togami, H. Takatsu, J. Nucl. Mater. 940 (1996) 233.

[2] S. Sato, T. Hatano, T. Kuroda, K. Fukuya, S. Hara, M. Enoeda, H. Takatsu, J. Nucl. Mater. 265 (1998) 258.

[3] S. Taehtinen, B.N. Singh, P. Toft, J. Nucl. Mater. 1238 (2000) 283.

[4] D.J. Edwards, J.W. Newkirk, F.A. Garner, M.L. Hamilton, A. Nadkarni, P. Sammal, ASTM ASTP 1175 (1993) 1041.

[5] Y. Aono, private communication.

[6] K.E. Lenox, M.L. Grossbeck, Fusion Materials, Semiannual Progress Report for Period Ending 31 December 1998, OOE/ER-313/25, p. 307.

[7] Q. Xu, D.J. Edwards, T. Yoshiie, J. Nucl. Mater. 1229 (2000) 283.

[8] M. Hatakeyama, H. Watanabe, M. Akiba, N. Yoshida, Mechanism of low void swelling in dispersion strengthened copper alloys of diverter heat sinks, these Proceedings.