Ion-Irradiation Hardening of Brazed Joints of Tungsten and Oxide Dispersion Strengthened (ODS) Ferritic Steel

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Irradiation hardening and microstructural change of the brazed-joint of W and oxide dispersion strengthened ferritic steel (ODS-FS) was investigated by nano-indentation hardness test and transmission electron microscopy after ion irradiation with 6.4 MeV Fe³⁺ ions at 500°C up to 10 dpa. Dual-beam irradiation of Fe³⁺ ions and energy-degraded 1 MeV He⁺ ion was also carried out. A considerable irradiation hardening occurred in the W base metal where dislocation loops and nano-scaled voids or He-bubbles were observed. Dual-beam irradiation enhanced the hardening. No significant hardening was observed in ODS-FS. The hardness of insert material was reduced after irradiation, which is due to the recovery of dislocations generated during joining process.

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1. Introduction

Tungsten (W) coated oxide dispersion strengthened ferritic steel (ODS-FS) is a candidate for the plasma facing component (PFC).¹⁻³ because the combined use of these materials is attractive from the view point that: (i) W has a high melting point, high thermal conductivity, high sputtering resistance and low tritium retention, (ii) ODS-FS has excellent materials performance, such as high-temperature strength,⁴,⁵ void-swelling resistance up to high doses⁶) and corrosion resistance against various coolants.⁷⁻⁹ We have developed a brazed joint of W and ODS-FS using an insert material of Fe–3B–5Si amorphous foil, of which the joint has excellent interfacer shear strength,¹⁰,¹¹ and the assessment of potential for irradiation resistance of the joints is now necessary. As for the irradiation effect on each material, W and ODS-FS, several researches on irradiation embrittlement have been done.⁶,¹²⁻¹⁴ However, the available data of transmutation helium effect on the mechanical properties of W is limited. Moreover, few results of irradiation effect on W/ODS-FS brazed joint exist.

In this study, the ion-irradiation hardening of the W/ODS-FS brazed joint irradiated up to 10 dpa with and without a simultaneous helium implantation up to about 150 appm at a temperature of 500°C was investigated. A dual-ion beam irradiation experiment enables to introduce both the displacement damage and helium into the joint up to a few micrometers in order to investigate the synergistic effects on the hardness distribution across the interface of brazed-joint and microstructural development in the W base metal and the joint interface. This paper mainly presents the results of W base metal.

2. Experimental Procedure

The substrate material is a reduced-activation ODS-FS (Fe–15Cr–2W–0.2Ti–0.35Y₂O₃). The coating material is a hot-rolled and stress relief-annealed W supplied by Plansee Japan Ltd. A commercial amorphous foil (Fe–3B–5Si in mass%, 25 µm thickness, Metglas Co., USA) was selected as an insert material for LPDB. The ODS steel was fabricated by mechanical alloying and hot extrusion process.

Surfaces to be bonded were wet ground with SiC emery paper to grits #2400. After surface grinding, samples were stored in acetone for cleaning the surface contamination. A stack of ODS steel-insert material-W was vertically placed and heated up to 1240°C at a heating rate of 30°C/min for LPDB. The base material pair without insert material is for SSDB. The bonding temperature was held at 1240°C for 30 min in a high vacuum (5 x 10⁻⁴ Pa) under a hydrostatic pressure of 10 MPa with uni-axial compressive loading mode. Bonding temperature was determined based on the results of TG-DTA (Thermogravimetry differential thermal analysis) in our previous work,¹⁰) which is as high as liquidus of the insert material. After the bonding process, compressive load was eliminated and the samples were cooled at a rate of 5°C/min in the furnace. Cross-sectional surface perpendicular to the joint interface was obtained and polished for the following ion-irradiation.

The cross-sectional surface of the joint specimens were ion-irradiated using DuET; a dual-ion beam accelerator facility, with a single-ion beam of 6.4 MeV Fe³⁺ ions or dual-ion beams of 6.4 MeV Fe³⁺ ions with a simultaneous implantation of energy-degraded 1 MeV He⁺ ions. The irradiation temperature was controlled at 500°C within an error of ±10°C by monitoring with infrared thermal vision. The depth profiles of displacement damage and implanted He/Fe ions were calculated for W and ODS-FS by a Monte Carlo simulation package (SRIM code 2008¹⁵) assuming an
average displacement energy of 40 eV for ODS-FS and 90 eV for W,\textsuperscript{16} as shown in Fig. 1. Nominal values of the displacement damage rate, total displacement damage and helium concentration for ODS-FS and insert material were $3 \times 10^{-4}$ dpa/s, 10 dpa and 150 appm, respectively, and those values for W were $4.4 \times 10^{-4}$ dpa/s, 14 dpa and 150 appm, respectively. The “nominal” ones stand for the calculated value at the depth of 600 nm depth.

Nano-indentation hardness was measured by the loading-unloading method with a maximum load of 9.8 mN using the Elionix ENT-1100a with a Berkovich type indentation tip.\textsuperscript{17,18} Indentation size effect (ISE) was also measured by using the Agilent Technologies, Inc. Model Nano Indenter G200 with a Berkovich type indentation tip. The continuous stiffness measurement (CSM) method was carried out. Microstructures in the ion-irradiated W base metal were observed by transmission electron microscopy (TEM). Cross-sectional thin foil specimens for TEM observation were fabricated by a focused ion beam (FIB) device JEOL JIB-4500 and finally electro-polished with a NaOH aqueous solution to remove the damaged and contaminated surface during FIB processing. Dislocation densities in insert materials were measured on TEM micrographs by a line-intersection method.\textsuperscript{19} Foil thicknesses were determined by the convergent beam electron diffraction method. The dislocation density in insert material was measured by exciting the reciprocal vector of $g = 002$, a single vector that made all the perfect dislocations in the bcc structure visible.

3. Results and Discussion

Figure 2 shows a typical cross-sectional image of the brazed joint obtained by scanning electron microscopy (SEM). As previously discussed for a similar ODS-FS,\textsuperscript{11} the brazed joint must have contained five characteristic regions: W base metal, an interdiffusion layer between insert material and W, insert material, a diffusion-affected zone of the ODS-FS, and ODS-FS base metal. In Fig. 2, however, a diffusion-affected zone of the ODS-FS was not clearly observed because it was difficult to distinguish a diffusion-affected zone of the ODS-FS only by SEM image.

Figure 3 shows nano-indentation hardness ($H$) distributions on the irradiated surface of the brazed joint before and after ion-irradiations with and without simultaneous implantation of helium. No cracking was observed even in the W-base metal and at around the brazing interface. After irradiation, the hardness of the insert material appeared to be reduced. A considerable irradiation hardening occurred in the W base metal, while no significant irradiation hardening was observed in ODS-FS as expected. The relatively large irradiation hardening in W at 500°C is a different feature from those of iron and steels.

Recently, it is shown that nano-indentation hardness depends on the selected indentation depth for the constant load measurement that is known as indentation size effect (ISE).\textsuperscript{20} Since the indentation depth was selected as 240 nm for the measurement of as-received W while that of dual-ion irradiated ones was selected as 200 nm, the hardness might be
influenced by ISE. So the indentation depth profile of the hardness was measured, and shown in Fig. 4 for unirradiated W. The difference in the hardness between the indentation depths of 200 and 240 nm is merely 0.5 GPa that is smaller enough to compare the difference between as received W and irradiated W (almost 1.5 GPa for single-ion irradiation and 2.5 GPa for dual-ion irradiation). It is concluded that the irradiation hardening occurred in W after single- and dual-ion irradiations, while the ODS-FS shows almost no change in the nano-indentation hardness after both single- and dual-ion irradiations, as shown in Fig. 3.

In the insert material, a small softening was observed after both single and dual ion-irradiations, as shown in Fig. 3. In order to clarify the causes of the softening, a W/ODS-FS brazed-joint was annealed at the same temperature as the irradiation temperature of 500°C. Figure 5 shows the hardness distribution across the joint before and after annealing at 500°C for 6 h, indicating a small softening is induced by the annealing. Figure 6 shows TEM micrographs of single-ion irradiated insert material. Dislocations in high density were observed but few irradiation defects such as dislocation loops and voids were observed in insert material. Dislocation density before and after irradiation was 6.6 \times 10^{16} and 4.2 \times 10^{16} m^{-2}, respectively. It is considered that the softening in the insert material was due to the annealing out of dislocations during ion-irradiation at 500°C that was similar to the behavior observed in the annealing experiment at 500°C. Moreover, the dislocations in high density play a role as sinks of irradiation defects and contribute to the suppression of formation of irradiation defects.

As for the defect structures in W, TEM observations revealed that irradiation defects, such as dislocation loops, voids and cavities, were formed by ion-irradiations (Fig. 7). White dots in weak beam images, Figs. 7(a) and 7(d), shows dislocation loops. Voids and cavities appear in over and under focus images, Figs. 7(b), 7(c), 7(e), 7(f). Numerical data of mean diameter and number density of loops, voids and cavities are shown in Table 1. The size and number density of dislocation loops in dual-ion irradiated W is larger than that in single-ion irradiated one. On the other hand, the size of voids and cavities in dual-ion irradiated W is smaller than that in single-ion irradiated one, although the number density in dual-ion irradiated W is higher than that in single-ion irradiated one. These irradiation defects are probably responsible for the irradiation hardening. Orowan type equation can describe the hardness change due to irradiation defects, such as dislocation loops, voids and cavities, as indicated in the following equations;

\[
\Delta \sigma_v = M \Delta \tau = M \sqrt{\Delta \tau_1^2 + \Delta \tau_2^2} \quad (1)
\]

\[
\Delta \tau_1 = \alpha \mu b \sqrt{\frac{N_{d_1}}{d_1}} \quad (2)
\]

\[
\Delta \tau_c = \frac{\alpha c \mu b \sqrt{N_c d_c}}{1 - 0.81d_c \sqrt{N_c d_c}} \ln \left\{ \frac{0.81d_c}{3.3b} (1 - 0.81d_c \sqrt{N_c d_c}) \right\} \quad (3)
\]
where, $\Delta \sigma_y$ represents the increase in yield stress due to obstacles, $M$ is Taylor factor, $\Delta \tau_l$ and $\Delta \tau_c$ represents the increase in shear stress due to loops and cavities, respectively. $\Delta \tau_l$ is described by average size and number density of loops $d_l$, $N_l$, strength factor $\alpha_l$, absolute value of Burgers vector $b$, and shear modulus $\mu$. $\Delta \tau_c$ represents increase in the shear stress due to cavities of size $d_c$, number density $N_c$, and strength factor $\alpha_c$. Stoller and Zinkle have shown that $M$ is actually an upper limit for the ratio of uniaxial yield stress to resolved shear stress and has the value of 3.06 for both FCC and BCC metals. Burgers vector $b$ and shear modulus $\mu$ for W are $2.74 \times 10^{-10}$ m and $1.61 \times 10^{-11}$ Pa, respectively.

Increasing in the yield stresses of dual-ion irradiated W ($\Delta \sigma_{y,\text{dual}}$) and single-ion irradiated W ($\Delta \sigma_{y,\text{single}}$) were calculated from the nano-indentation hardness by the empirical rule: $VHN = 3 \sigma_y$, and the reduction formula between $VHN$ and nano-indentation hardness $H$: $VHN = 92.59 \times H$ [GPa]. The reduction values were 373 MPa for $\Delta \sigma_{y,\text{single}}$ and 630 MPa for $\Delta \sigma_{y,\text{dual}}$, respectively. The strength factor $\alpha_l$ and $\alpha_c$ were calculated using eqs. (1), (2) and (3), and the values were 0.22 for $\alpha_l$ and 0.12 for $\alpha_c$, respectively. The research on the strength factor of dislocation loops and voids in W was limited. On the other hand, MD simulation on the interaction between point defect clusters and dislocation showed that small clusters, of which the size was rather small like 1 nm, behaved as strong obstacles. They suggested that the resistance of point defect clusters such as dislocation loops and voids increases with their size. For example, the critical stress of dislocation loop of which size was about 4 nm was about 200–300 MPa at 27°C in BCC Fe. As tungsten is also BCC metal, the results may not be much different. So the strength factors estimated in this research is considered to be appropriate.

The ODS-FS shows no meaningful change in nano-indentation hardness after both single and dual beam irradiations. Although chemical compositions are different from the present ODS-FS, it is worthwhile to point out that the K3 ODS-FS (16Cr–2W–0.3Ti–4.6Al–0.4Y2O3) showed no significant changes in the microstructure up to 20 dpa at 500°C.

4. Conclusions

Ion-irradiation hardening of W/ODS-FS brazed-joint was investigated by single-ion (Fe3⁺) and dual-ion (Fe3⁺ + He⁺) irradiation experiments at 500°C up to 10 dpa.

(1) In W, irradiation hardening was observed after both single- and dual-ion irradiations. The dual-iron irradiation hardening was much larger than single-iron irradiation hardening, indicating that helium implantation increased the irradiation hardening of W.

(2) Irradiation hardening is due to the formation of defects, interstitial dislocation loops and vacancy clusters. The strength factor of loops and voids were estimated to be 0.22 and 0.12, respectively, based on Orowan-type hardening mechanism.

(3) No significant change in the nano-indentation hardness was observed in ODS-FS.

(4) Small softening was observed in the insert material after irradiations. The softening is considered to be due to the recovery of dislocations generated during bonding process.
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