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# The microstructural evolution of precipitate strengthened copper alloys by varying temperature irradiation

Y. Sumino [a,*], H. Watanabe [b], N. Yoshida [b]

[a] Interdisciplinary Graduate School of Engineering Sciences, Kyushu University, 6-1 Kasuga-koen, Kasuga, Fukuoka 816-8580, Japan
[b] Research Institute for Applied Mechanics, Kyushu University, 6-1 Kasuga-koen, Kasuga, Fukuoka 816-8580, Japan

ABSTRACT

To investigate the effect of varying temperature irradiation on oxide dispersion strengthened copper alloy (GlidCop CuAl15), heavy ion irradiation was carried out by using 2.4 MeV Cu ions up to 30 dpa in the temperature range of 473–673 K. TEM observation of CuAl15 before irradiation showed high density of small alumina particles and dislocations. After 30 dpa at 673 K constant temperature irradiation, voids were formed. However, voids formation was strongly depended on grain size. On the other hand, by a varying temperature irradiation, the number density of voids was decreased as compared with that of 673 K constant temperature irradiation. The result of CuAl15 is consistent with our previous studies of pure copper irradiated under same irradiation conditions.



## 1. Introduction

Oxide dispersion strengthened (ODS) copper alloys have been suggested for fusion applications as diverter heat sink materials since they have high thermal conductivity and high strength [1–3]. Many studies have been conducted to understand the materials properties of ODS copper alloys under constant irradiation temperature [4–10]. The results of these experiments showed that dislocation loop nucleation of the materials was fundamentally almost same as that of pure copper, namely high density of dislocation loops was formed below 473 K [6,7,10]. However, voids formation of ODS copper alloys is different with pure copper and void swelling of ODS copper alloys was very low [9].

While, a few irradiation experiments were reported under varying temperature irradiation, which can be important for ITER. Our previous studies on Fe–Cr–Ni austenitic stainless steels under fission neutron irradiation using JMTR (Japan Materials Testing Reactor) [11,12] and ion irradiation [13,14] showed that, when variation of temperature crossed a characteristic border temperature of microstructure evolution (573–623 K for Fe–Cr–Ni alloys), the pre-irradiation at lower temperature is very efficient for suppression of interstitial loop formation. The results were explained by the vacancy rich conditions, which appears temporarily at the beginning of the high temperature irradiation. In the case of pure cooper and its alloys, on the other hand, a large number of small defects clusters is formed below 573 K, but only void is formed in the temperature higher than 633 K. Our previous studies on pure cooper revealed that, in comparison with constant irradiation at 673 K, void formation at 673 K was promoted by pre-irradiation at 473 K. void formation and void swelling were suppressed by increasing number of the temperature variation cycles [15]. The objective of the present study is to reveal the effects of varying temperature on the change of microstructure of ODS copper alloy and to understand the mechanisms of defect clustering procedure during varying temperature irradiation under ion irradiation.

## 2. Experimental procedure

ODS copper alloy GlidCop CuAl15 produced by SCM Metal Products was used in this study. The material contains 0.15 wt% Al in the form of $Al_2O_3$ particles. After sliced a sheet of about 0.15 mm thickness from ingot, 3 mm diameter TEM discs were prepared for irradiation. To remove surface contamination (namely oxides) before irradiation, they were electro-polished. Samples were irradiated up to 30 dpa by 2.4 MeV $Cu^{2+}$ ions at constant temperature of 473 K and 673 K. In addition to the constant temperature irradiation, periodic temperature irradiations were carried out between 473 K and 673 K (see Fig. 1). Specimens were irradiated at 473 K up to dose of 0.75 dpa, and then continuous irradiation was performed at 673 K up to 6.75 dpa. This process was repeated four times, namely samples experienced irradiation at 473 K during the 10% of the designed dose (namely 3 dpa) and at 673 K during the remaining (namely 27 dpa). After the periodic temperature irradiation, all samples were thinned in the near damage peak from the backside by back-thinning method. And, the microstructures were observed by transmission electron microscope.

* Corresponding author.
E-mail address: watanabe@riam.kyushu-u.ac.jp (Y. Sumino).

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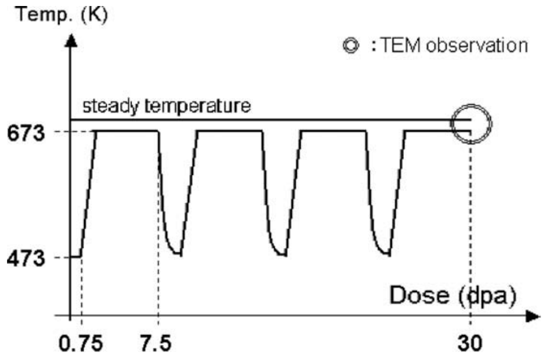


**Fig. 1.** Schematic view of 4 cycles temperature variation.

## 3. Results and discussion

### *3.1. Microstructure before and after the constant temperature irradiation*

Figs. 2 and 3 show the dark field images and corresponding void contrast images of the samples after the irradiation, respectively. In the figures, the images of unirradiated samples and the microstructure of varying temperature irradiation are also inserted for comparison. As shown in Fig. 2, before the irradiation, high density of small alumina particles is homogeneously dispersed. The number density of the particles was $5.2 \times 10^{22}/m^3$. The size of dispersed particles was ranged from 1 nm to 5 nm and the mean size of particles was 2.1 nm. In comparison with that of pure copper, the grain of CuAl15 was very fine which was approximately 800 nm and the sub-size grains contained high density of dislocations were observed.

After the irradiation at 473 K, in addition to high density of alumina particles, many small defect clusters such as dislocation loops and SFT were formed. The total number density of dislocation loops and SFT at 473 K were $1.7 \times 10^{23}/m^3$ and $3.0 \times 10^{21}/m^3$, respectively. At 673 K, on the other hand, the density of dislocation loops was remarkably reduced in comparison with 473 K irradiation.

In Fig. 4, the number density of dislocation loops and SFT measured by TEM observation are summarized. Our previous results of pure copper are also shown in the figure. The total number of small defects clusters (namely vacancy and interstitial type dislocation loops) in CuAl15 irradiated under constant temperature was almost same as that of pure copper irradiated by same irradiation conditions. It is revealed that the number density of dislocation loops decreased with increasing temperature as well as pure copper. The density of these defects at 673 K was about one order lower than that at 473 K. This result was consistent with the previous studies [8]. In the case of pure copper, number density of SFT is relatively higher than that of CuAl15. The number density of SFT in pure copper decreased with increasing irradiation temperature. On the other hand, the number density of SFT in CuAl15 was not changed drastically by increasing irradiation temperature.

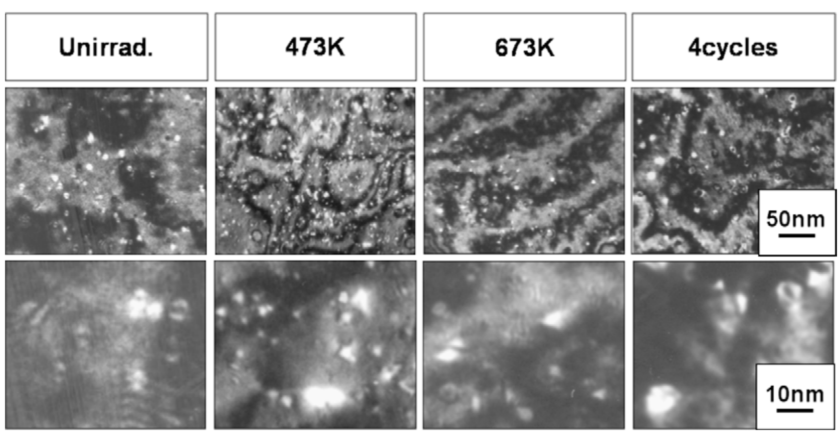


**Fig. 2.** Formation of the small defect cluster in GridCop CuAl15 during constant temperature and periodic temperature variation.

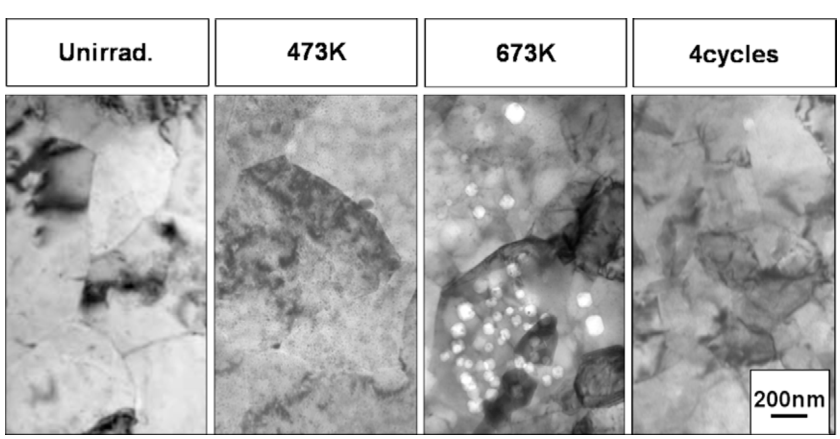


**Fig. 3.** Vold contrast images of the microstructure during constant temperature and periodic temperature variation.

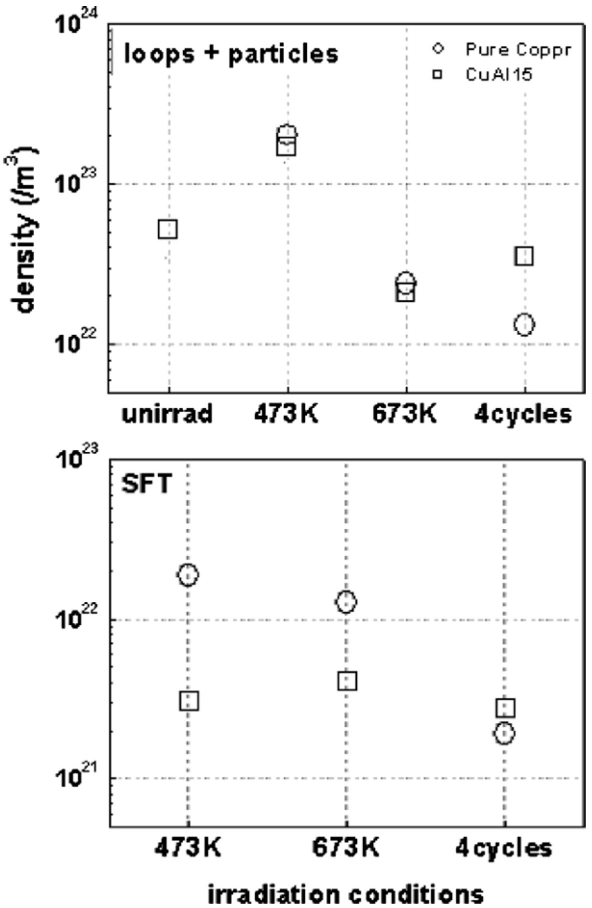


**Fig. 4.** Measured number density of small cluster and SFT.

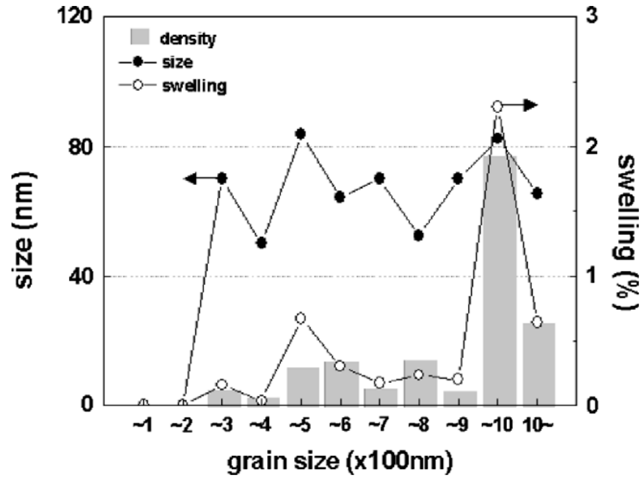


**Fig. 6.** Dependence of grain size on number density, size and void swelling in CuAl15 irradiated at 673 k to 30 dpa.

As shown in Fig. 3, void formation was only observed at 673 K in CuAl15. Lower magnification images of the sample irradiated up to 30 dpa were shown in Fig. 5. Void formation of CuAl15 was strongly depends on grain size. Measured void size and swelling of each grain are summarized in Fig. 6. It's found that the number density of void was higher at grain size more than 1 μm. The void number density and void swelling formed in the grains with 1 μm were $1.3 \times 10^{20}/m^3$ and 2.3%, respectively. However, only a few voids were formed in very fine grain of about 300 nm. These results showed that grain boundaries of CuAl15 act as a strong defect sinks for vacancies. And, therefore, void swelling was suppressed.

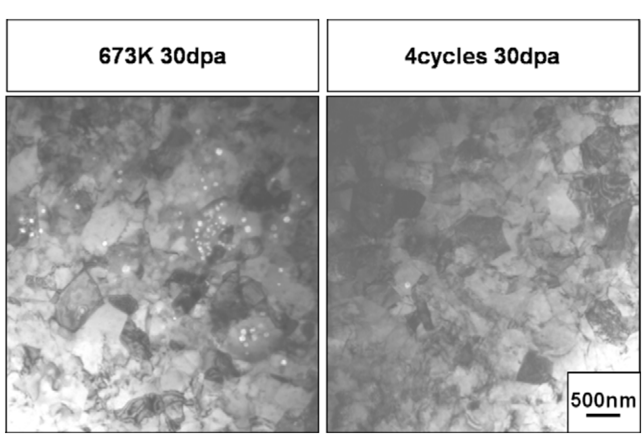


**Fig. 5.** Comparison of the vold images in GridCop CuAl15 irradiated at 673 K constant temperature (left) and periodic temperature variation (right).

### 3.2. Microstructure after varying temperature irradiation

The number density of dislocation and SFT in CuAl15 were not affected by the varying temperature irradiation. As shown in Fig. 3, on the other hand, void swelling of CuAl15 was completely suppressed by the varying temperature irradiation. Suppressed void swelling by varying temperature irradiation is consistent with our previous studies on pure copper [15] In the case of pure copper, small size of voids was formed at 673 K, however, they recombined with interstitials during the successive irradiation at 473 K and they disappeared. By repeating these processes, voids formation was suppressed. In the case of CuAl15, the role of high density of $Al_2O_3$ particles and grain boundary are also important for suppressed void swelling, because they act as a defect sinks for vacancies. By these reasons, void swelling in CuAl15 was completely suppressed.

## 4. Conclusions

To investigate the effects of varying temperature irradiation on the microstructural evolution of ODS copper alloy CuAl15, heavy ions irradiation was performed. The main results are summarized as follows:

(1) In comparison with pure copper, the SFT formation in CuAl15 is strongly suppressed during the constant temperature irradiation at 473 K and 673 K. But number density of SFT and dislocation loops are not affected by the varying temperature irradiation.
(2) The number density of voids after the constant temperature irradiation at 673 K strongly depended on grain size.
(3) By the varying temperature irradiation, voids formation was significantly suppressed at 673 K. This was consistent with previous studies on pure copper. The suppressed void swelling by varying temperature irradiation is explained by the recombination with interstitials during 473 K irradiation and the disappearance of vacancies at neutral sinks such as grain boundary and $Al_2O_3$ particles.

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