Types of Damages in Fission-Neutron Irradiated Cu and Cu Dilute Alloys at 200°C

著者	Yamakawa Kohji, Kojima Atsushi, Shimomura Yoshiharu
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Types of Damages in Fission-Neutron Irradiated Cu and Cu Dilute Alloys at 200°C

Kohji Yamakawa*, Atsushi Kojima* and Yoshiharu Shimomura*

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Synopsis

The damage structures in Cu and Cu based alloys neutron-irradiated at 200 °C were examined by transmission electron microscopy. Large dislocation loops of complex structure and small dot defects were formed in the specimens. The large dislocation loops were interstitial type. On the other hand, the small dot defects were vacancy type. They were stacking faulted tetrahedra.

The damage structure in these metals evolves as follows. Interstitial atoms produced by neutron irradiation aggregate and grow to large complicated loops which will become finally dislocation lines by intersection of them with further irradiation, by absorbing interstitials subsequently produced. By the consumption of the interstitials to the sinks, vacancy concentration increases locally to be high enough to form vacancy defects.

I. Introduction

To clarify fundamental mechanisms of the damage evolution in irradiated metals, the determination of the kind of the defect clusters induced is important.

It has been reported that various types of defects are formed in neutron irradiated copper and copper alloys following the irradiation conditions(1-5). They were dislocations, dislocation loops, small

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* Applied Physics and Chemistry, Faculty of Engineering,
Hiroshima University, Kagamiyama, Higashi-Hiroshima 724

dots(so called black dots), stacking fault tetrahedra(SFT) and voids. At low temperature irradiations, stacking fault tetrahedra were predominantly formed as vacancy type defect. On the other hand, the voids rather than SFT were formed at high temperature irradiations. In many cases, the authors claimed that the defect clusters with small triangular shape were small SFT. However, vacancy type clusters in pure Cu were also able to form triangular loops(TL)(6). For small clusters we can not easily decide by the shape observed from one direction whether these are SFT or TL.

In this study, the kind of the defect clusters in pure Cu and Cu alloys which were fission-neutron irradiated at 200°C, were examined by transmission electron microscopy.

II. Experimental Procedure

Pure Cu foils used in this study were supplied by Cominco Inc., nominal purity of which was 99.9999% pure. Cu alloys were made by argon arc melting with electrolytic pure Cu(99.998% pure) and Ni(99.99% pure) or Mn(99.99% pure). Specimens of pure Cu and the alloys were punched out from the foils of 50-100 μm thickness to the disks and were annealed in high vacuum. Annealed bulk specimens were irradiated in JMTR, Japan Material Test Reactor with the fluence of 2.5×10^{23} n/m^2 at 200°C. After the irradiation the specimens were electro-polished and observed at room temperature by transmission electron microscope. Experimental details are reported elsewhere(7).

III. Experimental Results and Discussion

In Fig.1, the damage structures in pure Cu, Cu-5at.%Ni and Cu-5at.%Mn are shown with bright field image. There are two types of the images; large dislocation loops and small dot defects scattered between the loops. Many loops are decorated by some small loops and have complex shapes. The small dot defects are more clearly shown in Fig.2 with a weak beam image. The size of large dislocation loops in pure Cu were considerably larger than those in Cu-5%Mn specimen but not so different in Cu-5%Ni specimen.

The nature of large dislocation loops in pure Cu and Cu-5%Ni were determined by the inside-outside method. In Fig.3, the large dislocation loops are shown for presented diffraction vector g with deviation parameter s<0 for dark field image. In Fig.3(a), the images of the loops, A-E are smaller than the image of corresponding each loop, respectively, in Fig.3(b), in which the vector g is reversed it in Fig.3(a). The planes of these loops were determined by size change of the images by rotation of the specimen. Thus, these loops were the interstitial type. For Cu-5%Mn, the nature of the loops could not be decided by this method because of the smallness of the defects. The defect clusters which were resolvable as the loops would be interstitial type similar to the case in Cu-5%Ni alloy specimens. Large interstitial loops were also formed in neutron irradiated pure Ni and Ni alloys and with further irradiation the loops grew and finally became dislocation line segments. Therefore, these loops in pure Cu and Cu alloys would become dislocation lines with further irradiations. The size of the small dot defects decreased by the addition of alloying element.

In pure Cu, comparatively larger defects in the small dot defects were resolvable as the defect clusters with triangular shape, when they were observed from <110> direction at thinner part of the specimen. The large dislocation loops escaped out in this thinner part from the specimen by the intersection with the surface. To decide whether the small dot defects were SFT or TL, the defect clusters in the same area were observed from near <100> and <110> and shown in Fig.4. The defects show and square image from <100> and triangle image from <110>. Detail inspection of the image showed that very good agreement exists between expected images from SFT and the observed images from observation direction (near <100> and <110>) are shown in under part of Fig.4, respectively. Two types of the image as trapezoid having opposite direction are observed at B and D in Fig.4 for <100>. The edges of the image were not perfectly parallel to <001> depending on the deviation from <100> axis. Direction of [$0\bar{1}0$] perfectly agree with basal edge of the trapezoid because the specimen were rotated with <001> axis. And also two types of the image(triangle) with opposite direction are observed at B and D for <110>. One of the edges of the image is perfectly parallel to [$\bar{1}10$] and other two edges of the image slightly deviated from [$1\bar{1}\bar{2}$] or [$\bar{1}1\bar{2}$] for deviation from <110> axis by rotation with <001> axis. This shows that in pure Cu, majority of the small dot defects were tetrahedra which were vacancy type defect. Small loops hardly observed among the tetrahedra(dot defects). The dot defects in Cu alloys would also be vacancy type, probably stacking fault tetrahedra. In Cu alloys the dot defects were a little bit smaller than those in pure Cu and had a higher number density. Therefore, shape of the defect images were not clear until now. In Cu and Cu alloys, the formation of large interstitial loops decreased at very near grain boundaries but the formation of small dot defects was scarcely affected by the boundaries. This fact also suggests that these two defects are different in nature.

Number of both interstitial atoms and vacancies which composed these defects were estimated from the number density and size of the these defects, respectively. The number of vacancies were considerably fewer than the number of interstitial atoms. This suggests that a lot of small vacancy clusters(probably SFT) which are unobservably small by electron microscope, are formed in this irradiation temperature, 200°C.

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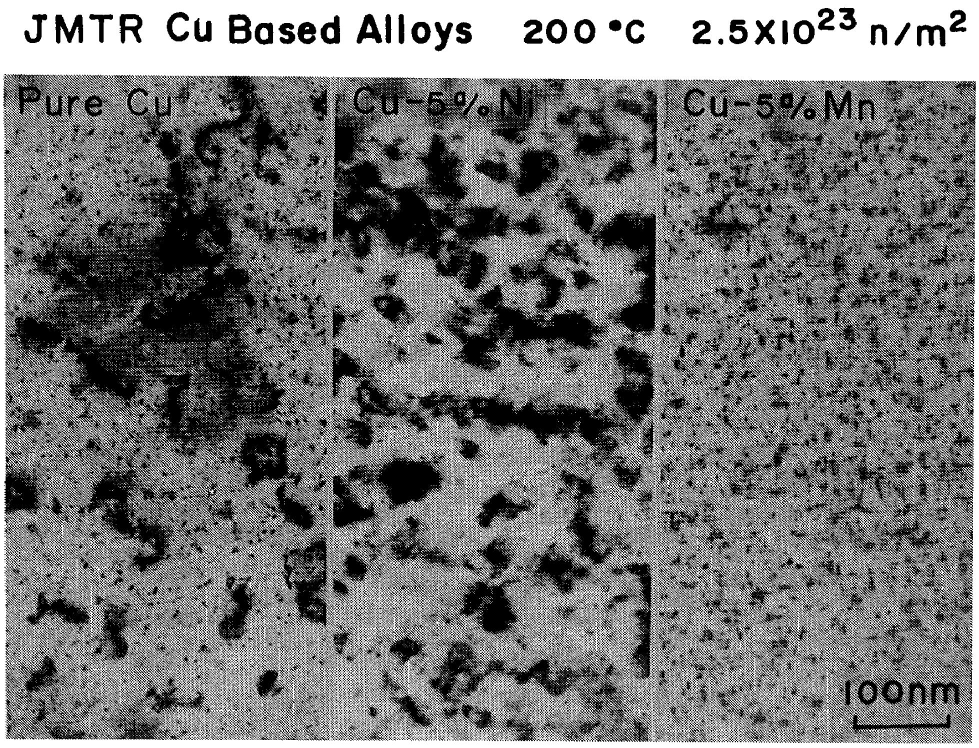

Fig.1 Damage structures of pure Cu, Cu-5at.%Ni and Cu-5at.%Mn which were neutron-irradiated at 200°C with $2.5x10^{23} n/m^2$. Large dislocation loops are clearly shown.

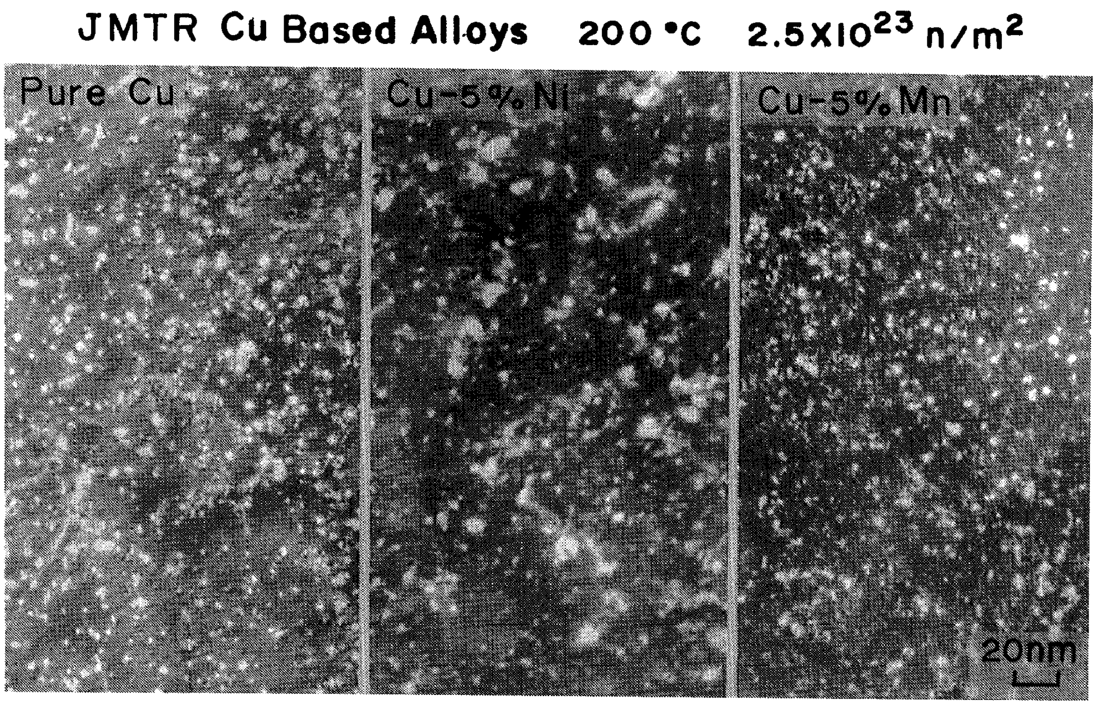

Fig.2 Damage structures of pure Cu, Cu-5at.%Ni and Cu-5at.%Mn are shown with weak beam images. High density of small dot defects are observed.

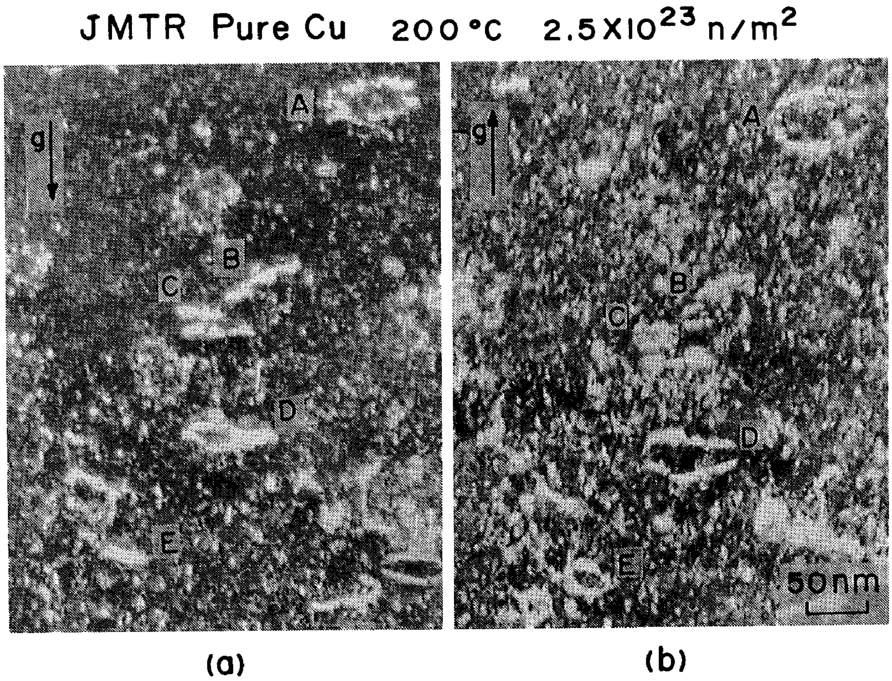

Fig.3 The images of large dislocation loops for the reverse diffraction vector with s<0 for dark field in pure Cu.

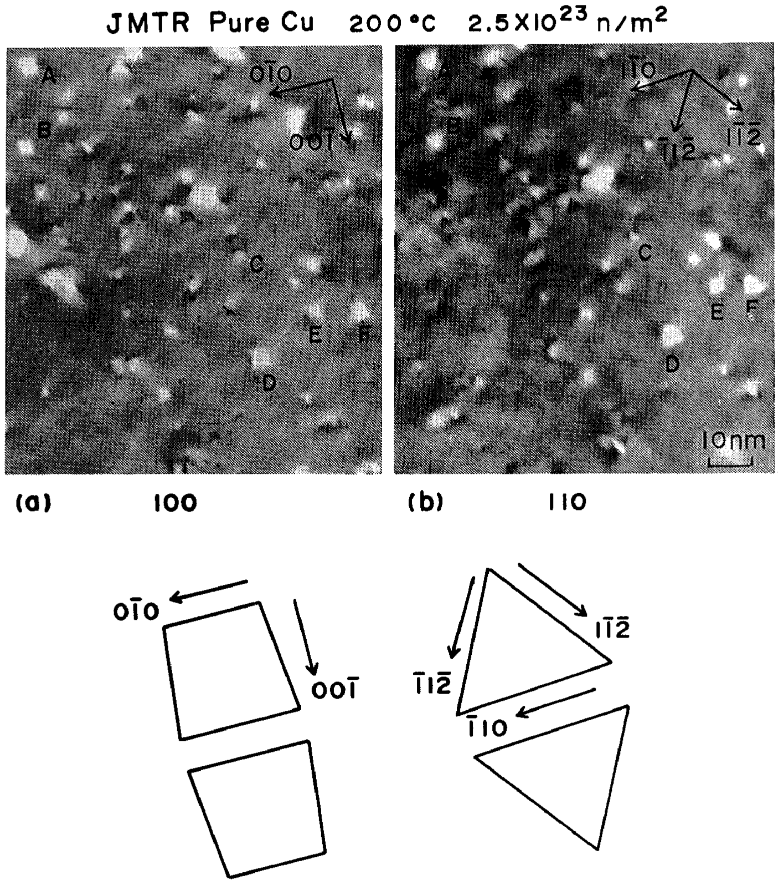

Fig.4 The small dot defects observed from two different directions, near <110> or <100>. The same marks in (a) and (b) show the image of the corresponding defect.