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Point Defects Observed in D-T neutron irradiated Copper, Silver and Gold at 288 K with a Rotating Target in FNS_JAERI

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ABSTRACT

A 14 MeV D-T(fusion) neutron irradiation was carried out at fusion neutron source facility (FNS) in Japan Atomic Energy Research Institute (JAERI). Specimen temperature was controlled to 288 K. Fluence was 6.1×10^{17} to 1.1×10^{21} n/m². Both TEM thin foil and bulk specimens were irradiated at the same position. At 10^{18} n/m², defects observed were single isolated dot defects. With increasing fluence, dot defects changed to complicate structure and made groupings. In a dot group, interstitial clusters and vacancy clusters were observed together. The present result was explained by the modeling that point defects in a nascent damage cascade move in crystal at 288 K and form their defect groupings.

INTRODUCTION

D-T neutron irradiations were performed at RTNS-II in LLNL previously [1,5]. In fcc metals such as Au, Ag and Cu, an average PKA energy which form a damage cascade are 100 keV, 200 keV and 400 keV, respectively [6]. In these fcc specimens which were irradiated below 20 K, only interstitial clusters were observed at 20 K with a cryotransfer technique to an electron microscope [7]. A group of stacking fault tetrahedra (sft) was observed in irradiated specimens at room temperature [8]. The formation of groupings of sft was observed in D-T neutron irradiated Au at room temperature to the range of 10^{22} n/m² at RTNS-II. Such groups were explained previously to be formed by nucleation of vacancy clusters at vacancy core of sub-cascade [9].

The present experiment was carried out to be a complementary experiment to the previous RTNS-II experiment to cover the low fluence regime of irradiation.

EXPERIMENTAL PROCEDURES

Irradiated specimens were Au, Ag and Cu disks of 50 μ m thickness and 3mm in diameter. They loaded in an irradiation chamber which was temperature controlled to 288 K during an irradiation [10]. Inside a chamber, vacuum was evacuated to 10⁶ Pa by a turbo-molecular pump. The irradiation was carried out for 10 hours. To measure the neutron fluence accurately, we loaded many dosimetry foils of Nb. After an irradiation, an activity was measure to determine the neutron fluence at each specimen position. D-T neutron irradiation was carried at FNS in JAERI. After cooling down of radioactivity, specimens were observed by electron microscopy. Defects were observed from the [110] direction with g = [002] under a (g, 5g) diffraction

EXPERIMENTAL RESULTS

<u>Gold</u>

Figs. 1(a), (b) and (c) show the defects which were observed in D-T neutron irradiated gold to 2.6 x 10^{18} , 2.6 x 10^{19} and 3.2 x 10^{20} n/m², respectively. An isolated defects as seen in Fig. 1(a) is an interstitial cluster. In the present work, the nature of defects were determined by TEM annealing experiments. When TEM specimens were annealed at 573 K for 10 min, vacancy cluster grew to sft. Disappeared defects were taken as an interstitial cluster. An interstitial cluster disappears by absorbing vacancies. Vacancy clusters in Au irradiated to 2.6 x 10^{18} n/m² were not observed. This is due to the fact that their structure did not relax to TEM visible ones. In Figs. 1(b) and (c), some defects show a triangular shape that indicates such defects are vacancy type. We count the number density of dot defects in Au versus the neutron fluence. Defects tend to form groupings with increasing of neutron fluence as seen in Fig. 1(a) – (c). The variation of dot defects during annealing was shown in Fig. 5(a).

Silver

Figs. 2(a), (b) and (c) show the defects observed in D-T neutron irradiated Ag to 2.0 x 10^{18} , 2.4 x 10^{19} and 1.2 x 10^{20} n/m², respectively. In Ag, both of interstitial clusters and vacancy clusters were observed in specimens irradiated to 2 x 10^{18} n/m². Majority of interstitial clusters and vacancy clusters of sft were singly isolated ones in Ag irradiated to 2 x 10^{18} n/m². One exception observed was that three sft aligned on a line were in Ag irradiated to 2 x 10^{18} n/m². With increasing the neutron fluence, dot defects tend to form their grouping as seen Fig. 3(b) and (c). It should be noted that interstitial clusters were observed as diffuse dots and triangular sft of vacancy cluster were included in the same grouping. In annealing experiment of TEM Ag specimen, a large sft appeared at 723 K on the position that a group of dot defects were observed previously. Results are shown in Figs. 6(a) and (b). We do not know whether dot defects as observed in Fig. 6(a) were vacancy in nature or unresolved vacancy clusters existed at this position. Inside one of crystal grain, a large size of sft were observed in one portion (left) while another portion (right) only small dot defects versus neutron fluence is shown. The variation of the number density of defects during annealing in Ag was shown in Fig. 5(b).

Copper

Figs. 3(a), (b) and (c) show the defects observed in copper D-T neutron irradiated to the fluence of 1.9×10^{18} , 1.0×10^{20} and 9.1×10^{20} n/m², respectively. It is clearly seen that at low fluence defects observed consist of one or two dots, while at 10^{21} n/m² defects form groupings. In Cu, an isolated sft was observed at low fluence. In Fig. 4(c), the relation of the number density of dot defects versus the neutron fluence was shown. In Fig 5(c), the variation of the number density of dot defects during annealing is shown for the annealing temperature. A 50% of dot defects in Cu disappeared at 473 K annealing. The defects remained at 623 K annealing were sft. The variation of the number density of defects in Fig. 5(c).



Figs. 1(a), (b) and (c) Point defect clusters observed in D-T neutron-irradiated Au at 288 K. (a) 2.6×10^{18} n/m², (b) 2.6×10^{19} n/m², and (c) 3.2×10^{20} n/m².



Figs. 2(a), (b) and (c) Point defect clusters observed in D-T neutron-irradiated Ag at 288 K. (a) 2.0×10^{18} n/m², (b) 2.4×10^{19} n/m², and (c) 1.2×10^{20} n/m².



Figs. 3(a), (b) and (c) Point defect clusters observed in D-T neutron-irradiated Cu at 288 K. (a) 1.9×10^{18} n/m², (b) 1×10^{20} n/m², and (c) 9.1×10^{20} n/m².



Figs. 4(a), (b) and (c) The relation of the number density of point defect clusters versus the neutron fluence. (a) Au, (b) Ag, and (c) Cu.



Figs. 5(a), (b) and (c) The relation of the number density of point defect clusters versus the annealing temperature. (a) Au, (b) Ag, and (c) Cu.



Figs. 6(a) and (b) Grown triangular loops appeared during annealing of D-T neutron irradiated Ag. At 723 K annealing, large size of triangular loops appeared in Ag. It is not sure if dot clusters convert to triangular loops or unresolved vacancy clusters grew to triangular loops.



Fig. 7 In one of crystal grain of Ag, well grown sft form in one portion (left) of grain while only usual size of dot defects were observed in the other side (right). These large sft seem to be larger than the size which form at one damage cascade. The crystal grain in Fig. 7 was connected to large crystal by only upper left part.

DISCUSSION

The present experimental results were explained by a following model. A large part of point defect clusters formed in displacement damage cascades are TEM invisible. At room temperature, they move as clusters. Such movements of point defect clusters were shown by computer simulation previously [11, 12]. At low fluence, they are attracted near dislocations [13]. Interstitial clusters are ejected from damage cascade, move in a crystal and may be able to form isolated immovable small clusters at a vacancy cluster which formed previously. By this mechanism, interstitial clusters form in crystalline grain. In Au, the number of defects is proportional to 1.14 power of neutron fluence at low fluence regime. At high fluence regime it is proportional to 0.74 power of neutron fluence. In a low fluence regime, invisible defects move in crystal and meet another defect of the same nature. Then they relax to visible structure. At high fluence regime, defects of the same nature agglomerate to grow to a large defect. The mutual annihilation of interstitial clusters and vacancy cluster decrease also the number of These cause a 0.74 power dependence of neutron fluence for the number density of dot defects. defects in high fluence regime. In Ag, sft of large size were observed as shown in Fig. 7. In Ag, a large sft form in deformed specimens. We think that sft observed in Fig. 7 were not produced by deformation. We observed an isolated sft in deformed Ag while many sft were observed in Fig. 7.. Moreover, these sft are larger than a vacancy cluster that is formed at one

damage cascade. This means that a small vacancy cluster in Ag moved at room temperature and agglomerated to a large cluster. Vacancy clusters of sft were observed together with interstitial clusters in defect groups in Ag. This suggests that vacancy clusters move and come to a region of defect group. In annealing experiment of irradiated thin foil of Ag, a large size of sft was appeared at 723 K. This suggests that a large TEM invisible vacancy clusters might exit near a defect grouping.

In Cu, a large number of small dots were observed near dislocations at $1.9 \times 10^{18} \text{ n/m}^2$ irradiation. This suggests that interstitial clusters which formed at the cascade in Cu was as small as observed near dislocations and move over long distance after formation of damage cascade. An isolated single sft was observed in Cu irradiated to $1.9 \times 10^{18} \text{ n/m}^2$. This suggests that small vacancy clusters move at room temperature. The formation of small dot defect group is promoted significantly with neutron fluence. The average size of dot defects in Cu increases with the fluence. The 0.9 power dependence of neutron fluence for the number density of dot defects is due to the coalescence of small TEM invisible dot defects for this fluence regime.

SUMMARY

In the case of present D-T neutron irradiation experiment, the fluence was accurately determined by measuring radioactivity of dosimetry foil. At low fluence as 10^{18} n/m², defects observed were isolated point defect clusters. With increasing neutron fluence, defects tend to form groupings of dot defects. These results was explained by the cascade damage modeling that point defects formed in nascent cascades were TEM invisible and moved as a cluster. They are collected to a group of dot defects by the effect of strain field on their movement. The present modeling is different from the previous damage formation at displacement cascades [14] that assumes a fraction of defect yield of damage cascades converted to TEN visible defects.

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