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Journal of Nuclear Materials 258–263 (1998) 1730–1734

journal of nuclear materials

Nucleation and growth of dislocation loops in austenitic stainless steels irradiated by fission and fusion neutrons

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Abstract

Nucleation and growth of interstitial type dislocation loops were investigated by fusion and fission neutron irradiations with improved temperature control. The density of loops formed by both irradiations increased linearly with fluence. The average size of loops also increased. It was concluded that the loops were nucleated directly by the defect processes in the cascade zone, and not by the reaction among the freely migrating point defects in the matrix. If the efficiency of loop formation is assumed to be directly proportional to the energy of the primary knock-on atom (PKA) produced by the neutron, the threshold energy of 20 keV and the efficiency of loop formation of 0.4% at this energy were obtained.

1. Introduction

To develop fusion reactor materials from the results of fission reactor irradiation, it is important to understand the process of microstructural evolution in materials and it is necessary to establish the fission–fusion correlation. Recent development of irradiation techniques, such as strict temperature control during irradiation by an electric heater without any help of gamma-heating [1], enabled us to obtain reliable data for these studies.

The nucleation process of defect clusters is fundamental for the understanding of the defect structure evolution. The nucleation of stacking fault tetrahedra in fcc metals has been studied well and it has been concluded that stacking fault tetrahedra are formed in the cascade zone directly. Each stacking fault tetrahedron represents a subcascade in the cascade at low temperatures [2]. Recent experimental results also show that interstitial type dislocation loops are considered to be formed in the cascade zone, and grow larger by absorption of freely migrating point defects [3].

In this paper, we will first show the dose dependence of nucleation of interstitial type dislocation loops induced by fission and fusion neutron irradiations. Secondly, we will estimate the threshold value of PKA energy needed to form loops and the efficiency of loop formation by comparison of the results between fission and fusion neutron irradiations. Thirdly, an evaluation of loop formation in the cascade zone is carried out using the rate theory based on the diffusion of mobile defects.

2. Experimental procedure

In the fission neutron irradiation, an Fe–16Cr–17Ni alloy was irradiated in Kyoto University Reactor (KUR) and Japan Materials Testing Reactor (JMTR) at 473 K to fluences of 2.1×10^{21} and 2.5×10^{23} n/m^2 ($E > 1$ MeV), respectively. Whereas in the fusion neutron irradiation, an Fe–15Cr–16Ni alloy was irradiated with D–T neutrons in the Rotating Target Neutron Source-II (RTNS-II) at LLNL at 473 K to fluences of 2.4–7.2×10^{22} n/m^2. Each specimen was polished for electron microscopy observation after irradiation, and observed with JEOL TEM 200CX and 2000FX operating at 200 keV.

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3. Result

Dark-field weak-beam images of microstructures formed by fission and fusion neutron irradiations are shown in Figs. 1 and 2. Dislocation loops were observed in all cases examined. The dose dependence of the loop density obtained from Figs. 1 and 2 is shown in Fig. 3. The loop density increased almost linearly with increasing irradiation dose. The size distribution of loops is shown in Fig. 4. The loop size also increased with increasing irradiation dose.

4. Discussion

4.1. The rate equation analysis

The nucleation of loops formed by freely migrating point defects in the matrix was analyzed using the rate theory, the features of the model are as follows:

1. Mobile defects are single interstitials and single vacancies.
2. Di-interstitials are the nuclei of interstitial clusters.

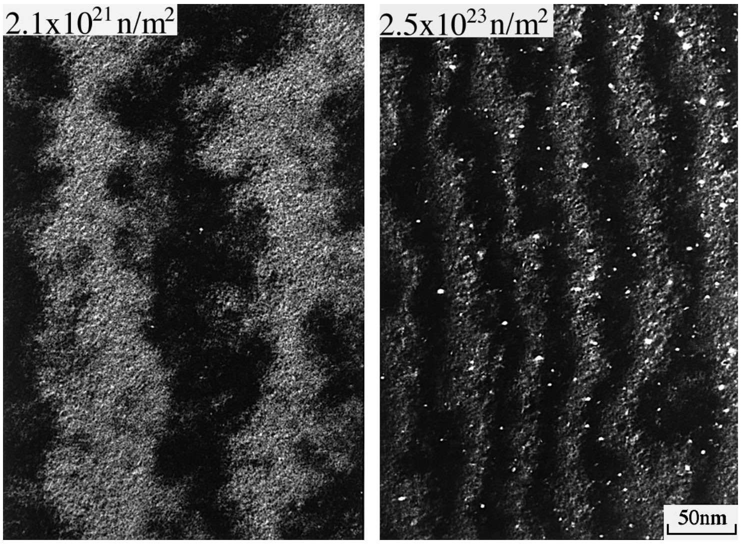

Fig. 1. Dark-field weak-beam images of microstructures formed in an Fe–16Cr–17Ni alloy irradiated by fission neutrons.

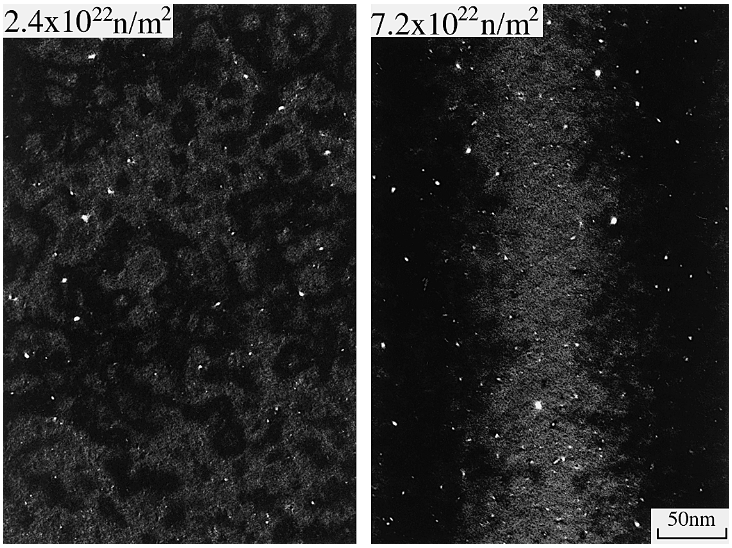

Fig. 2. Dark-field weak-beam images of microstructures formed in an Fe–15Cr–16Ni alloy irradiated by fusion neutrons.

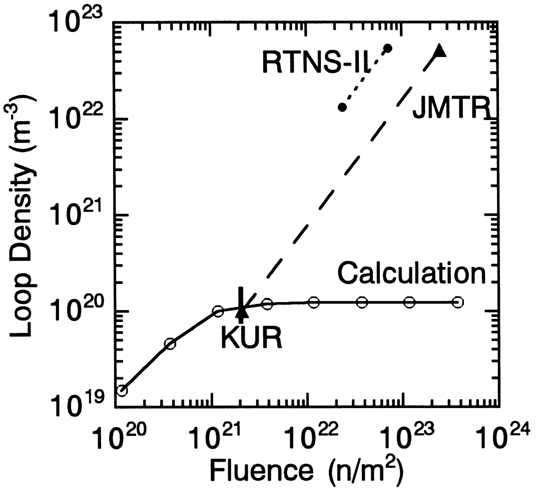

Fig. 3. Dose dependence of loop density in the Fe–Cr–Ni alloys.

3. Thermal dissociation of defects clusters is neglected since 473 K is a low temperature for the Fe–16Cr–17Ni alloy.

The concentrations of interstitial clusters $C_I(n)$ and vacancy clusters $C_v(n)$, in fractional units, are given by

$$\begin{aligned}\frac{\partial C_I(1)}{\partial t} &= P + \nabla^2 D_I C_I(1)\\ &\quad - Z_{IV}(1)(M_I + M_V)C_I(1)C_V(1)\\ &\quad - \sum_{n=2}^{N} Z_{II}(n)C_I(n)M_I C_I(1)\\ &\quad - \sum_{n=2}^{N} Z_{VI}(n)C_V(n)M_I C_I(1)\\ &\quad - Z_{SI}C_S M_I C_I(1), \qquad (1)\end{aligned}$$

$$\begin{aligned}\frac{\partial C_I(n)}{\partial t} &= Z_{II}(n-1)C_I(n-1)M_I C_I(1)\\ &\quad - Z_{II}(n)C_I(n)M_I C_I(1) - Z_{IV}(n)C_I(n)M_V C_V(1)\\ &\quad + Z_{IV}(n+1)C_I(n+1)M_V C_V(1), \qquad (2)\end{aligned}$$

$$\begin{aligned}\frac{\partial C_V(1)}{\partial t} &= P + \nabla^2 D_V C_V(1) - Z_{IV}(1)(M_I + M_V)C_I(1)C_V(1)\\ &\quad - \sum_{n=2}^{N} Z_{VV}(n)C_V(n)M_V C_V(1)\\ &\quad - \sum_{n=2}^{N} Z_{IV}(n)C_I(n)M_V C_V(1)\\ &\quad - Z_{SV}C_S M_V C_V(1), \qquad (3)\end{aligned}$$

$$\begin{aligned}\frac{\partial C_V(n)}{\partial t} &= Z_{VV}(n-1)C_V(n-1)M_V C_V(1)\\ &\quad - Z_{VV}(n)C_V(n)M_V C_V(1) - Z_{VI}(n)C_V(n)M_I C_I(1)\\ &\quad + Z_{VV}(n+1)C_V(n+1)M_I C_I(1), \qquad (4)\end{aligned}$$

where $Z_{ij}(n)$ is the number of spontaneous reaction site with an interstice or a vacancy around an interstitial cluster or a vacancy cluster, the subscripts i and j denote either interstitials or vacancies. $Z_{ij}(n)$ is assumed to be proportional to the square root of the cluster size n. The parameters are listed in Table 1, they are determined on the basis of our previous studies [4,5].

Here, we estimate the amount of defect clusters formed by freely migrating point defects, where their distributions are assumed to be homogenous in space and time.The results of numerical calculations are shown in Fig. 3. The loop density almost increased linearly with fluence at the beginning of irradiation, and then saturated. The loop density of experiments is much higher than that of calculations. This result suggests that the interstitial type dislocation loops are nucleated directly by the defect processes in the cascade zone, not by the reaction among freely migrating point defects in the matrix.

4.2. PKA energy analysis

To compare the nucleation of loops formed by fission and fusion irradiations, we assume that the loops are formed with the same efficiency by the cascade with the same PKA energy in both irradiations. In an analogous

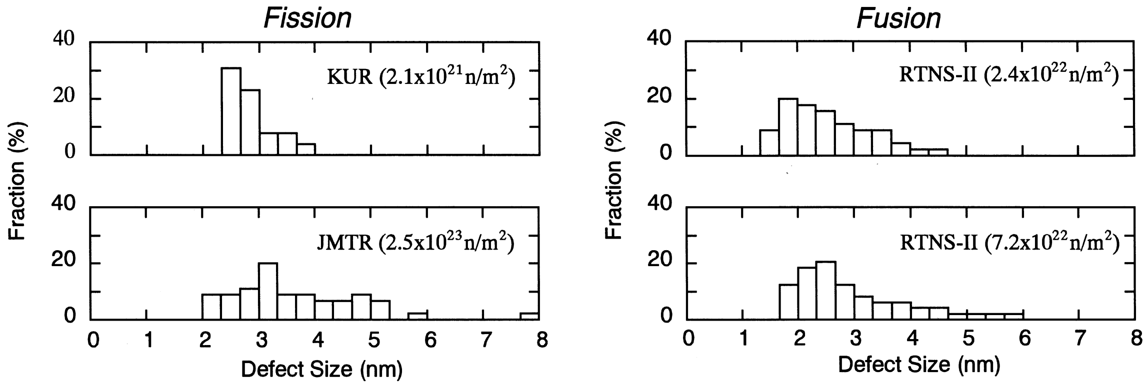

Fig. 4. Size distribution of loops formed by fission and fusion neutron irradiations.

Table 1
Parameters and values used in the calculations

Symbol	Quantity	Value
P	Point defect production rate	3.7×10^{-8} dpa/s
D_{I}	Interstitial diffusion coefficient	2.57×10^3 atomic distances^2/s
D_{V}	Vacancy diffusion coefficient	1.64 atomic distances^2/s
M_{I}	Interstitial mobility	2.57×10^3 jumps/s
M_{V}	Vacancy mobility	1.64 jumps/s
C_{S}	Concentration of permanent sinks	10^{-10}
$Z_{\mathrm{II}}(1)$	Number of sites for reaction between mobile interstices	0.001
$Z_{\mathrm{II}}(n),\ n > 1$	Number of sites for reaction between interstices and I loops with size n	$10n^{1/2}$
$Z_{\mathrm{IV}}(1)$	Number of sites for reaction between interstices and vacancies	100
$Z_{\mathrm{IV}}(n),\ n > 1$	Number of sites for reaction between vacancies and I loops with size n	$10n^{1/2}$
$Z_{\mathrm{VI}}(n), n > 1$	Number of sites for reaction between interstices and V clusters with size n	$10n^{1/2}$
$Z_{\mathrm{VV}}(n)$	Number of sites for reaction between vacancies and V clusters with size n	$10n^{1/2}$

procedure as in the Kinchin–Pease model for Frenkel pair production [6], an area obtained above a threshold energy is assumed to succeed in forming a loop. The number of loops formed by the collision cascade of the PKA energy E_p is $E_p/2E_{d(\mathrm{Loop})}$, where $E_d(\mathrm{Loop})$ is threshold energy of loop formation. The total number of loops can be written as

$$Y_{\mathrm{Loop}} = \alpha N_0 \phi t \int_{E_{d(\mathrm{Loop})}}^{E_{P.\max}} \frac{E_P}{2E_{d(\mathrm{Loop})}} \frac{\mathrm{d}\sigma_P(E_P)}{\mathrm{d}E_p} \mathrm{d}E_P, \tag{5}$$

where α, N_0, ϕ and t are the efficiency of loop formation, atomic density, flux and irradiation time, respectively. $\mathrm{d}\sigma_P(E_P)/\mathrm{d}E_P$ is the differential cross-section of the primary recoil energy spectrum. A similar equation was used for the analysis of subcascade structure in Cu [7].

The threshold energy, which satisfies the total number of loops in the fission and fusion neutron irradiations simultaneously, is 20 keV and the efficiency of loop formation is 0.4% at this energy. For the calculation, the recoil energy spectra obtained by Shimomura [8] were used. The efficiency of loop formation by the cascade is very low, one cascade with energy of 20 keV cannot form one loop. In order to understand this result, the loop formation in the cascade zone is simulated in Section 4.3.

4.3. Simulation of loop formation in the cascade

The primary damage states in collision cascades using molecular dynamics and binary collision modeling have been carried out recently [9–11]. These calculations described the distributions of clustered and unclustered defects at the beginning of a collision cascade. In a nascent cascade, interstitials are found at the periphery of the cascade zone, while vacancies are concentrated at the center of the cascade zone. The configuration of damage state in the material depends on the PKA energy. At high recoil energies, multiple, widely separated damage regions, where each of them is formed by subcascade, can be created in a single recoil event. In the typical fcc metals, such as Cu, Ni, Ag and Au, the average threshold energy to form the subcascade is estimated to be from 10 to 20 keV [12–14], which is close to the threshold energy of the loop formation in the present study. This means that the interstitial type dislocation loops formed by the fission and fusion irradiations are induced by the subcascade.

Nucleation of interstitial clusters has also been simulated using MD, some results indicated that a cascade with energy of 25 keV produced more than one interstitial cluster [15], which is much higher than the result of our experiments. The loops formed in the cascades are considered to be residual clusters after the reaction of interstitials and vacancies in the cascade zone.

In order to understand the experimental results, we simulate the process of loop formation in the cascade zone using the same model ascribed in Section 4.1. In this case, as the initial conditions of $C_{\mathrm{I}}(1)$ and $C_{\mathrm{v}}(1)$, the initial distributions of interstices and vacancies in the cascade zone are used. Fig. 5 shows the disposition of the initial configuration of the cascade, where the interstitial-rich and vacancy-rich regions are simulated by spheres with different radii. The radii of interstitial and vacancy regions are 10 and 3.5 nm, and the concentrations of $C_{\mathrm{I}}(1)$ and $C_{\mathrm{v}}(1)$ are 1.58×10^{-3} and 0.037, respectively. These values are obtained by the TRIM code for a 20 keV irradiation [16]. The size of grains is 10 μm. To save the time of calculation, clusters containing more than 10 defects are neglected.

Fig. 6 shows the efficiency of interstitial type dislocation loops formed by the cascade with energy of 20 keV as a function of interstitial and vacancy diffusion time. The mobility of interstitials is three orders of magnitude higher than that of vacancies, therefore, interstitial clusters are formed and their numbers saturate before vacancy migration. After vacancy migration, the number of loops decreases by absorbing the mobile

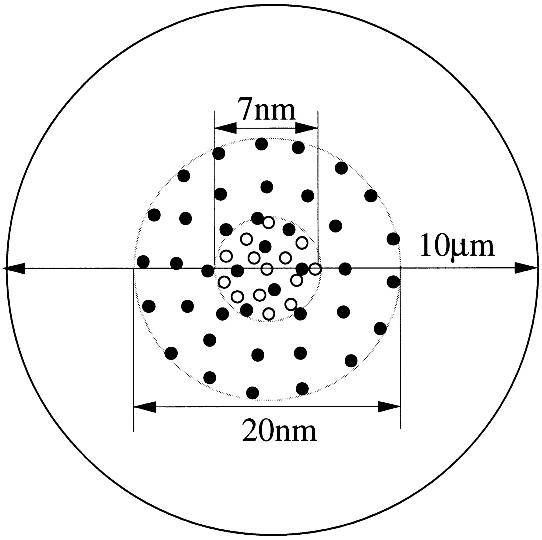

Fig. 5. Disposition of the initial configuration of interstice (●) and vacancy (○) in the cascade. The diameters of interstitial and vacancy regions was determined by the TRIM code for a 20 keV irradiation. The size of grains is 10 μm.

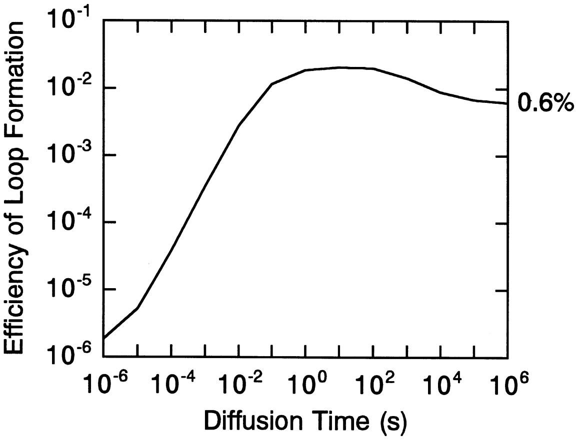

Fig. 6. Efficiency of loops formed by the cascade with an energy of 20 keV as a function of point defect diffusion time.

vacancies, and then it saturates as the amount of mobile vacancies decreases. The efficiency of loops formed by a cascade is 0.6%. This value is close to the 0.4% obtained by comparison of fission and fusion irradiations. The result suggests that the loops are nucleated by the reaction of interstitials and vacancies in the cascade, and the low efficiency of loop formation is due to the annihilation of clusters by vacancies.

5. Conclusions

For the understanding of the nucleation of point defect clusters by high energy neutron irradiation, it is necessary to clarify the effects of the cascade damage. Loop formation in an Fe–Cr–Ni alloy irradiated by fission and fusion neutrons was investigated. The results indicate that the loops were nucleated directly by the reaction of defects in the cascade zone. If the efficiency of loop formation is assumed to be proportional to the PKA energy, the threshold energy of loop formation is 20 keV, and the efficiency of loop formation at this energy is 0.4%. The efficiency of loop formation can be explained by the reaction of interstices and vacancies in the cascade.

This work is a trial to understand the nucleation of interstitial type dislocation loops. Further relevant data and calculations are required to obtain more reliable values.

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