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# Effects of oversized element Sn on diffusion of interstitial clusters in Ni irradiated by ions and neutrons

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**Abstract**

Effects of damage rate on defect structural development in Ni and Ni–Sn were studied by the comparison of neutron irradiation with Ni ion irradiation. Special attention was paid to the damage structural evolution near grain boundaries which was affected by the one-dimensional (1-D) motion of self-interstitial atom (SIA) clusters. The 1-D motion of SIA clusters was observed in Ni irradiated with low fluence in both cases of neutron irradiation and ion irradiation. The alloying element Sn prevented the 1-D motion. The irradiation temperatures to cause the same damage structural evolution in Ni and Ni–Sn was shifted to high temperatures (200 K and 100 K, respectively) with increasing damage rate.


## 1. Introduction

Ion irradiation is an important and useful technique in developing fusion nuclear materials at the present time while a powerful 14 MeV neutron source is not yet available. Usually, the irradiation damage rate and primary knock-on atom (PKA) energy spectrum are greatly different between ion irradiation and neutron irradiation. In order to extrapolate experimental data obtained by ion irradiation to fusion neutron conditions, it is necessary to know the correlation between two radiation environments. The effect of damage rate on microstructural evolution has been studied experimentally and theoretically [1–8]. For example, the temperature of the swelling peak increases with increasing damage rate for void formation. This phenomenon has been analyzed and discussed by Braisford and Bullough [4], and a further theoretical development has been made by Mansur [5]. The shift in the temperature of the swelling peak arises because of the changes in the ratio of recombination of interstitials and vacancies to the reaction rate at the radiation-induced sinks, which increases with increasing damage rate, and increases with decreasing temperature. For the formation of interstitial-type dislocation loops, results of experiment and simulation showed that the number density of loops was proportional to the square root of the damage rate [9].

In recent years, computer simulations have shown that the one-dimensional (1-D) diffusional glide of self-interstitial atom (SIA) clusters plays a key role in void swelling in metals under cascade damage [10–13]. Clear experimental evidence of

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the 1-D motion of SIA clusters and void growth has been demonstrated by the present authors [14–16]. In addition, alloying elements in Ni changed defect structures remarkably [15–18]. Alloying elements are expected to affect the motion of SIA clusters, and thus it is possible to investigate the role of 1-D motion of SIA clusters on microstructural evolution. In neutron irradiated pure Ni, well developed dislocation networks, which were formed by interstitial-type dislocation loops, and voids were observed at 573 K above a dose of 0.026 dpa. The loops and networks did not exist near grain boundaries, because of the escape of SIA clusters by the 1-D motion. On the other hand, after an addition of Sn, no voids were observed by transmission electron microscopy (TEM) [16] or detected by positron lifetime measurement [18]. The volume size factor of Sn in Ni, the ratio of difference in volume between Sn and Ni, is 74.08% [19]. Only some interstitial-type dislocation loops existed near grain boundaries. The existence of loops near grain boundaries was evidence of no 1-D motion of SIA clusters. Recently, simulation results have indicated that the diffusion of SIA clusters can be greatly reduced by substitutional impurities [20–22].

In the present study, damage structures induced by ion irradiation (high damage rate) and neutron irradiation (low damage rate) near grain boundaries were compared to investigate the effect of damage rate on the 1-D motion of SIA clusters and the role of the 1-D motion of SIA clusters in defect structural evolution.

## 2. Experimental procedure

Pure nickel (99.99%) and its binary alloy containing 2 at.% Sn as nominal were tested in the present study. Well annealed specimens were irradiated with neutrons at 573 K to 0.005 dpa in the Kyoto University Reactor (KUR) and to 0.4 dpa in the Japan Materials Testing Reactor (JMTR). In order to investigate void formation in Ni–Sn alloys, some Ni–Sn specimens were irradiated to 1 dpa at 703 K in the fast flux test facility (FFTF) with the materials open test assembly (MOTA). The damage rate at the JMTR irradiation was $1.0 \times 10^{-8}$ dpa/s (0.026 dpa) and $1.7 \times 10^{-7}$ dpa/s (0.4 dpa), and that at the FFTF irradiation was $7.0 \times 10^{-8}$ dpa/s. Ion irradiation was performed by Ni ions with 2.4 MeV using a Tandem type accelerator in

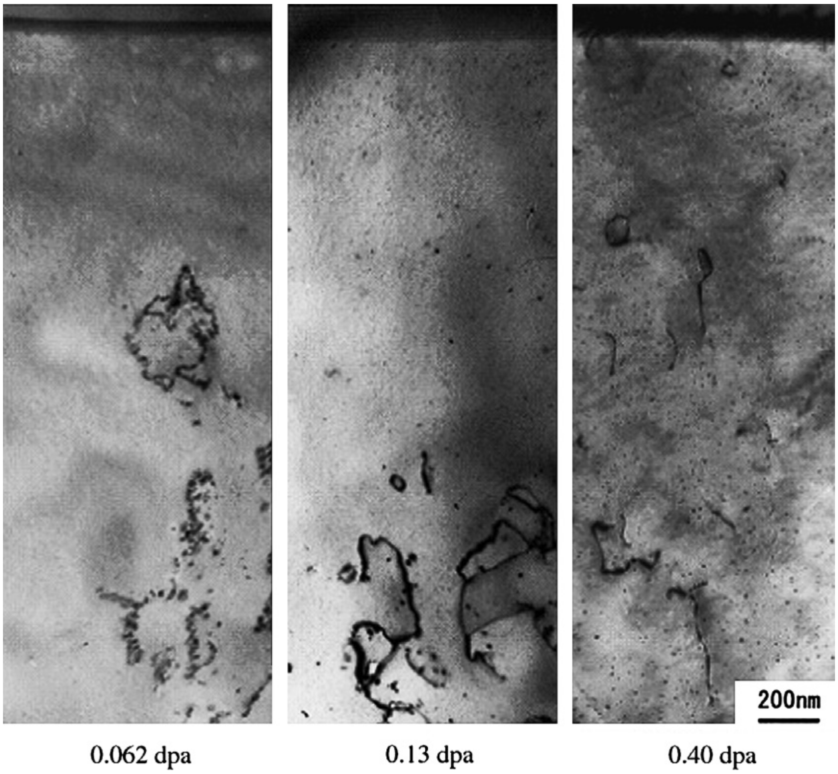


Fig. 1. Dislocation microstructures near grain boundaries in neutron irradiated Ni (JMTR) at 573 K.

Kyushu University. The temperature and dose were from 573 K to 873 K and from 0.01 dpa to 1 dpa, respectively. The damage rate was $5.6 \times 10^{-4}$ dpa/s. After irradiation, microstructural features such as dislocations and voids were observed by TEM. A single jet thinning of specimens was performed to observe the microstructures at the damage peak in specimens irradiated with ions. All dislocation and void analyses were done by imaging of a $g = [200]$ reflection near a [011] zone axis.

## 3. Results

### 3.1. Damage structures formed by neutron irradiation

Fig. 1 shows the dislocation images near grain boundaries in Ni irradiated with neutrons at 573 K. With increasing irradiation dose, interstitial-type dislocation loops grew to dislocations. There were no interstitial-type dislocation loops or dislocations near grain boundaries, and the width of this region was about 800 nm and 400 nm in irradiation to 0.062 dpa and 0.40 dpa, respectively. Voids were also observed by TEM and grew with increasing irradiation dose. There were no voids near grain boundaries [14].

Fig. 2 shows defect structures near grain boundaries in Ni–Sn irradiated by the JMTR at 573 K up to 0.4 dpa. Contrary to damage structures in Ni, interstitial-type dislocation loops were formed only near grain boundaries, and the width of this region was about 300–400 nm in the present irradiation doses. Voids were not observed in Ni–Sn under the JMTR irradiation. However, dislocations, grown from interstitial-type dislocation loops, were formed not only near grain boundaries but also in the interior of the matrix when the irradiation dose increased to 1 dpa and the irradiation temperature increased to 703 K, as shown in Fig. 3. Voids as well as dislocations were also formed in the matrix.

### 3.2. Damage structures formed by ion irradiation

Fig. 4 shows the dislocation image near grain boundaries in Ni irradiated with Ni ions. Although no voids were observed in ion irradiated Ni even to the dose of 1 dpa at 573 K, the defect structure at 0.1 dpa near grain boundaries, with no interstitial-

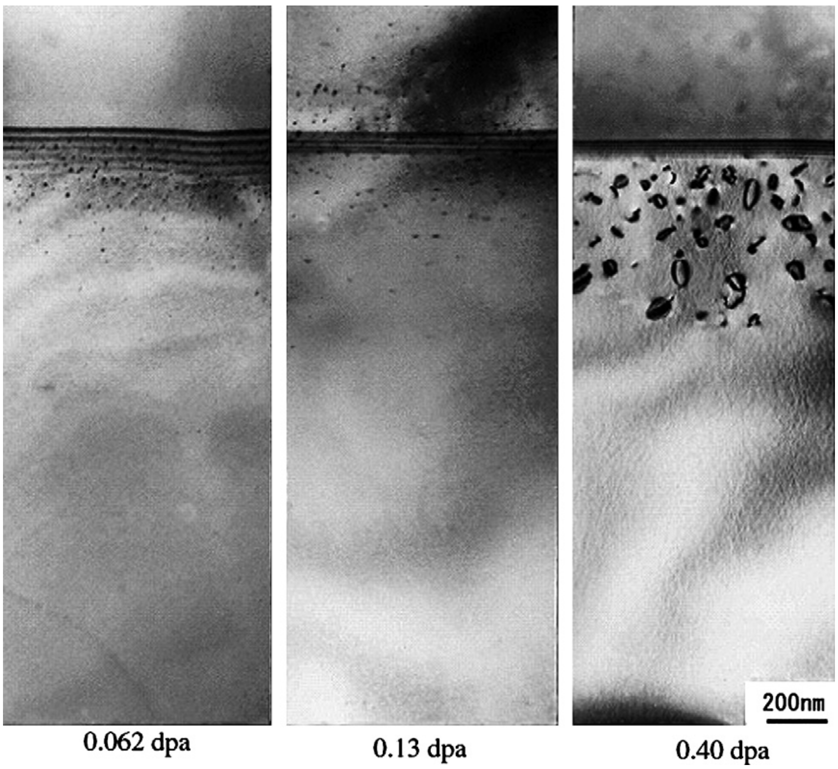


Fig. 2. Dislocation microstructures near grain boundaries in neutron irradiated Ni–Sn (JMTR) at 573 K.

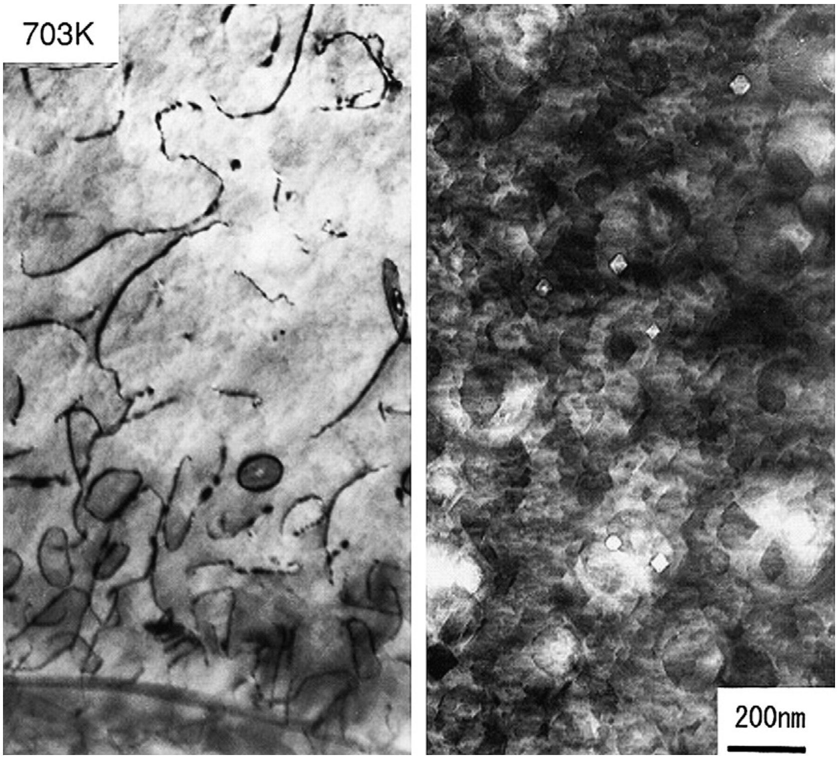


Fig. 3. Microstructures in Ni–Sn irradiated with neutrons at 703 K to 1 dpa.

type dislocation loops and no dislocations in a region about 200 nm wide, was the same as that in neutron irradiated Ni. With increasing irradiation dose, the density of interstitial-type dislocation loops near grain boundaries increased. It was almost the same between grain boundaries and the interior of the matrix after irradiation of 1 dpa, and no voids were observed. The interstitial-type dislocation loops were formed homogeneously in Ni–Sn alloy at 573 K from 0.1 dpa to 1 dpa, as shown in Fig. 5. At the high temperature of 673 K, however, the density of interstitial-type dislocation loops increased near grain boundaries in a region about 200 nm wide. Furthermore, the density of interstitial-type dislocation loops was again the same between grain boundaries and the interior of the matrix by irradiation at 773 K. Voids were not observed in Ni–Sn under these conditions.

### *3.3. Comparison of damage structures formed by neutron irradiation with ion irradiation*

The evolution of interstitial-type dislocation loops near grain boundaries in Ni–Sn is shown in Fig. 6. In Ni–Sn, the formation of interstitial-type dislocation loops near grain boundaries was shifted to high temperature by 100 K with ion irradiation, whereas the shift to high temperature was 200 K for Ni.

## 4. Discussion

It is thought that the dislocation loops and networks do not exist near grain boundaries since the SIA clusters escape to permanent sinks by 1-D motion. The width of the region where there are no dislocation loops is thought to be the free path of 1-D motion. The lack of interstitial-type dislocation loops and dislocations in Ni irradiated by neutrons at 573 K indicated that SIA clusters migrated by such 1-D motion, which induced the formation and growth of voids. In addition, the SIA clusters also migrated by 1-D motion in Ni irradiated by ion to 0.1 dpa at 573 K. This suggests that irradiation damage rate does not affect the 1-D motion of SIA clusters. The observation that no voids were formed in Ni irradiated by ions indicates that there are other factors that determine the void formation besides the 1-D motion of SIA clusters. The reason is thought to be that the recombination of intersti-

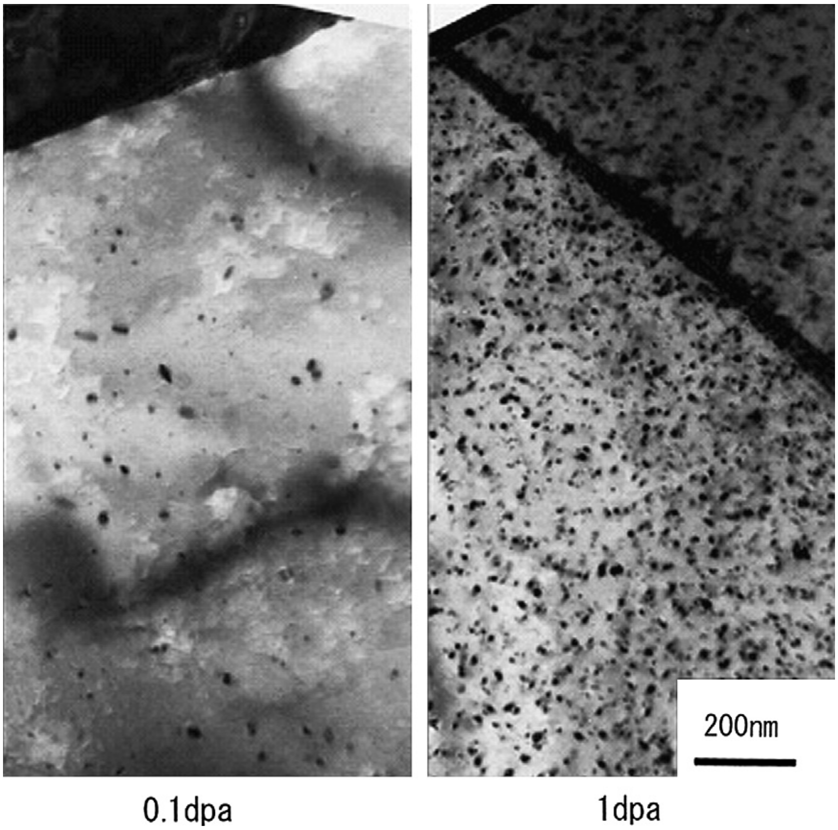


Fig. 4. Dislocation microstructures near grain boundaries in ion irradiated Ni at 573 K.

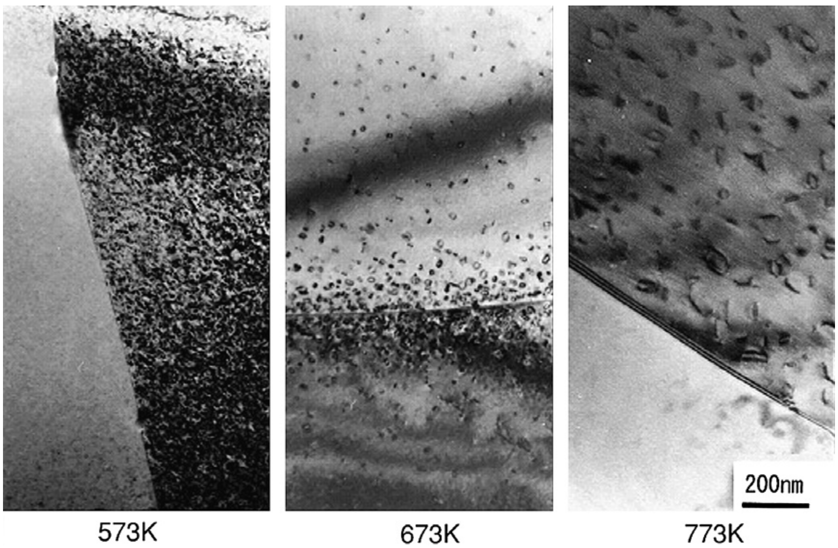


Fig. 5. Temperature dependence of interstitial-type dislocation loops induced by ion irradiation near grain boundaries in Ni–Sn.

tials and vacancies is higher in irradiation with ions than that with neurons. With increasing ion irradiation dose, the density of interstitial-type dislocation loops increased. The 1-D motion of SIA clusters was suppressed by increasing irradiation dose. The suppression can be explained by the shortening of

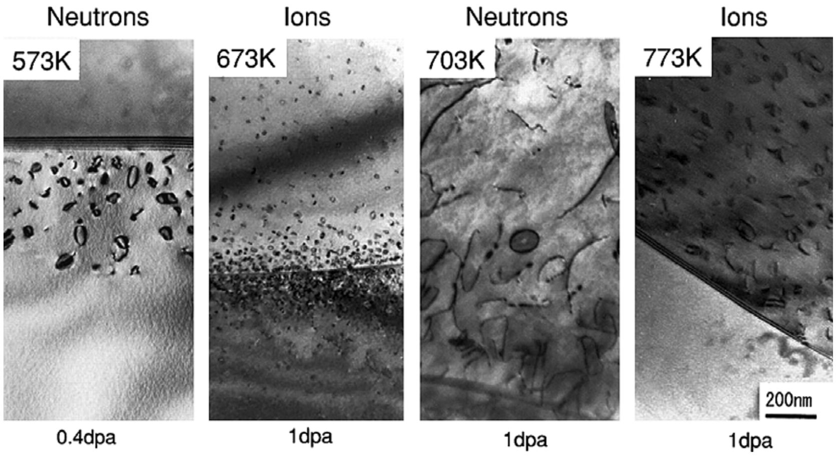


Fig. 6. Comparison of interstitial-type dislocation loops formed by neutron with ion irradiation near grain boundaries in Ni–Sn.

the free path of 1-D motion due to high SIA cluster density. This conclusion agrees with the experimental results shown in Fig. 1, where the free path of 1-D decreased with increasing irradiation dose.

Contrary to the damage microstructures in neutron irradiated Ni, interstitial-type dislocation loops were formed only near grain boundaries in neutron irradiated Ni–Sn at 573 K. This indicated that SIA clusters formed in the grain boundaries did not migrate in Ni–Sn. Oversized Sn may trap SIA clusters and decrease their mobility in their stress fields. In the interior of the matrix, vacancies recombined with interstitials clusters. As the result, the formation of interstitial-type dislocation loops and voids is suppressed in the matrix [14–16]. However, both in the neutron (703 K) and ion irradiations (773 K), the interstitial-type dislocation loops were formed in the interior of the Ni–Sn matrix, whereas voids were only formed by neutron irradiation. It was concluded that the oversized element Sn suppressed the 1-D motion of SIA clusters only at low temperatures (for example, below 673 K), and this effect of Sn disappeared with increasing irradiation temperature. The lack of void formation by ion irradiation led to the formation of a high density of interstitial clusters at high irradiation rate.

## 5. Conclusion

The effects of damage rate on the 1-D motion of SIA clusters were investigated in Ni and Ni–Sn by examining the defect structures near grain boundaries. The 1-D motion of SIA clusters was observed in Ni irradiated with neutrons and ions at low irradiation dose. The 1-D motion in ion irradiated Ni was suppressed by high dose irradiation. The oversized element Sn prevented the 1-D motion of SIA clusters. The temperature needed to develop dislocation structures by low damage rate irradiation with neutrons was increased by 200 K in Ni and by 100 K in Ni–Sn by high damage rate irradiation with ions.

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