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# Effects of chromium on the one-dimensional motion of interstitial-type dislocation loops in iron

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**Abstract**

The one-dimensional motion of interstitial-type dislocation loops in Fe–9Cr ferritic alloy is studied by in situ transmission electron microscopy. Loop motion is examined under 1000 kV high-energy electron irradiation at temperatures ranging from 110 to 680 K and under thermal annealing from 295 to 960 K without introduction of additional displacement subsequent to the irradiation at 110 K. The results are compared with those in pure bcc Fe, and the effects of Cr on the motion of loops are extracted. Under high-energy electron irradiation, the motion frequency of loops is only slightly reduced by Cr. In contrast, under annealing, the motion frequency of loops in Fe–9Cr is significantly reduced by Cr. The origins of such two-way effects of Cr on the motion frequency of loops are discussed.


## 1. Introduction

It is now well understood that one-dimensional high-speed motion of self-interstitial atom (SIA) clusters play important roles in radiation-induced microstructural evolution in metals and alloys (e.g. [1]). Therefore, the motion of SIA clusters constituted by small number (a few to several tens) of SIA atoms in several metals and alloys has been extensively studied by molecular dynamics calculations (e.g. [2]). In addition, the motion of larger SIA clusters of interstitial-type dislocation loops a few to a few tens nanometers in diameter has been experimentally examined by transmission electron microscopy (TEM) for pure metals such as fcc Ni [3], bcc Fe [3–5] and bcc V [6]. These experimental studies clarified that such small loops can make one-dimensional back-and-forth motion along the directions of their Burgers vectors parallel to the closest packing orientations by the application of force [3,5,6] or heat [5]. In contrast, the effect of the addition of impurity (solute) atoms on the motion of loops has been examined for a few metals, such as V [6] and Cu [7], by TEM. These studies showed that the addition of both substitutional and interstitial impurity atoms reduce both motion frequency and motion distance of loops, however the origin of such suppression of loop motion by impurities has not been elucidated.

In the present study, in situ TEM observation of the motion of loops in Fe–9Cr ferritic alloy, which is a candidate for the first wall structure materials of nuclear-fusion reactors (e.g. [8]), was performed under high-energy electron irradiation and under thermal annealing subsequent to the irradiation. The motion of loops in Fe–9Cr was compared with those in pure bcc Fe [4,5], and the extraction of the effects of Cr on loop motion was aimed at.

## 2. Experimental procedures

The specimen of pure bcc Fe adopted for the reference was high purity Fe (99.999%) supplied by Showa Denko Inc. It was rolled down to 0.08 mm in thickness. Sheets of pure Fe were pre-annealed at 1120 K for 1 h in

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a hydrogen atmosphere. Then, they were electrochemically polished for TEM [4,5]. The specimen of Fe–9wt%Cr ferritic alloy was cast from Fe (99.995%) and Cr (99.992%) supplied by Johnson Matthey in a hydrogen atmosphere in order to decarburize the specimen [9]. Sheets of Fe–9Cr of 0.08 mm thickness were pre-annealed at 1220 K for 1 h in a high vacuum of $10^{-6}$ Pa, which was followed by electrochemical polishing.

High-energy electron irradiation and in situ observation was performed in a high-voltage electron microscope H-3000 (Hitachi) operated at an acceleration voltage of 1000 kV at temperatures ranging from 110 to 680 K. Since the motion frequency of loops in pure Fe is higher with higher beam fluxes [5], the beam flux, was set to be a high value of $1\times10^{24}$ $e^-/m^2s$ which corresponds to a displacement per atom (dpa) rate of $6\times10^{-3}$ dpa/s.

In addition, after the induction of loops by high-energy electron irradiation at a beam flux of $9\times10^{22}$ $e^-/m^2\,s$ ($5\times10^{-4}$ dpa/s) to a fluence of $3\times10^{25}$ $e^-/m^2$ at 110 K, the motion of loops under isochronal annealing from 295 to 960 K was observed, where 200 kV TEM was employed to prevent displacement by irradiation.

For in situ TEM observation, bright-field imaging was used. The reflection adopted was mainly $\mathbf{g} = 110$ with the deviation parameter from the exact Bragg condition $s$ of 0.03 $nm^{-1}$ for 1000 kV TEM and 0.06 $nm^{-1}$ for 200 kV TEM. The images were recorded through CCD cameras with a time resolution of 1/30 s.

## 3. Experimental results

The results in Fe–9Cr will be described with reviewing of the summary of the experimental results in pure Fe [4,5]. The results in Fe–9Cr will be compared with those in pure Fe.

### 3.1. Behavior of loops under high-energy electron irradiation

In pure Fe under high-energy electron irradiation, loops were formed below 390 K, and they were not formed up to a fluence of $3\times10^{26}$ $e^-/m^2$ above 400 K. The volume density of loops was lower at higher temperatures. In Fe–9Cr, loops were formed at all the temperatures examined. The density of loops was comparable with that of pure Fe at comparatively low temperatures below around 200 K. In contrast, at higher temperatures, the density of loops in Fe–9Cr was remarkably higher than that in pure Fe. For example, at 295 K, density of loops in pure Fe was $5\times10^{21}/m^3$ as the maximum value, and that in Fe–9Cr was $2\times10^{22}/m^3$ as the saturated value, which was several times as large as the former. Yoshida et al. [10,11] have performed high-energy electron irradiation of Fe–10Cr, and concluded that enhanced nucleation of loops due to trapping of SIAs by mixed dumbbells constituted by an SIA and a Cr atom does not occur and the nuclei of loops in Fe–10Cr are homogeneous tri-intersititials. The enhanced nucleation of loops by Cr addition observed here seems to contradict with their conclusion. On the other hand, vacancies in pure Fe are known to be thermally mobile above around 220 K [12], while those in Fe–10Cr are thermally mobile above around 520 K [13]. Such suppression of the migration of vacancies by the presence of Cr is to be due to the trapping of vacancies by Cr atoms. This will lead to the excess of SIAs, which may cause the

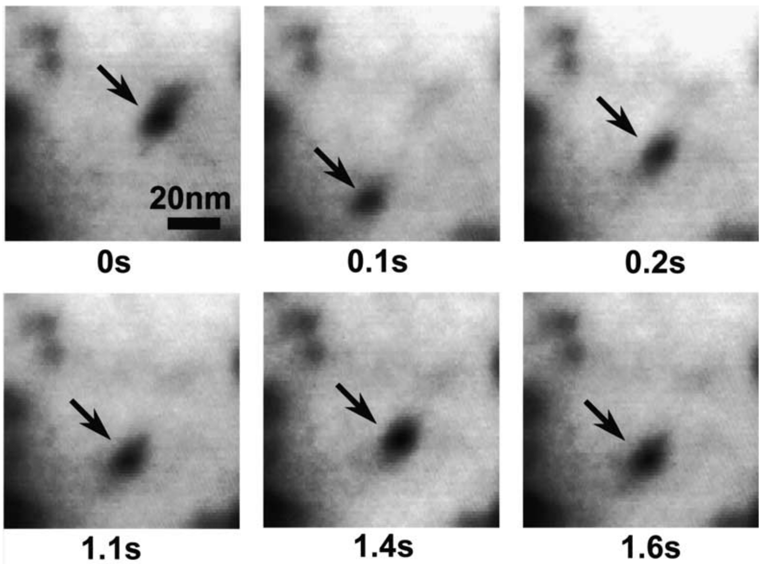


Fig. 1. Motion of interstitial-type dislocation loops in pure Fe under high-energy electron irradiation with a beam flux of $1\times10^{24}$ $e^-/m^2\,s$ at 190 K.

Table 1
Values of quantities related to the motion of interstitial-type dislocation loops in pure Fe and Fe–9Cr under high-energy electron irradiation with a beam flux of $1\times10^{24}$ $e^{-}/m^{2}s$ below a fluence of $6\times10^{25}$ $e^{-}/m^{2}$. The 'motion frequency' is defined as the ratio of the number of the occurrence of one-dimensional motion of loops to the number of all the loops per second

| | Temperature [K] | Pure Fe | Fe–9Cr alloy |
|---|---|---|---|
| Motion frequency [%/s] | 190 | 32(±8) | 7(±2) |
| | 295 | 1.0(±0.3) | 12(±4) |
| | 375 | < 0.4 | 1.0(±0.3) |
| Motion distance [nm] | 190 | 20(±10) | 10(±3) |
| | 295 | 30(±8) | 10(±3) |
| | 375 | – | 10(±3) |
| Volume density [/m$^3$] | 190 | $2\times10^{22}$ | $1\times10^{22}$ |
| | 295 | $3\times10^{21}$ | $< 2\times10^{22}$ |
| | 375 | $1\times10^{21}$ | $< 7\times10^{21}$ |
| Size [nm] | 190 | < 20 | < 20 |
| | 295 | < 20 | < 10 |
| | 375 | < 100 | < 10 |

enhancement of the nucleation of loops at comparatively high temperatures where the binding between Cr and vacancies occurs.

In pure Fe, loops isolated from other dislocations could make one-dimensional back-and-forth motion, as shown in Fig. 1. Burgers vectors of them was $\mathbf{b}=1/2\langle 1\,1\,1\rangle$, and the motion direction of loops was parallel to their Burgers vectors. In Fe–9Cr, loop motion similar to that in pure Fe was also observed. Table 1 indicates the values of quantities related to the motion of loops in pure Fe and Fe–9Cr below a comparatively low fluence of $6\times10^{25}$ $e^{-}/m^{2}$ where tangled dislocations were not formed, at 190, 295 and 375 K. Here, the motion frequency was defined as the ratio of the number of the occurrence of one-dimensional motion of loops to the number of all the loops per second. In pure Fe, the motion frequency monotonously decreases with the rise in temperature. In contrast, in Fe–9Cr, a relationship

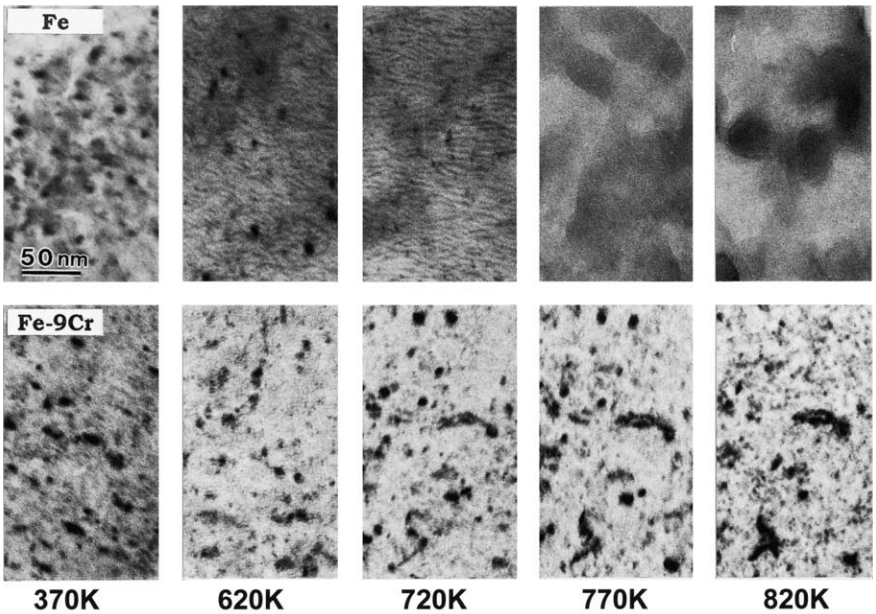


Fig. 2. Microstructural variation in pure Fe and Fe–9Cr under thermal annealing subsequent to high-energy electron irradiation with a beam flux of $9\times10^{22}$ $e^{-}/m^{2}\,s$ to a fluence of $3\times10^{25}$ $e^{-}/m^{2}$ at 110 K.

between the motion frequency and temperature is not monotonous. The motion frequency in Fe–9Cr is lower than that in pure Fe at 190 K. On the other hand, the former is higher than the latter at 295 and 375 K. The origins of such a strange effect of Cr addition on the motion frequency of loops will be discussed later. The values of motion distance during a jump of a loop in Fe–9Cr are smaller than those in pure Fe. Such reduction of the motion distance of loops by Cr addition is considered to appear because Cr atoms in the matrix act as the obstacles in the way of loops.

### 3.2. Behavior of loops under thermal annealing

Fig. 2 shows a comparison between the temperature variations in the microstructure in pure Fe and Fe–9Cr under thermal annealing. In pure Fe, the microstructure is coarsened mainly due to the escape of loops to the specimen surfaces or coalescence with other loops owing to one-dimensional motion of loops in the temperature range from 450 to 700 K. The motion frequency of loops located apart from others was higher at higher temperatures. This implies that loops also make one-dimensional motion by the application of heat not only by force. Some of loops begin to shrink above around 600 K, and the coarsening progresses due to the shrinkage and subsequent disappearance of loops above around 700 K.

In Fe–9Cr, the motion of loops was very hardly observed at all the temperatures examined. Loops began to shrink and disappear above around 750 K. From the comparison between the behaviors of loops in pure Fe and Fe–9Cr, it can be stated that both the motion and shrinkage of loops are significantly suppressed by Cr addition under the thermal annealing.

## 4. Discussion

Under the high-energy electron irradiation at comparatively low temperatures, the motion frequency of loops in Fe seemed to be reduced at 190 K by the addition of Cr, whilst it seemed to be raised at 295 and 375 K by it. The origins of such a strange effect of Cr addition are interpreted as follows. From the data in Table 1, a relationship between the motion frequency of loops and the volume density of loops can be made as shown in Fig. 3. The motion frequency monotonously increases with the increase in the volume density in both pure Fe and Fe–9Cr. Therefore, the most important factor governing the motion frequency of loops under this condition is considered to be the density of loops. When the density of loops becomes higher, the force acting on each loop by its surrounding loops becomes greater. Such an increase in the force may increase the motion frequency of loops. Since the motion frequency

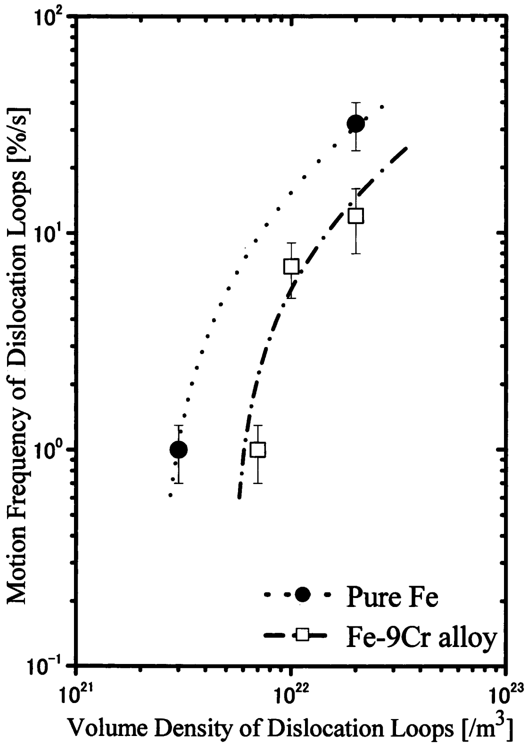


Fig. 3. Relationship between motion frequency of interstitial-type dislocation loops and the volume density of loops in pure Fe and Fe–9Cr under high-energy electron irradiation, made from the data in Table 1. Here, the curved lines are drawn for a comparison between the values of motion frequency in pure Fe and Fe–9Cr.

of loops in pure Fe at a density is marginally higher than that in Fe–9Cr at the same density, it can be stated that Cr has the effect of reducing the motion frequency of loops slightly.

In contrast, under annealing at comparatively high temperatures, the motion frequency was remarkably reduced by Cr even though the density of loops in Fe–9Cr was higher than that in pure Fe.

Such two-way effects of Cr on the motion frequency of loops can be understood by the following temperature dependence of the behavior of Cr. Under the thermal annealing, Cr atoms are considered to be remarkably segregated around loops due to the migration of Cr atoms towards the dilatational-strain field formed around loops by coupling with vacancies accumulated by the irradiation since Cr is a slightly over-size solute [14] and are bound with vacancies. In fact, segregation of Cr around loops has been proved for Fe–10Cr irradiated with high-energy electrons at 773 K [11]. Such segregation is considered to suppress the motion of loops severely due to strong pinning (locking) of dislocations. In contrast, under the high-energy electron irradiation at lower temperatures, segregation of Cr cannot effectively occur, therefore the motion of loops may be only slightly suppressed due to the weak pinning of dislocations by Cr atoms which are almost randomly located in matrix.

## 5. Conclusions

Two-way effects of an alloying element Cr in bcc Fe on the frequency of one-dimensional motion of interstitial-type dislocation loops were revealed by a comparison between the motion frequencies of loops in pure Fe and Fe–9Cr ferritic alloy. At comparatively high temperatures above around 450 K, Cr atoms are considered to suppress the motion of loops significantly due to the segregation of Cr at the periphery of loops owing to the migration of couples of Cr and a vacancy towards loops and trapping of Cr at the periphery of loops. In contrast, under the irradiation with energetic particles at comparatively low temperatures below around 390 K, such segregation may not occur and Cr atoms only slightly reduce the motion frequency of loops. In both temperature ranges, Cr atoms in the matrix are considered to stand in the way of loops, hence Cr has the effect of reducing the motion distance of loops.

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