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In situ observation of glide motions of SIA-type loops in vanadium and V–5Ti under HVEM irradiation

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Abstract

In situ observation of one-dimensional glide motion of defect clusters under HVEM irradiation has been conducted. In order to examine the effects of interstitial impurity atoms and alloying elements, vanadium, in two purity levels, and V–5Ti have been prepared and examined. In all materials, self-interstitial type dislocation loops are formed and grow during irradiation. Loop glide was frequently observed under conditions of extremely high irradiation intensity. Motion was more frequently observed when clusters were small, and generally, decreased as clusters grew. All clusters glide one-dimensionally, back and forth between other neighboring loops, indicating the driving force for motion is the variation of the force field gradient around loops. The frequency and distance of motion are significantly decreased by interstitial impurities and solutes in the material. The dynamical characteristics of one-dimensional motion under HVEM irradiation are discussed. It can be concluded that impacts of one-dimensional motion in practical alloys are less important than those in pure metals.

1. Introduction

It has been pointed out by recent extended atomic-scale modeling studies that one-dimensional motion of defect clusters plays a very important role in damage production, bias mechanism and microstructural evolution during irradiation, particularly under cascade damage conditions [1–3]. However, though extensive studies have been done on formation and evolution processes for defect clusters, there are only a few experimental studies on their stability and mobility. This is considered to be due to the difficulties of detecting such phenomena, which occur over short times and distances, involving pico- and microscales.

One-dimensional motion under HVEM irradiation has been reported by Kiritani, in fcc nickel and bcc iron [4]. He showed examples of direct and indirect evidence for motion, and successfully pointed out their possible mechanisms and impacts on defect reactions.

In this paper, the glide motion of and interactions among self-interstitial (SIA) clusters in vanadium and its alloys are reported and discussed. In order to examine the effects of interstitial impurities on the motion, high purity (HP) specimens as well as nominally pure (NP) ones have been examined.

2. Experimental procedure

Two types of vanadium with different purity and V–5(at.%)Ti specimens were prepared. V–5Ti has been selected in order to examine the effect of solutes on the one-dimensional motion, since titanium has been considered to be a major alloying element in vanadium alloys as fusion reactor materials because of its very attractive alloying effects [5]. The ingot materials were cold rolled to approximately 0.2 mm thick sheets. TEM disks 3 mm in diameter were punched from the sheet specimens, followed by electro-polishing for NP specimens. HP specimens were prepared by using a zirconium

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treatment. Zirconium foils 0.015mm in thickness were first bonded to both faces of the electro-polished vanadium sheets by cold rolling. Then TEM disks were punched from these foil-bonded sheets. More details of this purification method can be found elsewhere [6]. For all disk samples, recrystallization annealing was done at 1327 K in a vacuum of 2×10^{-4} Pa for 2 h. After these treatments, the disk specimens were thinned by electropolishing for TEM observation.

The results of chemical analysis of the major interstitial impurities in these specimens are presented in Table 1. In (HP) vanadium, impurities, particularly oxygen, have been drastically removed, by a factor of nine compared to (NP) vanadium, as shown in Table 1.

Electron irradiation and in situ microstructural observations were conducted in the HVEM (JEM ARM-1250) at the High Voltage Electron Microscope Laboratory in Tohoku University with an electron accelerating voltage of 1250 kV. The motions were recorded using a video recoding apparatus with a CCD camera, which was located under the fluorescent screen. The image acquisition of the CCD is TV rate, i.e. 60 frames s^{-1}. The detection and analyses have been done for intervals of 1/30 s. For small loops with a diameter of about 2 nm, images averaged over three successive frames were recorded, in order to obtain finer images for more detailed analysis. The irradiation temperature and damage rate were varied in the range from RT to 573 K and from 2.1×10^{23} to 1.1×10^{24} e m^{-2} s^{-1}, respectively.

Table 1
Main interstitial impurity content in the specimens examined in this study (appm)

	O	C	N	Amount
(NP) vanadium	560	115	22	697
(HP) vanadium	41	34	4	79
V–5Ti	1293	216	76	1585

3. Results

3.1. Characteristics of the one-dimensional motion of SIA clusters

The one-dimensional glide motion of an observable cluster in an electron microscope, within a diameter of above about 2 nm and containing several tens of SIAs, was detected and analyzed. The dynamical characteristics of the motion, however, are expected to reflect those of even smaller clusters.

The fundamental characteristics of the motion deduced from the study by Kiritani [4] and this study can be summarize as follows:

1. Observed glide motion is one-dimensional. Changes in the glide direction have never been observed, at least in this study.
2. The direction of the motion is along [1 1 0] in fcc and [1 1 1] in bcc (the close-packed directions), respectively.
3. The motion is intermittent, similar to the case of dislocation glide past stable obstacles under shear stresses.
4. Typical motion of small SIA clusters repeats a back-and-forth motion between two clusters.
5. Motion is frequently observed (only) under irradiation conditions of extremely high damage rate. Therefore, the motion is most frequent while the defect densities and growth rates are high.

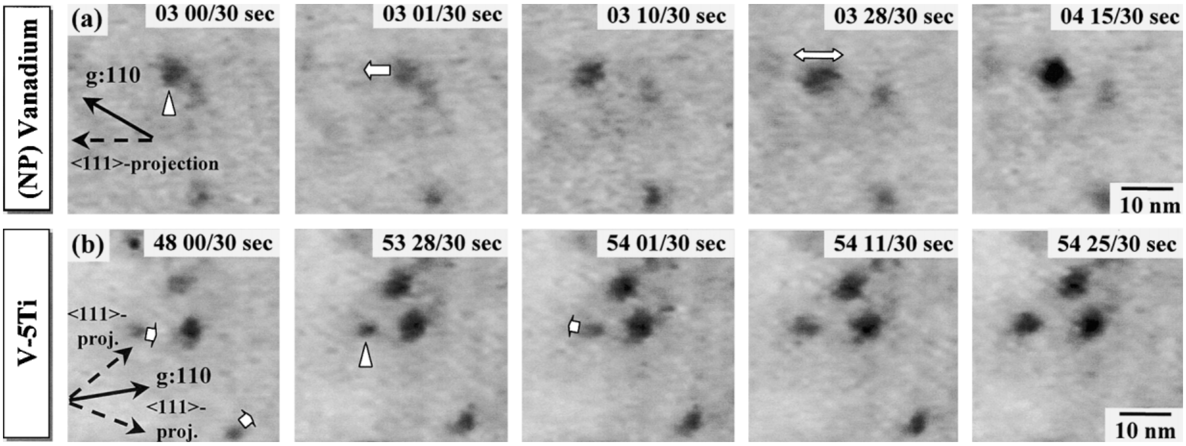

Fig. 1. Typical examples showing motion of small defect clusters in (NP) vanadium (a) and V–5Ti (b). Irradiated with 1250 keV electrons at an intensity of 1.1×10^{24} e m^{-2} s^{-1} and temperatures of RT in (a) and 373 K in (b), respectively. Note that all of the loci of the cluster motions are along (the projection of) $\langle 1\,1\,1 \rangle$ direction.

Fig. 1 shows typical examples of the cluster glide motions in (NP) vanadium (a) and V–5Ti (b). In (NP) vanadium (a), the contrast of the cluster with a loop shape (first image) weakened just before motion was observed (second image). Then, the cluster glided and its contrast recovered just after it stopped moving (third image). In the fourth image, the cluster again glided by vibrating over quite short distances. Finally, the defect stopped moving and again acquired strong contrast as a loop. This kind of back-and-forth motion is typical and was also observed quite often in (HP) vanadium and V–5Ti.

The glide distance (GD) is nearly proportional to the distance between clusters. In (HP) vanadium and V–5Ti, the distance drastically increased and decreased, respectively. It is worth emphasizing that in (HP) vanadium, glide of several tens of nms was often observed, contrary to the case of only a few nms in V–5Ti.

3.2. Other noticeable features of cluster glide motion

Fig. 2 shows examples of noticeable features of motion. In Fig. 2(a), it can be shown that a cluster with a loop shape repeatedly appeared and disappeared. This kind of phenomenon was observed at times in all materials examined in this study. Examples of correlated motion is shown in Fig. 2(b) and (c). Two large loops glided in the same direction at the same time, as seen in Fig. 2(b). In Fig. 2(c), a group of clusters sequentially glided one after another, after the first of them (marked by a triangular) glided and disappeared (third image), like a chain reaction. From these observations, the driving force for motion appears to be a function of the strain field gradient around the cluster itself and/or the surrounding nearby clusters. This possibility is also supported by the results shown in Fig. 3. Fig. 3 shows the correlation between the defect growth (above) and the motion frequency (below) in (NP) vanadium. As seen in the figures, the frequency is high while clusters are small and are growing rapidly, and decreases with decreasing growth rate. This correlation has also been reported by Kiritani [4]. In (HP) vanadium and V–5Ti, the growth rate and glide frequency of the clusters are much higher and lower than those in (NP) vanadium, respectively, indicating interstitial impurities and solutes act as strong trapping sites for point defects and obstacles against glide.

4. Discussion

4.1. Dynamical characteristics of the one-dimensional motion

As already stated above, the dominant factor responsible for the driving force for motion is considered

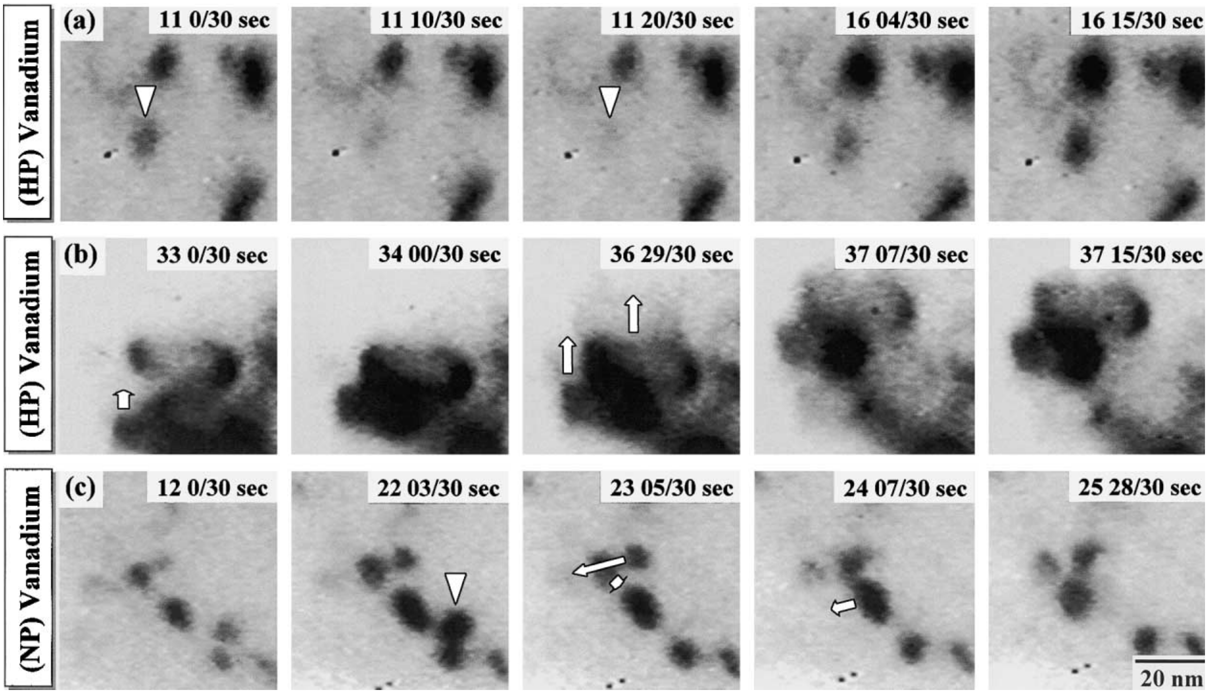

Fig. 2. Typical examples of peculiar motion of defect clusters under irradiation at temperature of RT and intensity of 1.1×10^{24} e m^{-2} s^{-1}. These kinds of phenomena were frequently observed in all of materials examined in this study.

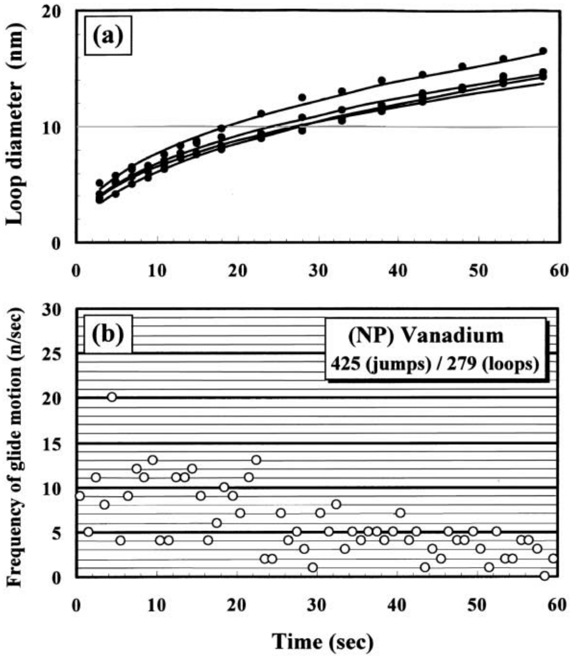

Fig. 3. Correlation between growth behavior (a) and frequency of motion (b) for SIA clusters in (NP) vanadium.

to be the stress interaction between the clusters themselves. Indeed, at high temperatures (around 573 K) where cluster density is quite low, the motion of small clusters was hardly observed. It is therefore difficult, at least at the temperatures examined in this study, to consider that other thermal effects on motion, e.g. electron beam heating and/or heat generation and scattering caused by the mutual recombination of both kinds of defects, are the dominant factors as the driving force.

The observed features of one-dimensional motion of small SIA clusters is probably similar to that of the athermal glide of dislocations under shear stress. Indeed, for the motion of larger loops observed in this study, this dislocation glide mechanism appears reasonable. Some of the observed phenomena, e.g. the frequent repetition of the back and forth motion of small loops, however, are a little difficult to understand in terms of the mechanisms based on conventional dislocation dynamics. It has been suggested that the one-dimensional motion of point defect clusters occurs in the form of groups of crowdions [7,8]. Kiritani et al. have pointed out that this kind of inertial motion in the form of a bundle of crowdions is the origin of the back and forth motion. This bundle crowdion mechanism may explain the phenomena observed in this study, particularly for the motion of smaller loops.

In this study, unfortunately, the motion of small loops was examined by taking averaged images in any case, so that it is difficult to proceed further with the discussion on the crowdion mechanism in terms of fine changes of defect contrast. More detailed observation with high resolution both in time and space will be a future subject in order to clarify the mechanism of the frequent repetition of back and forth motion and appear–disappear motion, as shown in Fig. 2(a).

4.2. Impacts of one-dimensional motion

The results of the measured GD in (NP) vanadium (a) and V–5Ti (b) are shown in Fig. 4. The glide distances have been measured from the projected tracks of the motion to the image plane by assuming all clusters have glided along the $\langle 1\,1\,1 \rangle$ directions and taking into account the angle of the $\langle 1\,1\,1 \rangle$ directions relative to the beam. The frequency and GD in V–5Ti were much lower and shorter, respectively, than those in vanadium, indicating titanium solutes strongly suppress point defect migration and cluster glide motion. In (HP) vanadium, the measured frequency and distance were still higher and longer, respectively, than those in (NP) vanadium.

From our previous study on the density of SIA loops from HVEM irradiation [6] and the results shown in Fig. 4, several parameters of one-dimensional motion were obtained, and these are listed in Table 2. It should be noted that the maximum jump frequency may be in fact filtered at high frequencies by the hardware limitation of the acquisition system. From Table 2 and Fig. 3, it can be concluded that the frequency of the motion has a

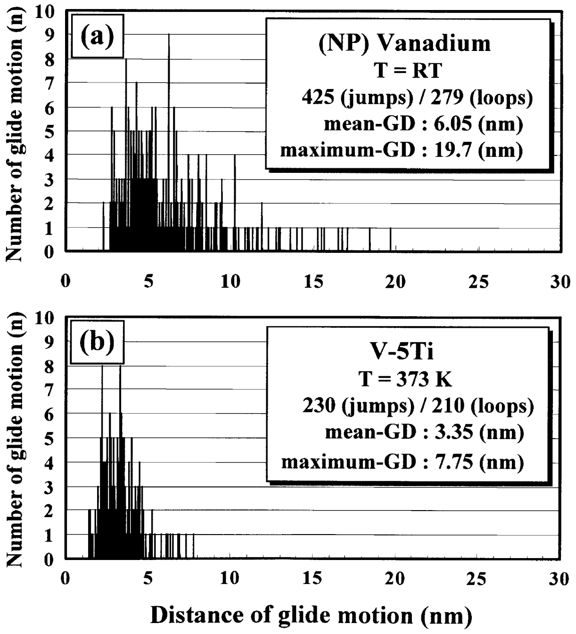

Fig. 4. Distance of glide motion in (NP) vanadium (a) and V–5Ti (b) irradiated at 1.1×10^{24} e m^{-2} s^{-1}.

Table 2
Parameters of the one-dimensional motion obtained in this study

	Glide frequency (s cluster)$^{-1}$ (average for 60 s)	GD (nm (s cluster)$^{-1}$)	Mean distance between two clusters (mean-DD) (nm)	Ratio (mean-GD/ mean-DD) (%)
(NP) vanadium	1.52	1.55×10^{-1}	3.27×10^{1}	18.5
(HP) vanadium	2.72	4.20×10^{-1}	4.20×10^{1}	18.9
V–5Ti	1.10	6.15×10^{-2}	2.54×10^{1}	13.3

strong relation with the growth rate of the clusters. Here again, the driving force for the motion is considered to be the variation of the strain field gradient of the clusters themselves. Indeed, it is worth noting that at high temperatures where cluster formation is quite low, although growth rate is high, glide motion was rarely observed.

On the other hand, the glide distance is strongly related to the distance between clusters. The simple ratio of mean-GD to the mean distance between two neighboring clusters (mean-DD) is shown in the table. In V–5Ti, the ratio is smaller than that in vanadium, indicating strong suppression of the motion by solutes. This tendency, generally, becomes more significant with increasing solute concentration. In more concentrated alloys, e.g. V–4Cr–4Ti and a Fe–15Cr–15Ni model alloy, the motions of the small clusters were rarely observed under the same irradiation conditions as in this study. It can be concluded that the impact of the one-dimensional motion on damage processes in practical alloys are less important than those in pure metals. In other words, one-dimensional motion of the clusters, containing more than several tens of atoms, plays important roles in the damage process in pure metals and under the damage conditions where defects are densely formed to interact with each other.

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