Changes in the Burgers Vector of Perfect Dislocation Loops without Contact with the External Dislocations

K. Arakawa,[1,*] M. Hatanaka,[1] E. Kuramoto,[2] K. Ono,[3] and H. Mori[1]

[1]*Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, 7-1, Mihogaoka, Ibaraki, Osaka 567-0047, Japan*
[2]*Research Institute for Applied Mechanics, Kyushu University, 6-1 Kasuga-koen, Kasuga, Fukuoka 816-8580, Japan*
[3]*Department of Material Science, Shimane University, 1060 Nishikawatsu, Matsue 690-8504, Japan*

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We report the observations of a new type of changing process in the Burgers vector of dislocations by *in situ* transmission electron microscopy. Small interstitial-type perfect dislocation loops in bcc iron with diameters less than approximately 50 nm are transformed from a $1/2\langle 111\rangle$ loop to another $1/2\langle 111\rangle$ one or an energetically unfavorable $\langle 100\rangle$ one; furthermore, a $\langle 100\rangle$ loop is transformed to a $1/2\langle 111\rangle$ one. These transformations occurred on high-energy electron irradiation or simple heating without contact with external dislocations. The origin of these phenomena is discussed.

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The irreversible plastic deformation of crystalline materials is often governed by the generation and motion of linear defects, termed dislocations [1]. Knowledge of the structure and dynamic processes of dislocations in a crystal is important for understanding the origin of the hardness and toughness of a crystal. These defects connect two parts of a crystal that are sheared on a plane with respect to each other by an atomic translation called the Burgers vector.

The Burgers vector of a dislocation is a major factor controlling the displacement field and the strain energy associated with the dislocation, moving direction, mobility of the dislocation, and so on. Dislocations always obey Kirchhoff's law for the Burgers vector [1,2], according to which the total Burgers vector measured in a closed circuit enclosing single or multiple dislocation lines is always conserved even at their nodes. Furthermore, according to this law, the Burgers vector of a dislocation can change if it joins another dislocation or branches out.

In this Letter, we present a new process for inducing a change in the Burgers vector of nanometer-sized interstitial-type perfect dislocation loops—agglomerations of self-interstitial atoms (SIAs) on a habit plane—without contact with external dislocations in bcc Fe, by using *in situ* transmission electron microscopy (TEM).

Pure bcc Fe (99.999%) supplied by Showa Denko, Inc. was used as the specimen. It was rolled into 0.08-mm-thick sheets. These sheets were preannealed at 1120 K for 1 h under a hydrogen atmosphere and electrochemically polished for TEM. We were able to observe the one-dimensional (1D) glide motion of interstitial-type dislocation loops along a direction parallel to their Burgers vector [3,4] and the process of change in their Burgers vector by the following two techniques: (1) high-energy electron irradiation upon which loops were formed due to knock-on displacements of Fe atoms by incident electrons and (2) simple heating. High-energy electron irradiation was performed in a high-voltage electron microscope H-3000 (Hitachi) operated at an acceleration voltage of 1000 kV, and the behavior of the loops was simultaneously observed. The beam fluxes were primarily $1 \times 10^{24}\ e^-/\mathrm{m}^2\,\mathrm{s}$ and $1 \times 10^{23}\ e^-/\mathrm{m}^2\,\mathrm{s}$, and the irradiation temperatures ranged from 110 to 290 K. Simple heating and *in situ* observations were performed using a general-use microscope JEM-2010 (JEOL) at an acceleration voltage of 200 kV to prevent the introduction of additional knock-on displacements into the specimens. The heating was performed following the induction of the loops by high-energy electron irradiation with irradiation doses ranging from 1×10^{26} to $1 \times 10^{27} e^-/\mathrm{m}^2$ at temperatures ranging from 110 to 190 K. The heating temperature ranged from 290 to 900 K. Bright-field imaging was used in the *in situ* TEM observations. The thickness of the areas observed ranged from approximately 100 to 200 nm. The observation axes were approximately $\langle 001\rangle$, $\langle 011\rangle$, and $\langle 111\rangle$. The reflections adopted were $\mathbf{g} = 110$ and $\mathbf{g} = 200$, with a deviation parameter from the exact Bragg condition, s, ranging from approximately 0.04 to 0.1 nm^{-1}. The images were recorded using a charge-coupled device camera with a time resolution of 1/30 s. In our experimental system, we were able to image loops larger with diameters greater than a few nanometers, determine the Burgers vector and the habit plane of loops with diameters greater than approximately 10 nm by using the conventional TEM technique [5], and detect the motion of those loops whose jump distance was greater than approximately 3 nm and whose jump frequency ranged from approximately 0.01 to 30 s^{-1}.

Two types of loops were formed upon irradiation—those with the Burgers vectors of $1/2\langle 111\rangle$ and $\langle 100\rangle$. For sizes ranging from approximately a diameter of 10 to 50 nm, the habit plane of the $1/2\langle 111\rangle$ loops was determined to range from approximately $\{110\}$ to $\{111\}$, while that of the $\langle 100\rangle$ loops was approximately $\{100\}$. These results mostly agree with those of some previous experimental studies on larger loops (see Refs. [6,7]) and those of computer simulation studies on smaller loops, which are based on classical molecular dynamics calculations (MD) (see Refs. [8,9]). It should be noted that both types of loops are perfect dislocation loops without stacking faults.

On simple heating, small $1/2\langle 111\rangle$ loops, whose diameters were less than approximately 20 to 30 nm, began to exhibit 1D thermal motion above approximately 450 K. The moving direction of these loops was parallel to their Burgers vector; this implies that the 1D motion of the $1/2\langle 111\rangle$ loops is a glide motion. On the other hand, small $\langle 100\rangle$ loops exhibited a similar glide motion at temperatures higher than approximately 770 K. Based on these results, the moving direction of the loops can be used to identify their Burgers vector. Furthermore, on irradiation, a 1D motion was easily induced in small $1/2\langle 111\rangle$ loops with diameters less than approximately 50 nm, even at low temperatures at which these loops did not exhibit thermal motion. In contrast, the small $\langle 100\rangle$ loops rarely exhibited 1D motion on irradiation. The motion of these loops will be described in detail elsewhere [10].

The Burgers vector of the mobile $1/2\langle 111\rangle$ loops occasionally changed to that of another $1/2\langle 111\rangle$ loop. As shown in Fig. 1, the projected direction of motion of the marked loop spontaneously changes from $[110]$ to $[1\bar{1}0]$ along an observation axis of approximately $[105]$; this implies that the Burgers vector spontaneously changes from $1/2[11 \pm 1]$ to $1/2[\bar{1}1 \pm 1]$. If the change in the Burgers vector shown here is due to the coalescence of the $1/2[11 \pm 1]$ loop and another loop, the coalescence process requires not a $[100]$ loop or a $[010]$ loop but a loop with the same Burgers vector as that of the $1/2[\bar{1}1 \pm 1]$ loop for a reason shown later. However, such a coalescence was not found. Thus, it was confirmed that, in this case, the Burgers vector changed from $1/2[11 \pm 1]$ to $1/2[\bar{1}1 \pm 1]$ without the coalescence of the loop with an external loop.

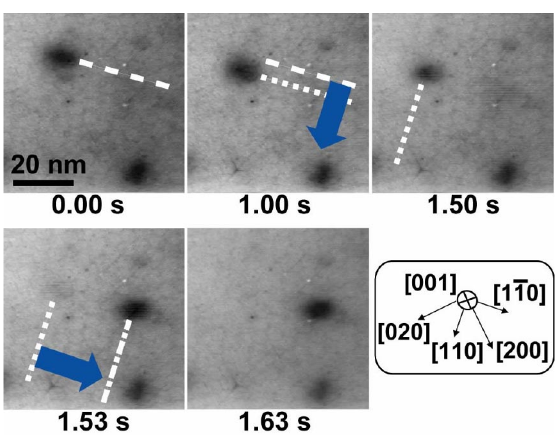

FIG. 1 (color online). Spontaneous change in the projected direction of the motion of an interstitial-type dislocation loop along the observation axis of [1 0 5] upon 1000-keV electron irradiation with a beam flux of $1 \times 10^{24} e^-/\mathrm{m}^2\,\mathrm{s}$ at 293 K. The reflection $\mathbf{g} = 020$ was adopted. The moving direction of the marked loop—indicated by arrows and additional lines—spontaneously changes from $[110]$ to $[1\bar{1}0]$ at 1.50 s. This spontaneous change corresponds to that in the Burgers vector of the loop from $1/2[11 \pm 1]$ to $1/2[\bar{1}1 \pm 1]$.

Other types of the changes in the Burgers vector of loops, such as that from $1/2\langle 111\rangle$ to $\langle 100\rangle$ and its reverse, occasionally occurred. In Fig. 2, the image of the marked loop changes individually. In order to identify the Burgers vector of the marked loop before and after the change, we carried out TEM image simulations of circular loops for various combinations of Burgers vectors ($1/2\langle 111\rangle$ and $\langle 100\rangle$), habit planes ($\{100\}$, $\{110\}$, $\{331\}$, $\{221\}$, and $\{111\}$), and diameters, as follows. A bright-field TEM image was obtained by mapping the square of the wave function of the transmitted electron beam at the bottom surface of the specimen. The wave function was calculated using the solution of the differential equation given by Howie and Whelan [11] under a two-beam condition. Here, the displacement field associated with the dislocation loop was calculated using the solution [12] of the Burgers equation [1] formulated on the basis of the isotropic linear elasticity theory. Figure 3 shows an extract from the images of the loops calculated using TEM image simulations. From a comparison between experimental images (a) or (b), and each generated image (c)–(l) except (h), it can be observed that the loop images are similar to the corresponding experimental image before the change in Figs. 3(c)–3(f), while in Fig. 3(k) the loop reproduces the experimental image after the change. Therefore, the Burgers vector of the loop prior to the change was identified to be $1/2[\bar{1}11]$, and the habit plane was identified in a range from $(1\bar{1}0)$ to $(1\bar{1}\,\bar{1})$. The loop after the change was identified to be $[010](0\bar{1}0)$. Here, the other Burgers vectors and habit planes are clearly ruled out. The slight distortion of the experimental image of the loop before the change is attributed to the bending of its habit plane and the deviation of the loop shape from a perfect circle. Marian *et al.* proposed another mechanism for the formation of $\langle 100\rangle$ loops, which were energetically unfavorable as compared with the $1/2\langle 111\rangle$ ones, using MD calculations [13]. They concluded that the $\langle 100\rangle$ loops were formed due to a

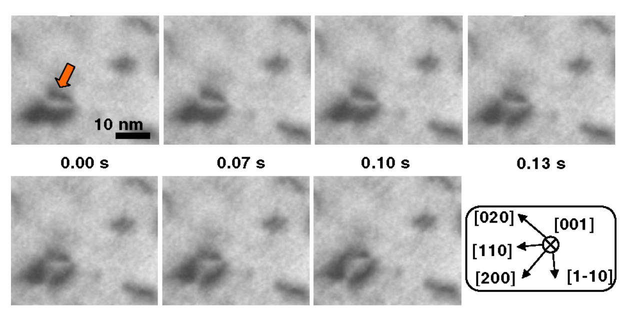

FIG. 2 (color online). Spontaneous change in the TEM image of an interstitial-type dislocation loop upon heating at 570 K. The beam incident direction was $[\bar{1}05]$. The reflection $\mathbf{g} = 0\bar{2}0$ and deviation parameter $s = 0.1\ \mathrm{nm}^{-1}$ were adopted. The foil thickness was approximately 100 nm.

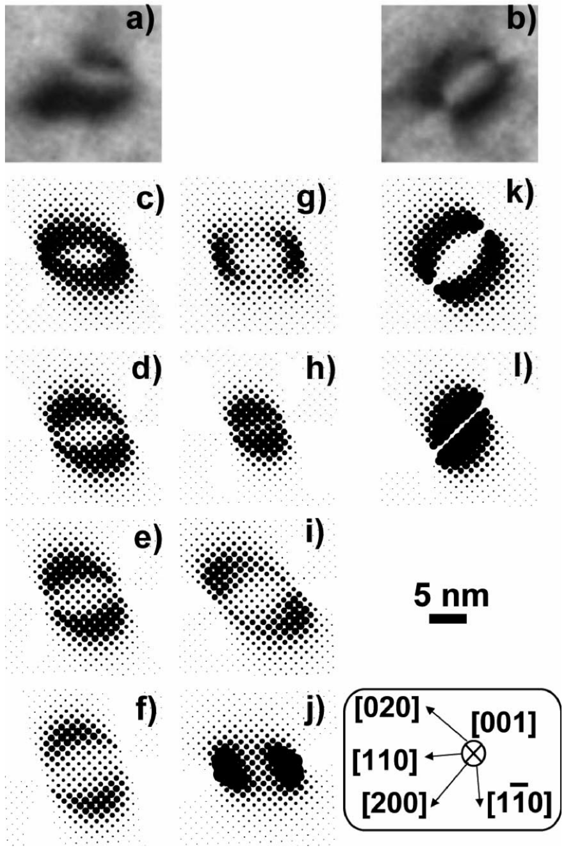

FIG. 3. Comparison among the TEM images of the dislocation loop before and after the change shown in Fig. 2 and generated images of the circular loops of various types. Panels (a) and (b) are the experimental images of the loop: (a) image before the change; (b) image after the change. Panels (c)–(l) are generated images. The Burgers vector, habit plane, and diameter of the loops for the calculations are as follows: (c) $1/2[\bar{1}11](1\bar{1}0)$, 12 nm; (d) $1/2[\bar{1}11](3\bar{3}\,\bar{1})$, 11 nm; (e) $1/2[\bar{1}11](2\bar{2}\,\bar{1})$, 11 nm; (f) $1/2[\bar{1}11](1\bar{1}\,\bar{1})$, 11 nm; (g) $1/2[\bar{1}1\bar{1}](1\bar{1}0)$, 12 nm; (h) $1/2[\bar{1}11](3\bar{3}\,\bar{1})$, 8 nm; (i) $[010](2\bar{2}\,\bar{1})$, 12 nm; (j) $1/2[\bar{1}1\bar{1}](1\bar{1}1)$, 11 nm; (k) $[010](0\bar{1}0)$, 10 nm; (l) $1/2[\bar{1}11](0\bar{1}0)$, 10 nm. In the calculation, the deviation of the observation axis from the [001] pole and the absorption of electrons were taken into account. The central position of the loop was located at the center of depth of the specimen. The diameters of the loops in (k) and (l) satisfy a condition that the major length of their coffee-bean images is equal to that of the experimental image of the loop after the change shown in (b). The diameters of the loops shown in (c)–(j) except (h) were set so that the number of SIAs contained within these loops was equal to that in the loop shown in (k). The diameter of the loop shown in (h) was set so that the number of SIAs in the loop was the half of that in the loop shown in (k).

conventional dislocation reaction during the coalescence of the mobile $1/2[\bar{1}11]$ and $1/2[11\bar{1}]$ loops: $1/2[\bar{1}11] + 1/2[11\bar{1}] \rightarrow [010]$. In the case of Fig. 2, the $1/2[11\bar{1}]$ loop, which undergoes the above reaction with the $1/2[\bar{1}11]$ loop, can be observed, if present, under the adopted reflection [5]; however, it was not observed. In addition, this mode of formation of a $\langle 100\rangle$ loop requires a coalescence between two $1/2\langle 111\rangle$ loops that are almost similar in size, as described below. However, the $1/2[\bar{1}11]$ loop, which accounts for half the total number of SIAs present in the loop after the change, as shown in Fig. 3(k), does not reproduce the experimental image of the loop before the change: this is shown in Fig. 3(h). Thus, it was confirmed that, at least in this case, the Burgers vector of a loop changed from $1/2[\bar{1}11]$ to [010] without coalescing with another loop.

These types of loop transformation occurred only when the loops were still small and their diameters were less than a maximum of approximately 50 nm. It should be noted that the $1/2\langle 111\rangle$ loops without stacking faults transformed, although the energy of the system appeared to remain constant or to increase due to this change. Hence, these changing processes of the perfect dislocation loops are considerably different from the other known case of spontaneous change in the Burgers vector of partial dislocation loops, wherein the inner areas are occupied by stacking faults, in face-centered cubic, hexagonal closed packing, and diamond-cubic lattices to perfect dislocation loops, resulting in unfaulting [1]. In unfaulting, the change in the Burgers vector is fundamentally related to the high value of the energy of large stacking faults. Therefore, the transformation of the partial loops occurs only when they become large.

The change in the Burgers vector of a prismatic loop without coalescing with external dislocations can be expressed as the nucleation and propagation of a proper shear loop, in which the habit plane is identical to that of the original prismatic loop and only the shear component exists in the Burgers vector inside the prismatic loop. For example, the process of the change in the Burgers vector from $1/2[\bar{1}11]$ to [010] results from the nucleation of a $1/2[11\bar{1}]$ shear loop and its propagation through the original $1/2[\bar{1}11]$ loop, i.e., $1/2[\bar{1}11] + 1/2[11\bar{1}] \rightarrow [010]$. If the vector $1/2[11\bar{1}]$ does not lie on the original habit plane, this process must accompany the change in the habit plane of the $1/2[\bar{1}11]$ loop to a plane such as the $(1\bar{1}0)$ plane that contains the $1/2[11\bar{1}]$ vector.

The nucleation and propagation of the shear loops is expected to occur by thermal fluctuation at high temperatures when the original prismatic loops are extremely small. Recent *ab initio* calculations have shown that the energy difference between a $\langle 100\rangle$ dumbbell and a $\langle 111\rangle$ crowdion is only 0.3 eV: this is not a very high value [14]. This suggests that the energy difference between small $\langle 100\rangle$ loops and $1/2\langle 111\rangle$ loops is also not very high, thus indicating the possibility of such a thermal transformation.

Nucleation and propagation of a shear loop will also occur when external loops in the vicinity of a loop act as a source of considerable shear stress. On high-energy electron irradiation or simple heating, loops can appear or disappear near a marked loop due to the agglomeration of SIAs and their glide motion; further, loops near a marked loop can extend or shrink by the absorption of SIAs, small loops, or vacancies. In such a case, the shear stress at the position of the marked loop varies. If the stress accidentally reaches a critical value, nucleation and propagation of a proper shear loop will occur, resulting in a change in the Burgers vector. In the case of Fig. 2, the shear stress applied onto the habit plane of a transformed loop in the direction of the Burgers vector of the nucleated shear loop by the nearest loop was roughly estimated to be a maximum of approximately 0.1 GPa using the isotropic linear elasticity theory [12]. Apparently, the best method for obtaining the maximum shear stress is to coalesce the external loop that has the same Burgers vector as that of the proper shear loop with the marked loop. However, according to our observations, even when a $1/2[\bar{1}11]$ loop and a $1/2[11\bar{1}]$ loop coalesced, the $[010]$ loop was not formed, and one loop absorbed the other. Further, such an absorption occurred in the case of the coalescence between a $1/2\langle 111\rangle$ loop and a $\langle 100\rangle$ loop. In accordance with Marian *et al.* [13], unless both the $1/2\langle 111\rangle$ loops including almost the same number of self-interstitial atoms coalesce, the $[010]$ loop is probably not formed. A special situation of this type will not occur frequently, at least not in the case of TEM-observable loops with diameters greater than a few nanometers. The interactions between two loops will be described in detail elsewhere [10].

In conclusion, we have presented, for the first time, the process of change in the Burgers vector of perfect dislocations without contact with external dislocations by using *in situ* TEM. The individual transformation of a small interstitial-type perfect dislocation loop with the Burgers vector of $1/2\langle 111\rangle$ in Fe, whose diameter was less than approximately 50 nm, to another $1/2\langle 111\rangle$ or $\langle 100\rangle$ loop has been presented; further, the reverse process occurred on high-energy electron irradiation or simple heating. This process is attributed to the nucleation and propagation of a shear loop on a prismatic loop due to thermal fluctuation for extremely small loops at high temperatures and the application of great shear stress by the loops near the marked loop.

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*Corresponding author.
Electronic address: arakawak@uhvem.osaka-u.ac.jp

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