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Direct observation of the coalescence process between nanoscale dislocation loops with different Burgers vectors

K. Arakawa [a,b,*], T. Amino [a], H. Mori [a]

[a] Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, 7-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan
[b] CREST, JST, 5 Sanbancho, Chiyoda-ku, Tokyo 102-0075, Japan

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Abstract

The dynamic behavior of nanoscale prismatic dislocation loops can significantly affect the microstructural variation in crystalline materials upon processes such as plastic deformation and high-energy particle irradiation. Using in situ transmission electron microscopy, this study experimentally demonstrates a reaction which follows the collision between two loops with different Burgers vectors in α-iron. Even after the formation of the junction, the reaction progresses further, unlike conventional reactions between dislocations of macroscopic length, and the larger loop finally absorbs the smaller one.

Keywords: Dislocations; In situ transmission electron microscopy; Ferritic steels

1. Introduction

Nanoscale or subnanoscale prismatic dislocation loops [1]—agglomerations of self-interstitial atoms (SIA) or vacancies on a habit plane—can be formed upon various processes, such as quenching [2], plastic deformation [3] and energetic particle irradiation [4]. It has been shown that interstitial-type $1/2\langle 1\,1\,1\rangle$ loops in body-centered cubic iron α-Fe, among two types of loops with $\mathbf{b} = 1/2\langle 1\,1\,1\rangle$ and $\mathbf{b} = \langle 1\,0\,0\rangle$ [5–9], can undergo one-dimensional (1D) glide diffusion in their **b** direction even in the absence of stresses, by molecular dynamics calculations (MD) [10–14], theories [15–18] and an in situ transmission electron microscopy (TEM) experiment [19]. The dynamic behavior of individual loops of this type is interesting from the viewpoint of dislocation theory, and it is believed to be strongly connected to the degradation processes that affect radiation-resistant materials used in nuclear-fission and fusion devices [20–22].

As well as the behavior of individual loops, interactions between two loops and among multiple loops are considered to play key roles in the microstructural variation in the processes where loops are formed. Recently, Dudarev et al. [23] showed experimentally that two adjacent 1/2[1 1 1] loops undergo correlated 1D diffusion via the elastic interaction between them. They developed an algorithm for simulating the correlated 1D diffusion, based on Langevin equations. When two adjacent loops with the same Burgers vector grow and collide side by side, they coalesce and a single loop is formed [24].

However, it remains unclear what phenomenon takes place when two loops with different Burgers vectors collide. Hereafter, this paper primarily considers what happens when a 1/2[1 1 1] loop (loop A) and a $1/2[1\,1\,\bar{1}]$ loop (loop B) collide side by side via their 1D motion.

When a dislocation with $\mathbf{b} = \mathbf{b}_1$ encounters another dislocation with $\mathbf{b} = \mathbf{b}_2$, a junction with $\mathbf{b} = \mathbf{b}_1 + \mathbf{b}_2$ may form. This is compliant with the conservation law applicable to the Burgers vector, called Kirchhoff's law [1]. According to the conventional view on the reactions between dislocations with macroscopic length, the reaction is considered to be finalized at this point. In α-Fe that is undergoing plastic

* Corresponding author at: Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, 7-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan. Tel.: +81 668797941; fax: +81 668797942.
E-mail address: arakawak@uhvem.osaka-u.ac.jp (K. Arakawa).

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deformation, stable junctions with $\mathbf{b} = \langle 1\,0\,0\rangle$ are often observed in addition to the most elementary dislocations with $\mathbf{b} = 1/2\langle 1\,1\,1\rangle$ [25]. This occurs because of the collision between two $1/2\langle 1\,1\,1\rangle$ dislocations, as expressed by the following reaction:

$$1/2[1\,1\,1] + 1/2[\bar{1}\,\bar{1}\,1] = [0\,0\,1] \quad (1)$$

Even in the case of the collision between loops A and B, the reaction can be anticipated to be finalized just after the formation of a [0 0 1] junction. It is noted that the stabilization of a [0 0 1] junction yields a sessile dislocation–loop complex. In contrast, Osetsky et al. [26], Marian et al. [7] and Terentyev et al. [27] showed, by using MD on $1/2\langle 1\,1\,1\rangle$ loops less than a few nanometers in diameter, that a larger loop can absorb the smaller one when loop A and loop B collide. In addition, Masters [5] and Marian et al. [7] proposed that a [0 0 1] loop can be formed in accordance with a dislocation reaction similar to the one shown in Eq. (1) when two loops with similar sizes collide [7]. Therefore, according to these former studies, there might be at least three candidates for the phenomena that take place following collision between loops A and B, as follows: (i) a junction is formed and the reaction is completed at this point; (ii) one loop absorbs the other; and (iii) a [0 0 1] loop is formed in another type of coalescence. The latter two reactions appear to contradict the conventional view on dislocation reactions. The present paper clarifies experimentally, using in situ TEM, what happens when loops A and B collide.

2. Experimental procedure

Polycrystalline pure α-Fe (99.998%) was used as the specimen. It was rolled into 0.08-mm-thick sheets. These sheets were pre-annealed at 1073 K for 2 h under a hydrogen atmosphere and electrochemically polished for TEM. Crystalline grains larger than ~100 μm in diameter whose surfaces were oriented approximately in the $(1\,\bar{1}\,0)$ plane were selected for the experiment. Thus, the **b** values of loop A and loop B were almost parallel to the surfaces; therefore, the force applied to the loops from the surfaces in the direction of the 1D motion was negligible.

Nanoscale interstitial-type $1/2\langle 1\,1\,1\rangle$ loops were produced via the agglomeration of SIA generated by the knock-on displacement of host atoms upon 2000 keV electron irradiation in a high-voltage electron microscope H-3000 (Hitachi). The beam flux was $1 \times 10^{23}\ e^-\ (m^2\ s)^{-1}$, irradiation dose was $3 \times 10^{25}\ e^-\ m^{-2}$, and the irradiation temperature ranged from 110 K to 190 K.

Simple heating and in situ observations of the behavior of $1/2\langle 1\,1\,1\rangle$ loops undergoing 1D motion via thermal activation [19] were performed using a conventional microscope H-800 (Hitachi), at an acceleration voltage of 200 kV in order to prevent the introduction of additional knock-on displacements into the specimens. The beam diameter was ~8 μm, and the beam flux was $\sim 5 \times 10^{20}\ e^-\ (m^2\ s)^{-1}$. Then, the effect of thermal stress due to beam heating on the loop motion was negligible. (See the supporting online material in Ref. [19].) The heating temperature ranged from 290 K to 800 K. Bright-field imaging was used in the in situ TEM observations. The thickness of the areas observed was less than ~100 nm. The observation axes were approximately $[1\,\bar{1}\,0]$. The reflections adopted were $\mathbf{g} = 1\,1\,0$ and $\mathbf{g} = 0\,0\,2$, with a deviation parameter from the exact Bragg condition s ranging from ~0.02 to 0.06 nm^{-1}. The images were recorded using a silicon intensifier target tube camera with a time resolution of 1/30 s. The experimental system was able to image loops greater than a few nanometers in diameter. The **b** value of each loop could be determined from the direction of its 1D motion. In addition, the **b** value of loops larger than ~10 nm in diameter could be verified using image simulations of loops. In the simulations, the displacement fields around the loops were approximated by the isotropic elastic fields of perfectly circular loops [1,28], and the wave functions at the bottom surface of the specimen were calculated by the two-beam column approximation [29].

3. Experimental results

Fig. 1 displays a series of sequential micrographs showing the collision between two loops and the subsequent reaction at 660 K. From the directions of movement of the two loops before the collision, the **b** values of loop A and loop B are determined as $\mathbf{b}_A = 1/2[1\,1\,1]$ and $\mathbf{b}_B = 1/2[1\,1\,\bar{1}]$, respectively. The validity of these values is also verified by the image simulation results attached to in the figure. At 1.40 s, loops collide with each other side by side and form a junction. Even after the formation of the junction, the reaction is not finalized and progresses further, unlike the conventional reaction between dislocations with macroscopic length. The junction moves toward the far end of loop A, and a single loop is finally formed at 5.64 s. The **b** value of the final single loop is identical to that of the original loop B, as determined by its direction of movement and the image simulation results. This shows that loop B absorbed loop A.

Fig. 2 shows a schematic view of the process displayed in Fig. 1. In Fig. 2, for simplicity, the shape of the loops is approximated as squares or rectangles. Here, the segments parallel to the junction, which is approximately parallel to the direction of the view, are noticed. The directions of all these segments were found to be identical, so that a group of these segments could be regarded as multi-poles. The directions were set to be from the near to the far side. Then, the direction of the **b** value of each segment was determined by the FS/RH convention [1]. From the **b** values of the two segments at which loops are colliding, the **b** value of the junction formed by the collision, $\mathbf{b}_j$, is determined as $\mathbf{b}_j = \mathbf{b}_A + (-\mathbf{b}_B) = [0\,0\,1]$. The absorption of loop A by loop B is completed through the reaction between the junction and the end of loop A: $\mathbf{b}_j + (-\mathbf{b}_A) = -\mathbf{b}_B$. The loop complex itself which involves the junction is essentially sessile; however, it is converted to a single glissile loop by the absorption reaction.