Formation Mechanism of Dislocation Walls during Cyclic Deformation in an Fe–Si Alloy

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Low-cycle fatigue tests of a polycrystalline Fe–3 mass%Si alloy were performed at room temperature under a constant total strain amplitude of $1 \times 10^{-2}$. Dislocation structures were observed by high-voltage scanning transmission electron microscopy. The development of dislocation walls parallel to $\{1\ 0\ 0\}$ started during the first few tens cycles of fatigue. The activation of a set of double slip systems, $(2\ 1\ 1)\{1\ 1\ 1\}$ and $(1\ 1\ 2)\{1\ 1\ 1\}$, contributed to the formation of $(1\ 0\ 0)$ walls. The $(1\ 0\ 0)$ walls lie in the directions bisecting the angles between the Burgers vectors of the two active dislocations of the double slip systems.

KEY WORDS: fatigue; cyclic deformation; dislocation structure; wall structure; labyrinth structure; iron; steel; silicon; high-voltage scanning transmission electron microscopy.

1. Introduction

The characteristic dislocation structures are developed in metals during cyclic deformation. Some studies have implied that the dislocation structures affect the fatigue strength. Yokoi et al.1) reported that the formation of a dislocation cell structure is suppressed in an Fe–1.5 mass%Cu alloy showing a higher fatigue strength than Fe. Ushioda et al.3) also found that dislocation cells are hard to form, and dislocation veins and dislocation walls are preferentially formed in Fe alloys containing 0.5–1 mass% of Si, which are known to have a higher fatigue strength than Fe. Shuto and Yokoi3) compared the fatigue limit and the dislocation structures of an Fe–0.5 mass%Si alloy and an Fe–2 mass%Ni alloy. These results implied that the fatigue limit of Fe may possibly be increased if the development of dislocation structures can be controlled, and the cell structure formation is prevented by adding secondary elements.

The formation mechanisms of the dislocation structures must be elucidated to control the dislocation structures. In face centered cubic (fcc) metals, even though there have been ongoing discussions,4) the formation mechanisms of the vein, wall, and cell structures were summarized.5,6) By contrast, the formation mechanisms of the dislocation structures in body centered cubic (bcc) metals have not yet been established because of the complexity and variation of its slip system. For example, the crystallographic orientation and the formation mechanism of the dislocation walls in a “labyrinth structure” are still not completely understood.

The labyrinth structure in Fe–based bcc alloys has been reported in several studies. Fielding and Stobbs5) found the labyrinth structure in a polycrystalline Fe–25 mass%Cr alloy. They concluded that both condensed dislocation walls are the $(1\ 1\ 0)$ walls. Turrene et al.6) also found the labyrinth structure in a polycrystalline low–carbon steel, and concluded that the two types of dislocation walls are mutually perpendicular (i.e., $(3\ 1\ 1)$ and $(0\ 1\ 1)$ walls). They pointed out that the dislocation wall planes were incorrectly determined by Fielding and Stobbs,5) and correctly identified them as the $(1\ 0\ 2)$ and $(0\ 1\ 0)$ walls, respectively. Tjong et al.10) and Roven and Nes10) found the labyrinth structure in an Fe–25 mass%Cr alloy and low–carbon steel, respectively; however, they did not analyze the crystallographic orientation of the dislocation walls. Petrenec et al.11) also

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reported a similar dislocation structure in a polycrystalline Fe–25 mass%Cr alloy. They concluded that the dislocation walls lie in the directions bisecting the angles between the directions of the screw dislocations in the two slip systems with the highest Schmidt factors. However, the dislocation wall orientation was unclear. Shuto et al.\textsuperscript{11)} inferred that the dislocation walls of the labyrinth structure are developed along two mutually perpendicular orientations, namely (110) and (001), in a polycrystalline Fe–1 mass%Si alloy. Overviewing the abovementioned studies, the geometrical configuration of the dislocation walls in the labyrinth structure is still controversial and unclear in ferrous alloys.

The formation mechanism of the dislocation walls in the labyrinth structure has not yet been well established. In fcc metals, two models have been proposed to explain the dislocation wall formation. The first model is the double pseudo–polygonization (DPP) model proposed by Dickson et al.\textsuperscript{8)} In the DPP model, three regular stacking networks of dislocation dipole loops can be constructed by rectangular–shaped dislocation loops with long edge segments. Two of these three networks are preferentially selected in terms of the “sweeping angle.” This model was extended to bcc metals by Turenne et al.\textsuperscript{9)} The other model was proposed by Li et al.\textsuperscript{5)} who considered a dislocation reaction between the primary and secondary slip systems. In this model, the dislocation walls lay perpendicular to the sum of two Burgers vectors. In other words, the dislocation walls are perpendicular to the directions of the sum of the Burgers vectors for the active dislocations in the two slip systems.\textsuperscript{2)} To investigate the validity of these two models in the bcc metals, the dislocation wall orientation must be revealed, and the active slip systems must be analyzed to contribute to the understanding of the dislocation wall formation. Therefore, the present study focuses on investigating the dislocation wall orientation in the labyrinth structure and identifying the active slip systems in an Fe–3 mass%Si alloy.

The dislocation structures in the fatigued specimens are clearly observed through scanning transmission electron microscopy (STEM) instead of conventional TEM because of STEM’s advantages, such as reduced bend contours, thickness fringes, and dynamical contrast effects. In addition, the usage of high–voltage electron microscopy enables the observation of sufficiently thick samples (approximately 500 nm for Fe). Therefore, high–voltage STEM (HV–STEM) was adopted herein to observe the dislocation microstructures in cyclically deformed specimens.

### 2. Experimental Procedure

The dislocation wall is formed in an Fe–Si alloy rather than in pure Fe.\textsuperscript{2)} Polycrystalline Fe–Si ingot was prepared in a vacuum induction furnace. Table 1 presents the chemical composition of the ingot. The ingot was forged at 900°C to reduce the shrinkage cavity. Ferrite grain was obtained with grain sizes of 2–20 mm. The ingot was cut into specimens for the fatigue tests with gage dimensions of 4 × 6 × 10 mm\textsuperscript{3}. The crystallographic orientation of each grain was measured prior to the fatigue tests using the electron backscattered diffraction method in an FE–SEM (JEOL JSM–7001F).

Fully reversed tension–compression fatigue tests were performed at room temperature under a constant total strain amplitude of 1 × 10\textsuperscript{−2} using a servo–hydraulic testing machine (MTS 810 Material Test System). The cyclic frequency was obtained as 0.05 Hz using a triangular command signal. The strain was measured with an extensometer mounted directly on the gage section. The stress response was monitored, and the hysteresis loops were recorded on a digital data logger. The fatigue test was interrupted at 30 cycles to observe the developing process of a dislocation structure.

The fatigued specimen was sliced into 3 mm disks parallel to the specimen surface. The crystallographic orientations of the disk planes were selected perpendicular to [001] and [11T]. These disks were ground down to a 0.16 mm thickness with silicon–carbide paper. Thin foils were prepared by electrolytic polishing on a twin–jet polisher (Struers Tenupol–5) in a solution of 10% perchloric acid and 90% methanol at 223 K. Microstructural observations were performed in a bright field mode on an HV–STEM (JEOL JEM–1000K RS) at an acceleration voltage of 1 000 kV. The electron probe diameter was 2 nm.

### 3. Results

#### 3.1. Stress–Strain Response

Figure 1 illustrates the change in the stress amplitude during the fatigue test. The stress amplitude was larger than the yield stress. Fatigue fracture occurred at 50 cycles after monotonic cyclic hardening. Figure 2 presents the plastic strain amplitude measured by a strain gage during the fatigue deformation. Although the plastic strain amplitude slightly decreased from 8.4 × 10\textsuperscript{−3} to 7.9 × 10\textsuperscript{−3}, it accounted for approximately 80% of the applied total strain amplitude.

![Fig. 1. Cyclic hardening curve of a specimen fatigued under a constant total strain amplitude of 1 × 10\textsuperscript{−2}. Monotonic hardening was observed until fracture at 50 cycles.](image-url)
3.2. Dislocation Structure

Figure 3 presents an HV‒STEM bright field image of the dislocation microstructure in a fatigued specimen sliced perpendicular to the [001] direction. The image was taken in a condition such that the incident beam direction is exactly parallel to [001]; hence, all dislocations existing in the foil were visible as a black contrast. Although typical labyrinth structure was not yet developed, unidirectional dislocation walls along the (110) plane were formed. The dislocations that lie parallel to [1 10] were distinguished in the channels between the walls. In our previous study, the dislocation walls developed by the cyclic deformation with a constant total strain amplitude of $5 \times 10^{-3}$ in the Fe–Si alloy were parallel to (110). If the crystallographic symmetry of the body centered cubic is considered, the dislocation walls in Fig. 3 are equivalent to those observed in the previous study. In other words, the {110} dislocation walls are frequently formed by the cyclic deformation of the Fe–Si alloy. Although the [001] foil is adequate for observing the dislocation walls, it is not appropriate for determining the Burgers vectors of the dislocations because two of the four projections of the (111) directions are overlapped. Therefore, the [111] foil was taken from a grain next to the grain observed in Fig. 3 to determine the Burgers vectors.

Figure 4 shows an HV‒STEM bright field image of the [111] foil. The incident beam direction was parallel to [111]. Two types of dislocations were observed: 1) dislocation lines extended from the lower left to the upper right and 2) ones from the lower right to the upper left. In addition, the dislocation walls were nucleated along the (110) plane in areas enclosed by white ellipses in Fig. 4. The orientation of these dislocation walls is crystallographically equivalent to that observed in Fig. 3. We conducted a conventional Burgers vector analysis based on the invisibility criterion: the dislocations are invisible at $g \cdot b = 0$, where $g$ is the diffraction vector, and $b$ is the Burgers vector of the dislocation. Figures 5(a) and 5(b) presents the HV‒STEM images taken under the condition of $g = 0[\overline{1}10]$ and $g = 101$, respectively, in the same area of Fig. 4. The dislocation lines elongated from the lower right to the upper left were visible in Fig. 5(a) but invisible in Fig. 5(b). Conversely, the dislocations along the upper right to the lower left were visible in Fig. 5(b) but invisible in Fig. 5(a). Therefore, the Burgers vector of the visible dislocations in Fig. 5(a) can be determined as $b_1 = a/2[\overline{1}1\overline{1}]$. By contrast, the Burgers vector of the visible dislocations in Fig. 5(b) is $b_2 = a/2[11\overline{1}]$. Considering the elongated directions of the two types of dislocations, the Burgers vector of the dislocations in Fig. 4 is $b_1 = a/2[\overline{1}1\overline{1}]$.
locations, both dislocations seemed to be a screw type, while some had a bowed-out shape. Furthermore, referring to the (11T) stereographic projection of the bcc crystal illustrated in Fig. 5(c), all the possible slip plane traces were drawn in Figs. 5(a) and 5(b). A stereo pair of images was taken in the same area to identify the slip plane parallel to the above-mentioned screw dislocations. Figure 6 presents a pair of STEM stereo micrographs taken under the same diffraction vector of $g = \overline{1}10$. The two images were inclined by 14° with respect to the diffraction vector direction. From a series of observations, two types of slip systems were then identified as $(211)[1\overline{1}1]$ for the $b_1$ dislocation and $(1\overline{1}\overline{2})[\overline{1}1\overline{1}]$ for the $b_2$ dislocation. This result is consistent with the trace analysis in Fig. 5. The present result indicates that the {112} $\langle 111 \rangle$ slip system is easily activated, but we cannot rule out the possibility of the {110} $\langle 111 \rangle$ slip system’s activation.

Fig. 5. (a and b) HV–STEM images taken under the condition of $g = 0\overline{1}1$ and $g = 101$, respectively, in the same area of Fig. 4 and (c) the (11T) stereographic projection of the bcc crystal. With the conventional Burgers vector analysis, the Burgers vector of the visible dislocations in (a) and (b) can be identified as $b_1 = a/2[1\overline{1}1]$ and $b_2 = a/2[1\overline{1}\overline{1}]$, respectively. The projection of $b_1$ and $b_2$ are shown in (a) and (b), respectively. All possible slip plane traces were drawn in Figs. 5(a) and 5(b) referring to 5(c).

Fig. 6. Stereo pair of the HV–STEM micrographs of the [11T] foil taken under the diffraction condition of $g = \overline{1}10$. The two images are inclined by 14° with respect to the diffraction vector direction. From a series of observations, two types of slip systems were identified as $(211)[1\overline{1}1]$ for the $b_1$ dislocation and $(1\overline{1}\overline{2})[\overline{1}1\overline{1}]$ for the $b_2$ dislocation.
4. Discussion

As mentioned in Section 1, there have been two models to describe the dislocation wall formation, namely the DPP\(^4,13\) and Li\(^5\) models. Here we try to adopt the two models to appreciate the reliability. First, the DPP model\(^4,13\) is considered. If the active slip systems are assumed to be \((211)[\bar{1} \bar{T} \bar{T}]\) and \((1\bar{T}2)[\bar{T} \bar{T}1]\), the dislocation walls are predicted to be parallel to \((101)\) and \((1\bar{T}0)\) from the DPP model. The predicted wall traces are drawn as white lines in Fig. 7. These traces are not parallel to the actual dislocation wall encircled in Fig. 7. Therefore, the DPP model is not applicable to the present case where the double slip of \((211)\{111\}\) is activated in the bcc alloy. Second, the Li model\(^5\) is discussed. The Burgers vectors of the dislocations on the active slip systems were identified as \(a/2[\bar{1} \bar{T} \bar{T}]\) and \(a/2[\bar{T} \bar{T}1]\) herein; thus, a dislocation wall normal can be estimated by the Li model as \([001]\) and \([1 \bar{T}0]\) through the following equations.

\[
a/2[\bar{1} \bar{T} \bar{T}]+a/2[\bar{T} \bar{T}1]=−a[001] \tag{1}\\
a/2[\bar{T} \bar{T}1]−a/2[\bar{1} \bar{T} \bar{T}]=a[1 \bar{T}0] \tag{2}
\]

Figure 7 depicts the predicted wall traces of these dislocation walls: \((001)\) and \((1\bar{T}0)\). The predicted \((1\bar{T}0)\) trace is parallel to the encircled dislocation wall in Fig. 7; therefore, the Li model is applicable to explain the dislocation wall orientation in the Fe–Si alloy with the bcc structure.

Note that the Li model supposes that the dislocation reactions of the primary and secondary slip systems occur as expressed in Eqs. (1) and (2) when the dislocation walls are nucleated. Some candidates for the reactions are found in the fcc metal; thus, the sum of the self-energy of the dislocations remains constant. This reaction in the fcc metals is known as “Hirth lock”\(^14,15\). Contrarily, the dislocation reaction such as Eq. (2) is hard to occur in bcc metals, because the sum of the self-energy of the dislocations in Eq. (2) increased if the reaction was performed. Therefore, strictly speaking, the \((1\bar{T}0)\) wall is not able to be formed by the real reaction expressed by Eq. (2). Nevertheless, the Li model seemed to be valid in terms of understanding the crystallographic orientation relationship between the active dislocations and the walls.

In the present study, the active slip system was not \((110)\{111\}\), but it is \((112)\{111\)\). Takeuchi et al.\(^16\) estimated the critical resolved shear stresses (CRSSs) of these slip systems in the bcc Fe alloys containing 0–4.4 mass% of Si. They reported that the ratio of CRSS of the \((112)\{111\) slip system to that of the \((110)\{111\) slip system is 1.1 when Si content is less than 3.2 mass%. This result implies that not only the \((110)\{111\) slip system but also the \((112)\{111\) slip system can be activated in the Fe–3 mass%Si alloy herein.

5. Conclusions

The present study performed low–cycle fatigue tests of a polycrystalline Fe–3 mass%Si alloy were performed at room temperature under a constant total strain amplitude of \(1 \times 10^{-2}\). The dislocation structures were also observed by HV–STEM. The following conclusions were drawn:

- (1) Dislocation walls parallel to \((1\bar{T}0)\) started to develop during the first few tens cycles of fatigue.
- (2) The \((1\bar{T}0)\) walls were formed by the activation of the double slip systems: \((211)[\bar{1} \bar{T} \bar{T}]\) and \((1\bar{T}2)[\bar{T} \bar{T}1]\).
- (3) The \((1\bar{T}0)\) walls lie in the directions bisecting the angles between the Burgers vectors of \(±a/2[\bar{1} \bar{T} \bar{T}]\) and \(±a/2[\bar{T} \bar{T}1]\) for the two active slip systems.

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