Evolution of Dislocation Structure and Fatigue Crack Behavior in Fe–Si Alloys during Cyclic Bending Test

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The evolution of dislocation structures was investigated by TEM in Fe–Si alloys with 0, 0.5 and 1.0 mass% Si during a cyclic bending test in conjunction with fatigue crack behavior. The addition of Si increased the fatigue strength. The evolution of dislocation structures was significantly influenced by the Si addition. Namely, in the steel without Si the dislocation cell structure develops, whereas in the steel with 1 mass% Si the vein structure develops, which is considered to lead to increased fatigue strength. The dislocation cell structure observed in the steel without Si is postulated to be caused by the easy cross slip of dislocations during cyclic deformation, whereas the vein structure that developed in the steels with Si is inferred to be caused by the difficulty in cross slip due to the decrease in stacking fault energy. Furthermore, the Si added steel shows a characteristic structure in a manner such that the dislocations are free in approximately 0.5 μm zones along grain boundaries. The examinations of the fatigue fracture surface revealed that transgranular fracture takes place in steel without Si, whereas in steel with 1 mass% Si many intergranular cracks were observed just beneath the top surface. The intergranular cracks in the 1 mass% Si steel were thought to be caused by the fact that a) strains are dispersed within grains owing to the vein structure and b) micro cracks are initiated and propagated along grain boundaries due to the dislocation free zones.

KEY WORDS: Fe–Si alloy; fatigue; fracture; dislocation structure; cell; vein; ladder; labyrinth; grain boundary; stacking fault.

1. Introduction

Efficiency and safety are required for automobiles, ships and so forth, while still achieving weight reduction. Increasing the strength of steels is a very effective means of achieving both the efficiency and safety of such structures.1,2) Many studies have been conducted on improving fatigue characteristics through increasing the strength.1–5) For example, high-strength steel sheet with a 590 MPa dual phase structure is used for automobile wheel discs.3) The structures of dual phase steel usually consist of ferrite hardened by Si in solid solution and martensite as a second hardening phase. The improved fatigue characteristics are inferred to be attributed to the evolution of the fine dislocation cell structure through cyclic hardening of the ferrite phase and the effect of the hardened phase to divert the fatigue cracks.5) The relationship between the strengthening mechanism and fatigue behavior of steels is extremely important. However in the case of practical steels, the strengthening mechanism is not necessarily clear as more than one mechanism are simultaneously involved. In order to clarify the mechanisms, it is important to separate the strengthening mechanism that controls the evolution of the dislocation structure and fatigue behavior.

This study focused on Si, which is typically used as a solid solution hardening element. A cyclic bending fatigue test using Fe–Si binary alloys was conducted and the relationship between the evolution of dislocation structures and fatigue crack behaviors was examined. Previous studies have been reported that Si in Fe lowers the stacking fault energy to suppress cross slip,6,8–10) which was considered to result in the planar dislocation structure under the fatigue test.6) However, dislocation behaviors have been reported mostly on the basis of tensile tests. On the other hand, regarding the dislocation behaviors at the fatigue crack tips of bcc metals, many studies were conducted from mechanical viewpoints in the 1960s, and the cause of fatigue was considered to be closely associated with screw dislocation behavior.11) Recent studies include research using a model in which the fatigue crack in Fe–Si alloy interacts with the grain boundary to be branched.12) Another study concerns the influence of an interstitial atom, initial grain size and the temperature at the evolution of cracks in terms of dislocation behaviors with the fatigue test conducted using pure
iron at a low temperature,\textsuperscript{13} but these are limited in number. Some studies have reported detailed analysis on the evolution of the dislocation structure during cyclic deformation using Fe–Si single crystals.\textsuperscript{14,15} However, no study has associated the evolution of the dislocation structure and fatigue crack behavior using Fe–Si binary alloys with the amount of Si and stress level changes systematically as in the present study.

Consequently, this study involved fatigue tests using polycrystalline simple Fe–Si binary alloys and concentrated on the evolution of the dislocation structure by closely and systematically observing the test specimen with a transmission electron microscope (TEM). Furthermore, this study attempted to clarify the relationship between the evolution of the dislocation structure and fatigue crack behavior.

2. Experimental Procedure

The specimens used for fatigue tests were Fe–Si binary alloys having the chemical compositions shown in Table 1. Laboratory vacuum-melted steels prepared with base materials of high-purity ultra-low carbon steel (referred to as ULC hereinafter) and Ti-bearing ultra-low carbon steels (Ti-IF) with respectively added Si contents of zero, 0.5 mass\% (referred to as % simply hereinafter) and 1.0\% were the starting materials. Test specimens were completely annealed at 800°C for 60 s to recrystallize the materials. In Ti-IF, the interstitial atoms are fixed in the form of Ti·(C, N) to make the structure interstitial free. Consequently, this study involved fatigue tests using polycrystalline simple Fe–Si binary alloys and concentrated on the evolution of the dislocation structure by closely and systematically observing the test specimen with a transmission electron microscope (TEM). Furthermore, this study attempted to clarify the relationship between the evolution of the dislocation structure and fatigue crack behavior.

Table 1. Chemical compositions of steels used for fatigue test (mass\%).

\begin{tabular}{|c|c|c|c|c|c|c|c|}
\hline
Steel & C & Si & Mn & P & S & Al & Ti & N \\
\hline
ULC1 & 0.0024 & <0.001 & <0.001 & 0.0002 & 0.0360 & - & 0.0015 \\
ULC2 & 0.0014 & 0.4900 & 0.0045 & <0.002 & 0.0001 & 0.0460 & - & 0.0011 \\
ULC3 & 0.0027 & 0.9990 & 0.0049 & <0.002 & 0.0001 & 0.0450 & - & 0.0009 \\
Ti-F1 & 0.0025 & 0.0021 & <0.001 & 0.0013 & 0.0055 & 0.0460 & 0.0360 & 0.0007 \\
Ti-F2 & 0.0023 & 0.4800 & <0.001 & 0.008 & 0.0005 & 0.0460 & 0.0360 & 0.0006 \\
Ti-F3 & 0.0032 & 0.9600 & <0.001 & 0.0009 & 0.0005 & 0.0440 & 0.0360 & 0.0008 \\
\hline
\end{tabular}

3. Experimental Results

3.1. Tensile Test

Figure 2 shows typical nominal stress and strain curves. The yield strength (YS) was defined to be the lower yield point strength for materials with yield point elongation and the 0.2\% proof stress for the materials without yield point

![Fig. 1. Shapes and dimensions of the specimens used for (a) tensile and (b) fatigue test (mm).](image-url)
The yield strength/tensile strength (MPa) of the materials are described as follows: ULC1, 127/218; ULC3, 178/311; Ti-IF1, 90/254; and Ti-IF3, 159/370. Irrespective of Ti addition, YS and TS values increased with increasing Si. However, in particular YS/TS ratio increased remarkably (Fig. 2). In other words, Si addition remarkably increased the work hardening rate.

3.2. Fatigue Test

Figure 3 shows S–N curves representing the results of the fatigue test. It is clear that the fatigue strength increased with an increase in the amount of added Si for both steel types. However, the increment in the fatigue strength due to Si addition becomes higher for ULC steels than for Ti-IF steels. In the case of steels without Si, Ti-bearing steel (Ti-IF1) having finer grain size showed higher fatigue strength than ULC1. When it comes to steels with 1.0% Si leading to smaller difference in grain size, the fatigue strength levels of Ti-IF3 and ULC3 became almost the same. Put simply, the Si in solid solution increased not only the TS remarkably but also the fatigue strength presumably through the change in the dislocation structure, as discussed in the following paragraph.

3.3. Evolution of Dislocation Structure

3.3.1. Steels Containing 0% Si

(1) Low-stress Amplitude Fatigue

The dislocation structures of the materials fatigue-tested at the low-stress amplitude were carefully observed with a TEM. Figure 4 shows the dislocation structure after applying a stress of 163 MPa to Ti-IF1 1,673,800 times repeatedly. The dislocation structures were not homogeneous, showing a cell structure with a relatively large size of 5 to 10 μm. The center of the photo shows the initiation of cell formation process. It must be noted that there are almost no dislocations inside the cell. In another area of the same material, many refined cells compared with the above can be observed in some positions, the cell structure being heterogeneous. Although the grain size became reduced as a result of Ti addition, the dislocation structure did not conspicuously change.

(2) Middle-stress Amplitude Fatigue

When the stress amplitude rose to 223 MPa, the cell structures became obvious and the cell size was reduced. Figure 5(a) shows slender cells with a cell width of 4 to 5 μm. Figure 5(b) shows the cell walls assumed to be in the midst of formation. In this case too, the dislocation structure did not show any remarkable change irrespective of Ti addition.

(3) High-stress Amplitude Fatigue

When the stress amplitude was further increased to 268 MPa, the cell structure developed more and the size of cell structure became finer and was reduced to 3 to 4 μm. The crystal orientations of individual cells measured across the cell walls within a single grain were confirmed to be almost the same. This means that the cell walls are boundaries with little difference in orientation. Figures 6(a) through 6(e) show cell structures developed in grains with various orientations, with the extended areas for observation respectively. Depending upon the crystal orientations,
the cell size varied to some extent; however, the cell structures themselves did not differ greatly. Previous studies using single crystals have shown that the substructure depends on the crystal orientation. Therefore, it is inferred that the influence of crystal orientation is not reflected in the case of polycrystalline materials. Figure 7 shows the cell structures in the respective crystal grains including the triple point. In this case, since the dislocation contrast conditions are about the same in respective grains, it may be that the orientations happened to be the same, which is recognized as one of the characteristics of IF steels. The figure indicates clearly that the cell structures became discontinuous in the initial grain boundaries. It is inferred that the cells were formed independently for respective grains and cells were blocked by the grain boundaries. In this case also, the dislocation structure did not show any remarkable change as a result of Ti addition.

3.3.2. Steel Containing 0.5% Si

Since no significant difference was observed in the dislocation structure as a result of Ti addition, ULC2 (0.5% Si) steels were focused on as representative materials in the results of observation.

(1) Low-stress Amplitude Fatigue

Figure 8 shows the results of TEM observation after applying a low stress of 189 MPa to ULC2 498,000 times repeatedly. In this case, the cell boundaries were not clear compared with the case of the 0% Si steels and bundle-like dislocations in several locations within grains were charac-
teristically observed.

(2) Middle-stress Amplitude Fatigue

Figure 9 shows the results of TEM observation after applying a middle stress of 222 MPa to ULC2 41 300 times repeatedly. In this case, there are regions that have a cell structure of a comparatively large size of approximately 6 to 10 μm, whereas there are also regions that have substructures with bundle-like aggregates of dislocations at intervals of approximately 1.5 μm (vein structure) and substructures with periodically arranged (about 2 μm interval) dislocation walls with a thickness of about 0.1 μm are mixed. The latter substructure has two possibilities, namely the ladder structure or the labyrinth structure, which is discussed later. Moreover, the fraction of these two different types of substructures differed from grain to grain.

(3) High-stress Amplitude Fatigue

Figure 10 shows the results of TEM observation after applying a high stress of 251 MPa to ULC2 39 700 times repeatedly. In this case, there were two kinds of regions where a) cell structures (about 1 μm) developed and b) the vein structure (at intervals of about 1 μm) and the periodically arranged dislocation wall structure (at intervals of about 1 μm) are mixed. Vein structures of bundle-like dislocations were observed in the vicinity of the boundaries of the periodically arranged dislocation wall structure. Furthermore, the periodically arranged dislocation wall structures were frequently observed in the grains in which the vein structures developed.

3.3.3. Steel Containing 1.0% Si

Since no significant difference was observed in the dislocation structure as a result of Ti addition in this case, either, Ti-IF3 (1.0% Si) steels were focused on as representative materials in the results of observation.

(1) Low-stress Amplitude Fatigue

When 1% Si was added, the dislocation structure indicated a remarkable change. Figure 11 shows the dislocation structures after applying a stress of 210 MPa to Ti-IF3 1353 200 times repeatedly. In this case, planar dislocations were evenly scattered and tangled with each other in some locations.

(2) Middle-stress Amplitude Fatigue

Figure 12 shows the dislocation structures after applying an increased stress of 268 MPa to Ti-IF3 1 353 200 times repeatedly. The dislocations, scattered evenly under low-stress amplitude, aggregated as the stress amplitude increased, forming a vein structure such as bundle-like dislocations as seen in the case of 0.5% Si steels. The structure consisted of a mixture of bundled dislocation regions and dislocation-free regions (channels with a width of approxi-
approximately 0.3–1 μm), which has an interval of 1 μm. No dislocation cell structure was observed. In addition, particular structures in which no dislocation was present along the initial grain boundaries within a distance of approximately 0.5 μm from them were observed characteristically. It must be noted that this type of structure, namely a dislocation free zone in the vicinity of grain boundaries, was observed near all of the adjacent grain boundaries in the grain shown at the center of Fig. 12.

(3) High-stress Amplitude Fatigue

Figure 13 shows the dislocation structures after applying a further increased stress of 305 MPa to Ti-IF3 35 600 times repeatedly. A typical vein structure was observed; bundle-like dislocation extended to be connected with the adjacent bundle-like dislocation, while taking paralleled positions at intervals of approximately 0.5 to 1.0 μm. In this case, too, there were regions observed in which no dislocation was present near the boundaries. On the other hand, after applying a stress of 381 MPa to Ti-IF3 8 500 times repeatedly, such periodically arranged dislocation wall structure as shown in Fig. 14 was observed in a vein structure. The in-
ters between the walls were the same, approximately 1 μm. No cell structures such as those seen in most grains of the 0% Si steels and in some grains of 0.5% Si steels were observed in the 1.0% Si steels. It was also confirmed that the heterogeneity of the dislocation structures from place to place under high-stress amplitude tended to decrease more than those under low-stress amplitude conditions.

3.4. Observation of Fatigue-fractured Surface

A fatigue-fractured surface with an initial crack developed in the top surface region was observed with an SEM. The specimens were 0% Si steels and 1.0% Si steels and stresses applied were low and high-stress amplitudes. Figure 15 shows the results of observation. Since the fractured surfaces did not differ with or without Ti addition, Ti-bearing steels are focused on here. Many striations were observed on the fractured surfaces regardless of the stress amplitudes in Ti-IF1 (0% Si) as shown in Figs. 15(a) and 15(b). These structures appear to be striations that developed normal to the direction of main crack propagation and are presumably assumed to have been formed as the cracks advanced within grains. On the other hand, in 1% Si steels, many clear grain boundary fractures as shown in Fig. 15(d) were observed just beneath the top surface when they were subject to a fatigue test under high-stress amplitude.

4. Discussion

4.1. Evolution of Dislocation Structure during Fatigue Test and the Influence of Si in Steel

The evolution of the dislocation structures at the top surface under the cyclic bending fatigue test is remarkably subject to the amount of Si added. As represented by the 3-dimensional description of the dislocation structure under high-stress amplitude as shown in Fig. 16, dislocation cell structures with small angle boundaries were observed in the steels without Si and vein structures with bundle-like dislocations were observed in the steels with 1% Si. The dislocation on the {011} slip plane expanded most conspicuously. A substructure with periodically arranged dislocation walls (about 1 μm interval) with a thickness of about 0.1 μm was also observed in other areas of observation.

It is commonly assumed that the evolution of the dislocation structure under cyclic fatigue completes during a few percent of the total life of the fatigue and the saturated structure is formed with an increasing number of cycles. Saturated structure, as discussed in this study, depends on the cyclic stress amplitude and material parameters. Among many material parameters, the influence of Si addition in particular was found to be most obvious in this study.

It has been reported that the difficulty of cross slip depends on stacking fault energy and Si addition to steel materials decreases stacking fault energy to make it more difficult for the cross slip to take place.6,8–10) The mechanism can be attributed to the assumption that the Si-free steels are considered to be pure iron with comparatively high stack fault energy that results in stable dislocation cell structures with small angle boundaries, owning to cross slip of dislocations under cyclic stress. On the other hand, Si addition lowers stacking fault energy to make it more difficult for dislocations to cross slip, which can be inferred to form structures, typically vein structures, containing bundle-like dislocations, because dislocations are forced to glide on the primary slip system resulting in frequent dislocation interactions. It is worthwhile mentioning that the periodically arranged dislocation walls observed in the vein structure are formed in 0.5% Si and 1.0% Si steels. This kind of dislocation structure is similar to the ladder structure found in copper single and poly crystals, which is formed by the activation of only a primary slip system7,16,17) but at the same time it may be called a labyrinth structure, which is formed by the activation of more than two slip systems as reported in Fe–3%Si14) alloy, Fe–25%Cr18) alloy and Cu.19–21) The dislocation structure shown in Fig. 10, for instance, may be called a labyrinth structure, in the sense that the mixed dislocation structures consisting of periodically arranged dislocation walls and cell structures are observed, which is very similar to the dislocation structures in copper, which have been identified as a labyrinth structure.17,19,21) Furthermore, the dislocation structure shown in Fig. 13 may be recognized to be comprised of dislocation walls perpendicular to each other, which are frequently observed in the labyrinth structure in Cu.17,20,21) Turenne et al.22) proposed a theoretical model on the formation mechanism of a labyrinth structure, namely a geometrical model of dipolar dislocation structures based on the stacking of dipole loops, and verified that the labyrinth walls in bcc Fe–25Cr correspond to a pair of {011} walls. The present study has not clarified the crystallographic relationship between a slip system and a dislocation wall, which is necessary in order to identify the dislocation structure and to elucidate its formation mechanism. So, further research is required.

As for the relationship between the evolution of a dislocation structure and the initial grain boundary, the following were observed. 1) The cell structures were sharply blocked by the initial grain boundaries in the Si-free steels. 2) Low dislocation-density regions were found along all of the adjacent initial grain boundaries with a distance of ap-
proximately 0.5 μm in the Si-bearing steels. Concerning the interaction between dislocations and grain boundaries during the cyclic test, detailed studies on Cu polycrystal,\textsuperscript{17,23)} Cu bicrystal\textsuperscript{24} and Al polycrystal\textsuperscript{25} were carried out taking into account the grain boundary characteristics. Winter \textit{et al.}\textsuperscript{17} and Luoh \textit{et al.}\textsuperscript{23} reported that dislocation free zones (DFZ) along grain boundaries were formed when the vein structures developed within grains, whereas DFZ were not formed in the case of cell structures, which is very similar to the results of the present study using Fe–Si alloy. Although further study is required, Luoh \textit{et al.}\textsuperscript{23} concluded that the multipolar dislocation loop patches (MLPs) and walls (MLWs) are built up due to grain boundary incompatible stresses providing secondary glides, which are thought to cause annihilation of dipole loops by intersecting MLPs or MLWs. Referring to this model, it is inferred that in the Si-bearing steels the difficulty of cross slip leads to the annihilation of dislocation dipole loops in the vicinity of grain boundaries by intersecting MLPs and MLWs, or dislocations simply sinks into grain boundaries. In the case of pure iron after the deformation of cold rolling, localized softening near the initial grain boundaries has been reported.\textsuperscript{26}} Further systematic quantitative study in bcc metals is necessary to clearly understand the mechanism.

The present study shows that the saturated dislocation structure varies with the cyclic stress amplitude. As shown in Fig. 17, the cell width and vein width varied with the

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stress amplitude, respectively. More specifically, the cell width remained comparatively large, although varying from approximately 5 to 8 μm under low to middle-stress amplitude, and clear cell structures developed under high-stress amplitude with comparatively homogenous and fine cell sizes of 2 to 3 μm. On the other hand, the vein width varied largely under low-stress amplitude, bundled dislocations started to appear under middle-stress amplitude, and the vein width converged to 0.5 to 1 μm dimension homogeneously with further increasing stress amplitude with a channel width of about 0.5 μm. The relationship between the development of dislocation structures and stress amplitude during cyclic deformation has already been investigated from both experimental and theoretical points of view mainly on Cu.²⁷,²⁸ The cyclic stress (σ) of materials with periodically arranged dislocation walls is expressed by the sum of the stress necessary for dislocation to bow out into the channel (σ_{bowing}) and interaction stress (σ_{dip}) of screw dislocation dipoles within the channel. σ_{bowing} is expressed by \( Gb/d_c \) and σ_{dip} is by \( Gb/4\pi h \), where G, b, d_c and h are shear modulus, Burgers vector, channel width and interval of slip planes for interacting screw dislocations, respectively. These equations suggest that the dislocation substructure becomes fine with increasing stress amplitude as demonstrated in Fig. 17. σ_{bowing} estimated by the above equation using the experimentally obtained \( d_c \) value (Fig. 13) is smaller than actual stress σ, which indicates the significant contribution of σ_{dip}. The value of h may be estimated to be 10–100b in order to obtain a realistic value of σ_{dip}, which is in agreement with the value reported for Cu.

4.2. Fatigue Cracks Behavior

As clearly shown by the SEM observation of fatigue fracture surfaces (Fig. 15), striation structures were observed in regions extending from the top surface to the inner layer of the Si-free steels regardless of Ti addition. On the other hand, the intergranular cracks were observed just beneath the top surface under high stress amplitude particularly in the 1.0% Si steels regardless of Ti addition. Figure 18 shows the length of fatigue crack per unit area as a function of the depth from the top surface of the 1.0% Si steels. The length of fatigue crack is the longest at the top surface and such cracks are mainly located along the grain boundaries. Figure 19 shows a SEM image of the intergranular cracks just beneath the top surface of the 1.0% Si steels. These results are assumed to support the results of the SEM observation of fatigue-fractured surfaces shown in Fig. 15.

For the consideration of the relationship between the dislocation structures just beneath the top surfaces of specimens and Si addition, fatigue crack behavior in terms of dislocation arrangement can be represented schematically as shown in Fig. 20. It is thus inferred that in the case of 0% Si steels an obvious dislocation cell structure was formed below the top surface and micro cracks were evolved on the cell boundaries. In this case, the fractured surface is of striation structure. When it comes to the 1.0% Si steels, the strain concentration within grains is dispersed since the dislocation structure is of a vein structure, leading to concentrated strain in the grain boundaries. Furthermore, taking into consideration the fact that the region approximately 0.5 μm from the initial grain boundaries is extremely low in dislocation density, it seems to be the case that the strain due to the cyclic deformation concentrated in the grain boundary, evolving micro cracks in the grain boundary, and leading to cracks propagating further along the grain boundaries with the dislocation free zone.

![Fig. 17. Influence of Si addition on the cell size and vein interval in dislocations structures as a function of applied cyclic stress.](image1)

![Fig. 18. Crack length per unit area as a function of depth from the top surface of the specimen tested under a high stress amplitude of Ti-IF3 (1% Si) (σ=364.2 MPa, N=19 200).](image2)

![Fig. 19. SEM image of intergranular cracks observed just beneath the top surface of Ti-IF3 (1% Si) tested under a high stress amplitude (σ=364.2 MPa, N=19 200).](image3)
5. Conclusions

The evolution of dislocation structures in Fe–Si binary alloys based on ultra-low carbon steels during cyclic bending test were closely observed with a TEM and the relationship between the evolution of dislocation structures and fatigue crack behaviors was examined. The following results were obtained.

(1) Si addition increases both yield strength and tensile strength; the increase in tensile strength, i.e. work hardening, is more remarkable. Besides, Si addition increases fatigue strength.

(2) The evolution of dislocation structure under the cyclic bending test is remarkably subject to Si addition. In the case of 0% Si steels, dislocation cell structures having small angle boundaries are evolved. In the case of 1.0% Si steels, vein structures with bundle-like dislocations are formed. It is inferred that improvement in fatigue strength by Si addition is closely associated with such dislocation structures in principle.

(3) It is also inferred that cell structures evolved in Si-free steels because cross slip was easy, and vein structures were formed in Si-containing steels because cross slip became difficult due to lowered stacking fault energy. The presence of localized periodically arranged dislocation wall structures in the vein structure was also observed. In addition, characteristic dislocation free zones were observed in Si-containing steels within a distance of approximately 0.5 μm from the grain boundaries.

(4) The observation of fatigue-fractured surfaces and the propagation behavior of fatigue cracks revealed transgranular fractures in 0% Si steels, while it revealed intergranular cracks just beneath the top surface in 1.0% Si steels under high-stress amplitude. It is assumed that micro cracks evolved in cell boundaries first and the cracks propagated thereafter along the cell boundaries to cause such transgranular fractures in 0% Si steels. On the other hand, in 1.0% Si steels it is also inferred that vein structures evolved and strain concentration was dispersed within the grains, causing the cracks to form in the boundaries and to propagate along them due to the presence of the dislocation free zone near the boundaries.

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