Evolution of dislocation structure and fatigue crack behavior in Fe-Si alloys during cyclic bending test

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Abstract. The evolution of dislocation structures was investigated by means of 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. In steel without Si the cell structure develops, whereas in steel with 1% Si the vein structure evolves, which is considered to lead to the increased fatigue strength. The cell structure in 0% Si steel is postulated to be caused by the easy cross slip of dislocations, whereas the vein structure in the steels with Si is inferred to be caused by the difficulty in cross slip presumably due to the decrease in stacking fault energy. Furthermore, the steel containing Si shows a dislocation free zone (DFZ) along grain boundaries. A transgranular fracture takes place in 0% Si steel, while in 1% Si steel many intergranular cracks were observed just beneath the top surface, which was 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 a DFZ.

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 these requirements. 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 completely understood as more than one mechanism is simultaneously involved. This study focused on Si in Fe, which is typically used as a solid solution hardening element. Previous studies [1-4] have reported that Si in Fe presumably lowers the stacking fault energy to suppress cross slip which was considered to result in planar dislocation structures under fatigue testing [1]. However no study has associated the evolution of dislocation structures with fatigue crack behavior, which is the focus of the present study.

2. Experimental procedure
The specimens used were Fe-Si alloys having the chemical compositions and average grain size shown in Table 1. Ti-bearing ultra-low carbon steels (Ti-IF) with respectively added Si of 0, 0.5 and 1.0% were processed through hot rolling, cold rolling and annealing [4].
Table 1 Chemical compositions (mass%) and grain size (μm) of steels used for fatigue testing

| Steels   | C     | Si     | Mn    | P    | S    | Al    | Ti    | N    | Grain Size |
|----------|-------|--------|-------|------|------|-------|-------|------|------------|
| Ti-IF1   | 0.0025| 0.0021 | <0.001| 0.0013 | 0.0005 | 0.0460 | 0.0360 | 0.0007 | 24         |
| Ti-IF2   | 0.0023| 0.4800 | <0.001| 0.0008 | 0.0005 | 0.0460 | 0.0360 | 0.0006 | 20         |
| Ti-IF3   | 0.0032| 0.9600 | <0.001| 0.0009 | 0.0005 | 0.0440 | 0.0360 | 0.0008 | 21         |

The fatigue test was conducted on 1mm thick recrystallized specimens. The test machine was a planar cyclic bending type [4]. The frequency was 7 Hz. The stress ratio, R, was equal to –1 and the applied stress amplitude was within a range of 160MPa to 380MPa. The point at which the bending moment lowered by 50% from the saturation point was defined as a fracture. The shapes and dimensions of the specimens for the tensile test and fatigue test were the same as those described in the literature [4]. The evolution of the dislocation structure was investigated with TEM. The evolution and propagation behaviors of fatigue cracks were evaluated by measuring the length of cracks per area as a function of the depth from the top surface.

3. Experimental results

3.1. Tensile and fatigue test
The typical stress and strain curves obtained indicate that the Si addition increased the work hardening rate [4]. Figure 1 shows the results of S-N curves. It is clear that the fatigue strength increased with an increase in the added Si amount.

Fig.1 Influence of Si addition in Ti-IF steels on S-N curves.

Fig.2 TEM micrographs of Ti-IF1 (0%Si) tested under a) a low (σ=162.8MPa, N=1,673,800) and b) a high (σ=267.8MPa, N=15,100) stress amplitude.

3.2. Evolution of dislocation structure
The dislocation structures fatigue-tested at the low-stress amplitude in steel containing 0%Si were carefully observed with TEM. Figure 2 a) shows dislocation cell structure with a relatively large size of 5 to 10μm. The center of the photo shows the initiation of the cell formation process. In another area, many comparatively refined cells with a heterogeneous structure can be observed in some positions. When the stress amplitude rose, the cell structures became clear and the cell size was reduced to 4 to 5μm. As the stress amplitude was further increased, the cell size became smaller and was reduced to 3 to 4μm (Fig.2 b). 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.
When 1%Si was added, the dislocation structure was changed remarkably. Figure 3 a) shows dislocation structures with low stress amplitude and that the planar dislocations were evenly scattered and tangled with each other in some locations. As the stress amplitude increased, the dislocations aggregated. The structure consisted of a mixture of bundled dislocation regions and dislocation-free regions (channels with a width of 0.3-1μm), which had an interval of 1μm. No dislocation cell structure was observed. In addition, particular structures were observed in which no dislocation was present along the initial grain boundaries within a distance of 0.5μm (Fig.4). It must be noted that this type of structure, namely a dislocation free zone (DFZ), was observed near all of the adjacent grain boundaries in the grain. Figure 3 b) shows the dislocation structures after applying further increased stress. A typical vein structure or a labyrinth-like structure was observed. Again, in this case a DFZ was confirmed.

3.3. Observation of fatigue-fractured surfaces
A fatigue-fractured surface in the top surface region was observed with SEM. Many striations were observed in 0%Si steels, while grain boundary fractures were observed in 1%Si steels [4]. Figure 5 shows the length of fatigue cracking per unit area of the 1.0%Si steels. The length of fatigue cracking is the longest at the top surface and such cracks are mainly located along the grain boundaries.
4. Discussion
The 3-dimensional description of the dislocation structure in Fig. 6 reveals that dislocation cell structures were observed in the 0%Si steel and vein structures with bundle-like dislocations were observed in the 1%Si steel. The dislocation on the {011} slip plane expanded most conspicuously.

The mechanism can be attributed to the assumption [1, 2] that the Si-free steels, having comparatively high stack fault energy, resulted in stable dislocation cell structures owning to the cross slip of dislocations. On the other hand, in the Si-added steels, presumably having lower stacking fault energy, it was more difficult for dislocations to cross slip leading to vein structures. It is worthwhile mentioning that the periodically arranged dislocation walls observed in the vein structures are similar to the ladder structure found in Cu [6], which is formed by the activation of only a primary slip system, but at the same time they may be referred to as a labyrinth structure, which is formed by the activation of more than two slip systems as reported in Fe-3%Si [5] alloy and Cu [6]. The present study has not clarified the crystallographic relationship between a slip system and a dislocation wall. Therefore, further research is required.

Concerning the interaction between dislocations and grain boundaries, Luoh et al. [7] carried out a detailed study on Cu polycrystal [7] taking into account the grain boundary characteristics. They reported [7] that a DFZ formed along grain boundaries when the vein structures developed, whereas a DFZ did not form in the cell structures, which is very similar to the results of the present study.

For the consideration of the relationship between the dislocation structures and fatigue crack behavior in terms of dislocation arrangement, a model was represented schematically in Fig. 7. It is thus inferred that in 0%Si steels an obvious cell structure was related to the fractured surface with a striation. In the case of 1.0%Si steels, the strain within grains is dispersed uniformly due to a vein structure, while in the grain boundaries the strain can be concentrated due to a DFZ, leading to the formation of micro cracks and their propagation along a DFZ.

5. Conclusions
The results obtained are as follows:
1. Si addition increases fatigue strength.
2. The dislocation cell structures are evolved in 0%Si steels, whereas in 1.0%Si steels vein structures are formed. It is also inferred that in Si-free steels cross slip was easy to perform, while in Si-containing steels cross slip became difficult.
3. In addition, DFZs along initial grain boundaries were observed in Si-containing steels.
4. Transgranular fractures occurred in 0%Si steels, while in 1.0%Si steels intergranular cracks were observed. Dislocation structure was considered closely related to fracture behavior.

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