Irregular or Smooth Grain Boundaries Evolved after Secondary Recrystallization of Fe–3%Si Steel

Hyung-Ki Park1,2, Chang-Soo Park1, Tae-Wook Na1, Chan-Hee Han2 and Nong-Moon Hwang1,*

1Department of Materials Science and Engineering, Seoul National University, Seoul 151-742, Korea
2POSCO Technical Research Laboratories, POSCO, Pohang, Korea

Irregular or smooth grain boundaries after secondary recrystallization are formed depending on whether interpass aging is adopted or not during cold rolling of Fe–3%Si steel. Interpass aging induces the primary texture of high intensity of {111} and {411} orientations, which have a 29 relation with respect to the Goss orientation and therefore are favorable for Goss grains to grow by solid-state wetting. Under this condition Goss grains come in contact with each other by wetting without leaving any small grains in between, resulting in irregular boundaries.

Keywords: abnormal grain growth, secondary recrystallization, grain boundary wetting, interpass aging

1. Introduction

Abnormal grain growth (AGG), which is also referred to as secondary recrystallization, often occurs in polycrystalline metals. During AGG only a few grains grow abnormally consuming the neighboring matrix grains. This phenomenon is particularly famous in Fe–3%Si alloy because the core loss of Fe–3%Si can be minimized by properly controlling AGG. Since the first report of the selective AGG of Goss grains in Fe–3%Si alloy by Goss in 1935,

Highly irregular grain boundaries are produced with interpass aging whereas smooth grain boundaries are produced without interpass aging. The purpose of this work is to study why irregular grain boundaries are evolved after secondary recrystallization in the Fe–3%Si sheet specimens undergoing interpass aging. Rather than studying the beneficial effect of the interpass aging, our study is limited to the mechanism as to the different morphology evolution of grain boundaries between the two processes with and without interpass aging.

2. Experimental Procedure

An ingot of Fe–3%Si, which employed AlN as an inhibitor of grain growth to produce the highly grain-oriented electrical steel, was used as a starting material. The steel ingot was hot rolled to 2.3 mm and the thickness was further reduced to 0.3 mm by one stage cold rolling method, which is equivalent to a total engineering thickness reduction of ε = 87%. To investigate the effect of interpass aging during cold rolling on the microstructure evolution after secondary recrystallization, specimens with and without interpass aging were prepared during cold rolling. The specimen with interpass aging was given four separate treatments for 5 min at 250°C at intermediate thicknesses of 1.5, 0.95, 0.62, and 0.43 mm. Hereafter, these two specimens with and without interpass aging will be referred to as ‘A’ and ‘B’, respectively.

Precipitates play a critical role in inducing AGG. In highly grain-oriented electrical steel, precipitates such as AlN serve as a grain growth inhibitor at low temperature and AGG occurs when precipitates start to dissolve or coarsen at high
temperatures. The effect of AlN on secondary recrystallization would not be changed by such a low temperature heat treatment by interpass aging, and therefore would be the same in both specimens. The two specimens were annealed at 850°C for 150 s for decarburization and primary recrystallization. These specimens underwent a stepwise heat treatment at 15°C/h up to 1200°C and held for 10 h for secondary recrystallization. The sheet specimens were etched with 50% water-50% HCl at 80°C to identify the grain boundaries of abnormally-grown grains.

To investigate the texture after primary recrystallization at the surface layer, where Goss nuclei for AGG were distributed, the two specimens after primary recrystallization with a thickness of 0.3 mm were polished down to a thickness of 0.26 mm. The macrotextures were examined by a conventional X-ray texture goniometer (D8 Advance, Bruker) using Co Kα radiation. From three incomplete pole figures, the three-dimensional orientation distribution function (ODF) was calculated by the series expansion method as presented in the orientation space definition (ODF) was calculated by the series expansion method and presented in the orientation space defined by the Euler angles (ψ, Φ, ϕ). To determine the orientation relationships of individual grains relative to the Goss orientation, [110](001), the specimens after primary recrystallization were analyzed by electron backscattered diffractometer (EBSD) attached to a field-emission scanning electron microscope (JSM-6500F, JEOL). EDAX/TSL software was used to analyze the orientation.

### 3. Results and Discussion

Figures 1(a) and 1(b) are the microstructures of ‘A’ and ‘B’, respectively, after secondary recrystallization and its corresponding (100) and (110) pole figures are shown on the right inset of each figure. The pole figures indicate that abnormally-grown grains in both specimens had a Goss orientation. Figure 1(a) for ‘A’ with interpass aging shows highly irregular grain boundaries whereas Fig. 1(b) for ‘B’ without interpass aging shows smooth grain boundaries. This different grain boundary morphology would come from the different surroundings of Goss grains in the texture after primary recrystallization, which affects the growth rate of the Goss grains during secondary recrystallization. Indeed, Homma and Hutchinson reported the effect of the texture on the growth rate of the Goss grains. Also, in the viewpoint of solid-state wetting mechanism, the primary texture affects the growth rate of the Goss grains because it determines the distribution of the orientation relation between the Goss grains and matrix grains. Therefore, to clarify the different grain boundary morphology between the ‘A’ and ‘B’, the textures after primary recrystallization were compared by X-ray diffraction.

Figures 2(a) and 2(b) show ODFs of ‘A’ and ‘B’ after primary recrystallization, respectively. Mainly evolved orientations were the same between the two specimens. However, the texture of ‘B’ was more broadly distributed than that of ‘A’. In both specimens, {111}(112) and {411}(148) orientations developed during primary recrystallization. Heavily cold rolled body-centered cubic steel, consisting of α-fiber (RD // (110)) and γ-fiber (ND // (111)) textures, is known to have a tendency to generate {111}(112) and {411}(148) orientations after primary recrystallization. The stored energy of the grains developed after cold rolling is maximal in the {111}(112) orientation, resulting in preferred nucleation of this orientation. The {411}(148) orientation was experimentally shown to nucleate in the deformed α-fiber texture. Although the development of the {411}(148) texture is well established, its formation mechanism has not been understood clearly.

To clarify the difference of the texture in more detail, the ODF sections at ϕ = 45° of the specimen with and without...
interpass aging, which include orientations mainly evolved in body-centered cubic iron, were magnified as shown in Figs. 2(c) and 2(d), respectively. The drastic difference in the texture intensity between the two specimens is revealed. Compared with the main texture after primary recrystallization of ‘B’, that of ‘A’ has a significantly high intensity of orientations near \{111\}\{112\} and \{411\}\{148\}, which have a $\Sigma 9$ relationship with respect to the Goss orientation. Indeed, the changes of texture after primary recrystallization by interpass aging were reported.\textsuperscript{15}\textsuperscript{17} In relation to this change of texture, Matsuo \textit{et al.}\textsuperscript{15} reported that interpass aging during cold rolling causes free carbon to pin dislocations, which inhibits a complex slip such as cross-slip, and promotes the deformation by a single slip. Therefore, interpass aging would affect the deformation behavior during cold rolling, which might again affect the change of the texture after primary recrystallization.

The difference of the texture determines a percentage of low energy boundaries relative to the Goss orientation, which affects the growth rate of Goss grains during secondary recrystallization. In the mechanism of secondary recrystallization by solid-state wetting, the high growth rate corresponds to the high probability of wetting, which would depend on the texture after primary recrystallization.\textsuperscript{14} For example, if the texture produces a higher percentage of low energy grain boundaries with respect to the Goss orientation, the wetting probability of Goss grains increases.\textsuperscript{7,10} For this, the percentage of coincidence site lattice (CSL) boundaries, which would be considered as low energy boundaries,\textsuperscript{24,25} relative to the Goss orientation was calculated using EBSD. In fact, the crystallographic relationship as well as the boundary plane should be considered to determine the boundary energy.\textsuperscript{26} However, the data for the boundary plane are not available; we had to assume that the boundaries with CSL relationship have low energy.

Figure 3 shows the percentage of CSL boundaries relative to the Goss orientation in the specimens after primary recrystallization. $\Sigma$ values were calculated up to $\Sigma 29b$ using Brandon’s criteria.\textsuperscript{27} In order to get statistically reliable data, a sufficiently large number of grains were examined: 9105 and 8926 grains in the ‘A’ and ‘B’, respectively. The black and gray colored bars indicate the percentage of the matrix grains with the CSL relationship in the ‘A’ and ‘B’, respectively. The specimen with interpass aging had a higher percentage of CSL boundaries, especially low $\Sigma$ index ($\Sigma 3$–$\Sigma 9$), than that without pass aging. The higher percentage of $\Sigma 9$ boundary can be understood by the primary texture of the specimen with interpass aging which had a high intensity.
of the \{111\}(112) and \{411\}(148) orientations having $\Sigma$9 relation with the Goss orientation.

If $\Sigma$1–$\Sigma$9 boundaries in Fig. 3 are assumed to have low energy enough for the Goss grains to undergo solid-state wetting, the difference in their percentage between ‘A’ and ‘B’ is worth noticing. The percentage of $\Sigma$1–$\Sigma$9 boundaries in Fig. 3 is 8.62% in ‘A’ and 6.9% in ‘B’, which suggests that the wetting probability in ‘A’ should be higher than that in ‘B’. This means that the growth rate of Goss grains by solid-state wetting in ‘A’ would be higher than that in ‘B’ due to its higher percentage of low energy boundaries. Therefore, the rate of secondary recrystallization in ‘A’ would be higher than that in ‘B’.

In the case of ‘A’, an abnormally-growing Goss grain comes in contact with another abnormally-growing Goss grain by solid-state wetting without leaving any small grains in between, before the appreciable normal grain growth (NGG) occurs among matrix grains. It should be noted that the growth front of Goss grains growing by solid-state wetting is highly irregular as featured by very high frequency of peninsular grains. As a result, the grain boundaries of ‘A’, whose growth front proceeds by solid-state wetting, would be highly irregular as shown in Fig. 1(a).

In the case of ‘B’, however, the rate of secondary recrystallization is relatively low and some NGG of matrix grains would occur before abnormally-growing Goss grains meet each other during secondary recrystallization. When NGG occurs, high energy boundaries between the matrix grains are replaced by low energy boundaries. If the matrix grain boundaries have predominantly low energy, solid-state wetting is not favored. In this condition, the abnormally-growing Goss grains undergo further growth not by solid-state wetting but by the non-wetting mode, where the boundary migrates by the difference in the grain boundary curvature. Therefore, the microstructure after secondary recrystallization is similar to that of NGG with smooth grain boundaries as shown in Fig. 1(b).

### 4. Conclusion

In conclusion, the Fe–3%Si sheet steel, which underwent interpass aging during cold rolling, shows highly irregular grain boundaries after secondary recrystallization. This aspect is attributed to the high growth rate of Goss grains by solid-state wetting, which is again attributed to a higher percentage of low energy boundaries relative to the Goss orientation resulted from interpass aging.

### Acknowledgements

This work was financially supported by POSCO Technical Research Laboratories.

### REFERENCES