The Improvement of Primary Texture for Sharp Goss Orientation on Grain Oriented Silicon Steel

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The relationship between primary and secondary recrystallization texture, and the improvement of primary texture on grain oriented silicon steel was examined. The specimens were prepared by using different C content and different hot rolling conditions in the acquired inhibitor method. Higher reduction of the last two passes of hot rolling gave the same effect to primary texture as more C content. As a result, the sharpness of Goss texture became similar. Higher reduction of the last two passes of hot rolling caused {411} [148] texture to enhance, and higher C content caused {111} [112] texture to enhance in primary texture. Both orientations are Σ9 orientations of Goss. As a result I1, Σ9 of Goss is enhanced. Furthermore, not only Σ9 orientations but also Σ5 might contribute the secondary recrystallization.

KEY WORDS: electrical steel; silicon steel; grain boundary; texture; physical property; secondary recrystallization.

1. Introduction

Grain Oriented Silicon Steel (GO) is mainly used as the core material in transformers, and it is the only product manufactured in the steel industry that applies secondary recrystallization phenomenon. The most successful texture control has been achieved in the industrial scale. It is essentially to enhance both the sharpness of the Goss texture and the magnetic properties, low core loss and high permeability along the rolling direction are closely related to the secondary recrystallization texture, i.e., the sharpness of {110} (001) (Goss) texture. Therefore, it is essential to enhance the sharpness of the Goss texture.

The magnetic anisotropy of Fe-single crystal was discovered in 1926. In 1934, the principal production process on GO was invented by Goss. Since then, a lot of effort has been made to improve this material. As a result, the average deviation angle of (001) axis from the rolling direction has been improved from 7 degrees to 3 degrees in Japan, and the slab reheating temperature can be decreased from an extra high temperature to ordinary one, thus improving both quality and production technology.

The production technology can be classified into two categories. One is the ordinary technology where slabs are reheated over the solution temperatures of the inhibitor substances (such as MnS, AlN and MnSe) in order to cause them to finely disperse regardless of the inhibitor kind. Another is the new nitriding technology, developed by Nippon Steel, where slabs are reheated at sufficiently low temperatures in order to precipitate inhibitor substances. The manufacturing process using the former technology for the preparation of precipitates is named “the inherent inhibitor method” and the latter is named “the acquired inhibitor method”.

In the acquired inhibitor method, as three major factors (inhibitor, structure and texture) of secondary recrystallization for GO can be given separately, many studies by applying this technology have been carried out and its secondary recrystallization mechanism has been clarified.

The mechanism of secondary recrystallization on grain oriented silicon steel has been investigated to improve its magnetic properties and to establish an easier production technology. Two hypotheses have been proposed: the CSL model (where coincident site lattice boundaries play a significant role) and the HE model (where high energy boundaries play a significant role).

The CSL model on GO has become popular and many investigations on the relationship between the primary and secondary recrystallization texture have been carried out. By applying vector method analysis, Harase et al. have proposed that Goss or cube secondary recrystallization evolve by higher frequencies coming in contact with mobile boundaries of these orientations in the cause of grain growth, and that these mobile boundaries are Σ9 (Goss texture) or Σ7 (cube texture). Furthermore, they have concluded that the conditions for a viable nucleus of secondary recrystallization in a primary recrystallized stage, the orientation N should satisfy, are as follows (CSL model):

(a) \( P_{\text{CS}} \Sigma 1 \) (the product of \( I_N \) (the intensity of nucleus orientation N) and \( I_1 \Sigma 1 \) (intensity of \( \Sigma 1 \) coincidence orientation)) of nucleus orientation N should be smaller than certain values (\( P_{\text{CS}} \Sigma 1 \lambda_1 \)),

(b) \( I_N \) should be larger than certain critical values (\( I_{\text{crit}} \)),

(c) Nucleus orientation N should have a higher probabili-
The authors have already reported$^{21}$ the relationship between the primary and secondary texture of the acquired inhibitor method, by changing the cold rolling reduction (CR) and carbon content, and concluded as follows:

(1) For primary texture formation, C content and CR function similarly, i.e., their increases enhance the $I_9 \Sigma 9$ of Goss orientation ($I_9 \Sigma 9$: intensity of $\Sigma 9$ coincident orientations). The relationship between the primary and the secondary texture can almost be explained with CSL model.

(2) $\Sigma 9$ coincidence boundary might move much faster than $\Sigma 5$ boundary, because the grain boundary energy of $\Sigma 9$ might be much lower than that of $\Sigma 5$.

The purpose of this study is to show that the C content and hot rolling condition function similarly by the improvement of the primary texture for the sharpness of the Goss secondary texture in the acquired inhibitor method by applying CSL model. For the systematical analysis, cold rolling reduction was also varied.

### 2. Experimental Procedure

The chemical compositions of the specimens were Si: 3.25, 3.26, C: 0.056(A), 0.015(B), Al: 0.027, N: 0.008, S: 0.007 mass%, as shown in Table 1.

Experimental procedure in the laboratory for the secondary recrystallization was as follows:

Ingots were reheated at 1423 K and hot-rolled. Table 2 shows the pass schedules of hot rolling. The entry temperature was controlled at 1343–1353 K and the exit temperatures were from 1093 to 1163 K for Material A and from 1223 to 1303 K for Material B. The coiling temperature was controlled at 833–853 K. The total time of hot rolling was about 60 s.

Material (A) was hot-rolled to the thickness of 1.80, 2.20, 2.70 and 4.05 mm. The reductions of the last two passes of hot rolling were controlled to about 20 and 15%.

Material (B) was hot-rolled to the thickness of 1.80, 2.40, 2.90 and 3.65 mm. The reductions of the last two passes of hot rolling were controlled to about 40 and 45%. The main differences were the C content and hot rolling condition, and the reduction of the last two passes was intentionally changed.

These specimens were annealed at 1393 K in an atmosphere of 100% N$_2$ (hot band annealing). The specimens were cold-rolled to the thickness of 0.335 mm with the reductions of 84.2, 88.1, 90.2 and 92.2% for Material B.

Following the cold rolling, the specimens were annealed in a wet atmosphere of 25% N$_2$ and 75% H$_2$ for decarburization and primary recrystallization. The annealing temperatures were 1103 K, 1113 K, 1123 K and 1133 K. Nitrogen was injected up to around 0.020 mass% and the annealing separator, that mainly consists of MgO, was coated on the specimen surfaces. The secondary recrystallization annealing was carried out under a 25% N$_2$ and 75% H$_2$ atmosphere with the heating rate of 15 K/h up to 1473 K and maintained for 20 h at 1473 K under a 100% H$_2$ atmosphere for purification. The specimen of the highest magnetic flux density (B$_8$ (T)): in an applied field of 800 A/m at the specific primary recrystallization annealing temperature was examined for the analysis hereafter.

The magnetic flux density (B$_8$ (T)) was measured by Single Sheet Tester (60 mm width, 300 mm length). The textures of primary and secondary recrystallized specimens were measured by X-ray diffraction method. The primary (recrystallization) textures at the surface (1/5th thickness) and the central positions of the specimens were obtained by the complete {100} pole figures, and analyzed by the three dimensional texture analysis by Vector method.$^{25}$ Furthermore, as shown in the previous study,$^{21}$ in the case of acquired inhibitor method, because the secondary recrystallization temperature is so high,$^{26,27}$ the secondary nuclei can nucleate not only at the surface layer but also at other position. Therefore, the result is discussed by using the texture not only at the surface layer but also at the central layer. The secondary textures were measured by back-reflection Laue diffraction method.

The intensity of any coincidence orientation, $\Sigma i$, for a nucleus orientation N is expressed as $I_9 \Sigma i (N)$.$^{28}$ $I_9 \Sigma 9$ and $I_9 \Sigma 5$ of Goss ($\{110\} \{001\}$), dispersed Goss ($\{110\} \{229\}$) and other orientations of primary textures were calculated.

### 3. Result and Discussion

#### 3.1. Primary Texture and Magnetic Property

As B$_8$ value has very close relationship with the sharpness of Goss orientation, the relationship between primary (recrystallization) texture and B$_8$ value is discussed. As the C content for both Materials, A and B, after decarburization annealing was less than 0.0020 mass%, secondary recrystallization was not affected by C content. Unfortunately, the specimen at the lowest cold rolling reduction 81.4%...
of Material A had fine grains, i.e., poor secondary recrystallization.

**Figure 1** shows the relationship between cold rolling reduction (CR) and B8. As CR increased, B8 increased and the Material A coincided with the Material B. This will mean, in general terms, the higher reduction of the later passes of hot rolling also gives the same effect to secondary texture as C content in the range of the actual cold rolling reduction because in the previous study\(^2\) it has already been shown that C content as such gives the influence to the B8 values.

In this study the final thickness was different between Material A and B. In the case of acquired inhibitor method, as the primary grain size is large (21–23 μm) regardless of the final thickness\(^1\)\(^7\)\(^,\)\(^2\)\(^6\)\(^,\)\(^2\)\(^7\), the gradient of texture to the grain number in thinner gauge might be steeper than thicker case. Therefore, when other conditions (inhibitor, primary grain size) are the same, the thinner case will have weaker grain growth selectivity to secondary recrystallization. As Material A has stronger selectivity than Material B, B8 might be underestimated and the above-mentioned understanding could be reasonable in spite of different final thickness.

**Figure 2** shows the secondary (recrystallization) texture measured by back-reflection Laue diffraction method. For the same level of cold rolling reduction, their features were almost the same. In the case at lowest cold rolling reduction (CR: 84.2%) of Material B, dispersed Goss grains increased. The dispersed Goss was identified as \{110\}(229). As this dispersed Goss is obtained by rotating from Goss around ND (Normal direction) axis, and rolling direction departs from the easy magnetizing axis ((001)), B8 deteriorates (decreases).\(^2\)

Here, as already shown\(^2\)\(^7\), I\(_i\)Σ\(_9\) and I\(_c\)Σ\(_5\) of dispersed Goss and dispersed Goss play a significant role in the relation between primary and secondary texture. **Figures 3** and **4** show the relationship between I\(_i\)Σ\(_9\) and I\(_c\)Σ\(_5\) of Goss and B8. A considerably good relation was found. Therefore, in the one stage of cold rolling process, not only Σ\(_9\) orientations but also Σ\(_5\) might contribute to secondary recrystallization of Goss. K. T. Lee et al.\(^2\)\(^9\) also reported the role of Σ\(_5\) boundary of Goss orientation in case of two stages of cold rolling process and, in that case, I\(_c\)Σ\(_9\) was weak.

**Figures 5** and **6** show the relationship between I\(_c\)Σ\(_9\) and I\(_c\)Σ\(_5\) of dispersed Goss and B8. I\(_c\)Σ\(_9\) did not show a good relation (Fig. 5), rather, as I\(_c\)Σ\(_5\) increased, B8 enhanced (Fig. 6). As the effect of \(\Sigma i\) boundary for any orientation (N) should be the same, the relationship between the effects of I\(_c\)Σ\(_5\) and I\(_c\)Σ\(_9\) (Goss) should be the same as that of dispersed Goss. If so, the increase in I\(_c\)Σ\(_5\) might cause the dispersed Goss to secondary recrystallize more and B8 to deteriorate. However, assuming that Σ\(_9\) boundary contributes much more than Σ\(_5\) to secondary recrystallization, as indicated already, and comparing between I\(_c\)Σ\(_5\) and I\(_c\)Σ\(_9\) of Goss and dispersed Goss, this could be explained. Therefore, the former result\(^2\)\(^7\) could be reconfirmed.

**Figure 7** shows the relationship between I\(_N\) (Goss) and B8. As I\(_N\) (Goss) increased, B8 deteriorated. **Figure 8**
Fig. 3. Relation between $I\Sigma 9$ of Goss and B8. (a) At surface layer, (b) at central layer.

Fig. 4. Relation between $I\Sigma 5$ of Goss and B8. (a) At surface layer, (b) at central layer.

Fig. 5. Relation between $I\Sigma 9$ of dispersed Goss and B8. (a) At surface layer, (b) at central layer. (Dispersed Goss means (110) (229).)

Fig. 6. Relation between $I\Sigma 5$ of dispersed Goss and B8. (a) At surface layer, (b) at central layer. (Dispersed Goss means (110) (229).)
Fig. 7. Relation between intensity of Goss and B8. (a) At surface layer, (b) at central layer.

Fig. 8. Macrostructures of secondary recrystallized grains.

Fig. 9. Relation between cold rolling reduction and intensity of Goss. (a) At surface layer, (b) at central layer.

Fig. 10. Relation between cold rolling reduction and $I \Sigma^9$ of Goss. (a) At surface layer, (b) at central layer.
shows the macrostructures of the secondary recrystallized grains. Higher CR, higher B8, gave the large secondary grain size and this means if broad Goss secondary recrystallizes, the secondary grain size becomes smaller. Therefore, in order to obtain sharp Goss texture (high B8), more than certain value of $I_{\text{IN}}$ (Goss) might be unnecessary and the limited $I_{\text{IN}}$ could develop the sharp Goss orientation of secondary recrystallization. This suggests the existence of the upper limit for $I_{\text{IN}}$ (Goss) on condition (b) in CSL model for sharper Goss texture.

3.2. The Formation of Primary (Recrystallization) Texture

In the previous section, the relationship between primary and secondary texture was discussed and reconfirmed by applying the concept of CSL model. In this section, the effect of C content and hot rolling condition to primary texture will be discussed.

Incidentally, $\Sigma 9$ orientations of Goss are \{(110)(447), (778)(447) (\approx (111)(112), (411)(148) and (1154)(481) (\approx (211)(471)). Furthermore, \{778\}(447) (\approx (111)(112)) and \{411\}(148) are main orientations.

3.2.1. Primary Texture

Figures 9, 10 and 11 show the relationship between cold rolling reduction (CR) and $I_{\text{IN}}$ (Goss), $I_{\text{IC} \Sigma 9}$ and $I_{\text{IC} \Sigma 5}$ of Goss. As CR increased, $I_{\text{IN}}$ (Goss) decreased and $I_{\text{IC} \Sigma 9}$ increased a little bit. Although $I_{\text{IC} \Sigma 5}$ for Material A did not vary, it for Material B increased a little bit. Therefore, generally $I_{\text{IC} \Sigma 5}$ and $I_{\text{IC} \Sigma 9}$ of Goss might increase, as CR increases. Furthermore, although Material A had a little bit stronger values, a big difference between Material A and B was not found, i.e., for the significant parameters ($I_{\text{IN}}$ and $I_{\text{IC} \Sigma 9}$ for Goss) of Goss secondary recrystallization, C content and hot rolling condition gave the same effect to primary texture. Therefore, as shown in the previous section the sharpness of Goss for Materials A and B might be similar.

3.2.2. Microstructure and Texture before Cold Rolling

Figure 12 shows the microstructures before cold rolling (after hot band annealing). Generally, the feature was typical as GO,$^{30,31}$ Yoshitomi et al. reported about the effect of the later passes of hot rolling. Their higher reductions bring about smaller grain size after hot rolling.$^{33,34}$ As the reduction of the last two passes of hot rolling is high, the grain size becomes smaller because of the increase of nucleation site. However, Fig. 12 shows the opposite result, and Material A
Fig. 13. Three dimensional orientation distributions of primary recrystallization texture (x random) at surface layer. (a), (b), (c) and (d): Material A with cold rolling reductions of 81.4, 84.8, 87.6, and 91.7%. (e), (f), (g) and (h): Material B with cold rolling reductions of 84.2, 88.1, 90.2, and 92.2%.
Fig. 14. Three dimensional orientation distributions of primary recrystallization texture (x random) at central layer. (a), (b), (c) and (d): Material A with cold rolling reductions of 81.4, 84.8, 87.6, and 91.7%. (e), (f), (g) and (h): Material B with cold rolling reductions of 84.2, 88.1, 90.2, and 92.2%.
had smaller grain size than B (less than half). Higher C content gave finer grain size before cold rolling, because carbides divide grains. In this study, as the grain size before cold rolling was rather large, it was difficult to identify its texture. Therefore, from the viewpoint of grain size, the formation of primary texture is being discussed.

A lot of studies on primary annealing texture of flat products in the field of steel have been reported. These results of single-crystal can not be applied to poly-crystal simply and automatically. Matsuo et al. reported the influences on primary texture of grain size and cold rolling reduction of pure iron, that large grain size causes \(110/110\) and \(111/110\) to develop and \(111/112\) to decline. Abe et al. reported the same phenomenon.

Here, in this study, the series of \(100/100\)~\(411/148\)~\(111/112\) is named \(a\)-orientation, that originates from \(a\)-fiber \(100/100\). Furthermore, the series of \(111/011\)~\(111/112\) is also named \(\gamma\)-orientation, that originates from \(\gamma\)-fiber \(111/011\)~\(123/111\)~\(111/112\).

Figure 13 shows the three dimensional orientation distribution of primary texture at the surface layer. Material A had stronger \(\gamma\)-orientation, and Material B had stronger \(\alpha\)-orientation. This might be caused by the grain size before cold rolling, i.e., Material A had smaller grain size, which might be connected with \(\gamma\)-fiber, and Material B had larger grain size, which might be connected with \(\alpha\)-fiber. \(\gamma\)-orientation contains \(111/112\) which is close to \(\Sigma 9\) orientation for Goss. \(411/148\), which is one of the main \(\Sigma 9\) orientations for Goss, is located in \(\alpha\)-orientation. As a result, both Materials had same level of \(I_c/\Sigma 9\) for Goss.

Figure 14 shows the three dimensional orientation distribution of primary texture at the central layer. Material A and B indicated the same level intensity of \(\alpha\)-orientation, but Material A indicated stronger intensity of \(\gamma\)-orientation than Material B. This might be caused by the grain size. This is the reason why in Fig. 11, Material A had a little stronger \(I_c/\Sigma 9\) of Goss.

3.3. Secondary Recrystallization of Cube Texture \((100)/(001)\)

As shown in Fig. 2(e), in the case of Material B with CR of 84.2%, not only Goss and dispersed Goss but also Cube \((100)/(001)\) grains secondary-recrystallized. Figure 15 shows the cube-texture grains \((100)/(001)\) obtained from Fig. 2(e).

Harase et al. reported that the secondary recrystallization of Cube texture would be related to \(\Sigma 7\) boundary. This was the case of “cross” cold rolling method. And they have summarized that Goss is related to \(\Sigma 9\) and Cube would be related to \(\Sigma 7\) boundary. The reason was not referred.
Considering the existence of the upper limit of IN on condition (b) and condition (c) in CSL model, it can be proposed that the relative intensities between viable nuclei of secondary recrystallization and mobile grain boundaries should be higher than certain values. The relative intensity is defined as \( I_c \Sigma i / I_N \), \( \Sigma i \) coincident orientation density for nucleus N. In this study, \( \Sigma 5 \) and \( \Sigma 9 \) of Goss, dispersed Goss and Cube are considered.

Figure 16 shows the relationship between CR and \( I_c \Sigma i / I_N \) (i=5 or 9, N=Goss, dispersed Goss and Cube orientation). Higher CR caused stronger \( I_c \Sigma 5 / I_N \) and \( I_c \Sigma 9 / I_N \) for Goss and dispersed Goss, but for Cube lower CR caused stronger ones. This means that at higher CR Goss and dispersed Goss could be viable and at lower CR Cube could be viable. In addition, considering that \( \Sigma 9 \) coincidence boundary might move much faster than that of \( \Sigma 5 \), it could be reasonable that at higher CR Goss secondary recrystallization occurs.

Some Cube grains could secondary recrystallize because the secondary recrystallization is the phenomena that very few grains survive at the cost of a huge number of grains as a "probability process". The concept of \( \Sigma 7 \) was not necessary.

4. Conclusions

In order to enhance the sharpness of the Goss texture on GO, it is necessary to clarify the relationship between primary and secondary recrystallization texture and develop the primary texture.

By using materials that bore different C content and different hot rolling condition, and by changing cold rolling reduction, the relationship between C content and hot rolling condition was examined in the acquired inhibitor method by applying CSL model:

(a) Higher reduction of the later passes of hot rolling gave the same effect to primary texture as more C content. As a result, the sharpness of Goss texture became similar.

(b) Higher reduction of the later passes of hot rolling caused \{411\} (148) texture to enhance, and higher C content caused \{111\}/(112) texture to enhance in primary texture. Both orientations are \( \Sigma 9 \) orientations of Goss. As a result \( I_c \Sigma 9 \) of Goss is enhanced. Furthermore, not only \( \Sigma 9 \) orientations but also \( \Sigma 5 \) of Goss might contribute the secondary recrystallization.

(c) The formation of Cube secondary texture can also be explained by \( I_c \Sigma i / I_N \) (\( \Sigma i \) coincident orientation density for nucleus N). This is a kind of the application in CSL model.

REFERENCES