The Relationship between Primary and Secondary Recrystallization Texture of Grain Oriented Silicon Steel

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The relationship between primary and secondary recrystallization texture of grain oriented silicon steel, of which process is the only one case applying the secondary recrystallization phenomenon in the steel industry, was examined. The specimens with various kinds of the primary texture were obtained by changing the cold rolling reduction and carbon content, and were secondary-recrystallized by injected inhibitor method. It was reconfirmed that in order to realize the sharp Goss orientation ([110](001)), the intensity of Σ9 coincidence boundaries for Goss orientation will be strong and Σ9 coincidence boundaries will move faster than Σ5 boundaries.

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

1. Introduction

Grain Oriented Silicon Steel (GO) is mainly used for the core materials of transformer, and it is the only one product, which is being manufactured by applying the secondary recrystallization phenomenon in the field of steel industry. The most successful texture control has been achieved in the industrial scale.1) Its 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}k001l (Goss texture). Therefore, to enhance the sharpness of Goss texture is essential, and it is no exaggeration to say that its history has been to find out the technology to improve the sharp Goss texture and to develop its easier manufacturing processes.2)

In 1934, the principal process was invented by Goss,3) and was industrialized and developed by Armco (CGO: conventional grain oriented silicon steel).4,5) The average deviation angle of (001) axis from the rolling direction is around 7 degrees. In the 1960’s, high permeability grain oriented electrical steel (its average of the deviation is around 3 degrees)6) was developed by Nippon Steel7–9) (HI-B: high permeability grain oriented silicon steel). Later in the 1970’s Kawasaki Steel developed the similar grade.10,11) The major differences among these three processes are the kind of the precipitates (inhibitor substances such as MnS, AlN and MnSe) and cold rolling condition (final cold-rolling reduction) for primary recrystallization texture. However the technology for the preparation of inhibitors is the same, i.e., in order to obtain the finely-dispersed inhibitor, the temperature of slab re-heating at hot-rolling should be higher than their solution temperatures.

Recently, another technology for the preparation of inhibitor (i.e., nitrogen injection technology) was developed by Nippon Steel.12–13) In this method, as three major factors (inhibitor, structure and texture) of secondary recrystallization for grain oriented silicon steel can be given separately, many studies in the field of this technology have been carried out and its secondary recrystallization mechanism has been clarified.14–17)

In this study, in order to avoid the influence of inhibitor due to the thickness difference, specimens were prepared to have the same final thickness and their primary textures were prepared by changing the cold rolling reduction and the carbon content, and the relationships between primary and secondary texture were investigated under the same inhibitor and structure conditions.

2. Experimental Procedure

The chemical compositions of the specimens were Si: 3.25, C: 0.017(A), 0.056(B), Mn: 0.09, Al: 0.027, N: 0.008, S: 0.007 mass%, as shown in Table 1. Experimental procedure for secondary recrystallization was as follows; Ingots were reheated at 1423 K and were hot-rolled to the thick-
ness of 1.8, 2.2, 2.7 and 3.85 (4.05) mm in the laboratory. The reduction of last two passes of hot-rolling were controlled such as 20 and 15% in order to secure same texture after hot rolling.

These specimens were annealed at 1 393 K in an atmosphere of 100% N₂. The specimens were cold-rolled to the same thickness of 0.335 mm with the reductions of 81.4, 84.8, 87.6 and 91.3 (91.7)%, respectively. Following the cold-rolling, the specimens were annealed in a wet atmosphere of 25% N₂ and 75% H₂ for decarburization and primary recrystallization. The annealing temperatures were 1 103, 1 113, 1 123 and 1 133 K. The specimen of the highest magnetic flux density (B₈ (T) in an applied field of 800 A/m) at the specific annealing temperature was examined. Nitrogen was injected up to around 0.020 mass% N₂ 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% N₂ and 75% H₂ atmosphere with the heating rate of 15 K/h up to 1 473 K and keeping for 20 h at 1 473 K under a 100% H₂ atmosphere for purification.

B₈-values were measured by the 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 central positions of the specimens were obtained by the complete {100} pole figures, and analyzed by the three-dimensional texture analysis by Vector method. The secondary textures were measured by back-reflection Laue diffraction method.

The intensity of any coincidence orientation, Sᵣ, for a nucleus orientation N is expressed as Iₛ Sᵣ. The intensities of S9 and S5 coincidence orientation to Goss (⟨110⟩{001}) and dispersed Goss (⟨661⟩{126}) orientation of primary textures, Iₛ S9 and Iₛ S5, were calculated.

3. Result and Discussion

3.1. Magnetic Properties and Secondary Recrystallization Textures

Figure 1(a) shows the relation between cold-rolling reduction and magnetic flux density (B₈). In case of grain oriented silicon steel, B₈-value corresponds to the sharpness of Goss texture. Unfortunately the specimen at cold rolling reduction 81.4% of material B had fine grains, i.e., poor secondary recrystallization. Material B has high B₈ at the highest cold rolling reduction (91.7%). But material A does not have so high B₈-value even at the highest cold reduction (91.3%).

Figure 2 shows textures of secondary recrystallized grains obtained by back-reflection Laue diffraction. The feature seems to be the same as B₈, i.e., in case of the lower cold-reduction, the specimen has large dispersion from Goss orientation. Main orientation of dispersed grains is near ⟨110⟩{227}(more accurately ⟨661⟩{126}). This orientation is rotated by 22.5° from Goss around the normal direction axis.

Figure 3 shows the secondary recrystallization microstructures having peculiar features.

Table 2 shows the size classification of secondary recrystallized grains.

Figure 4 shows the secondary textures for each grain size classification. Smaller grains have larger deviation from Goss and correspond to lower B₈. On the other hand, large grains are sharp Goss texture.

Three misorientation angles (α, β, and γ) with respect to ND (Normal direction), RD (Rolling direction) and TD

<table>
<thead>
<tr>
<th>Element</th>
<th>Material A</th>
<th>Material B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>3.25</td>
<td>3.25</td>
</tr>
<tr>
<td>C</td>
<td>0.017</td>
<td>0.056</td>
</tr>
<tr>
<td>Mn</td>
<td>0.09</td>
<td>0.09</td>
</tr>
<tr>
<td>Al</td>
<td>0.027</td>
<td>0.027</td>
</tr>
<tr>
<td>N</td>
<td>0.008</td>
<td>0.008</td>
</tr>
<tr>
<td>S</td>
<td>0.007</td>
<td>0.007</td>
</tr>
</tbody>
</table>

Table 1. Chemical compositions (mass%).

Table 2. Size classification of secondary recrystallized grains.
(Transverse direction) from Goss texture are defined as follows; ND ($\alpha$) is the angle formed by the longitudinal direction and the projection of the [001] on specimen surface, TD ($\beta$) is the angle of rotation of the specimen about the [001] from the equiangular position and RD ($\gamma$) is the tilt angle of the [001] out of specimen surface, respectively.\textsuperscript{20) Table 3 shows the averages of these misorientation angles ($\alpha$, $\beta$, and $\gamma$). Even if grain size classification is the same, misorientation angle is different, i.e., material A has larger misorientation angle than material B. Especially, in case of smallest grain size (1) as shown in Table 2, the average misorientation angles ($\alpha$ and $\beta$) from Goss of material A are rather large and its texture is almost \{110\}$_{227}$. This will be the reason why B8 between material A and B are different. This is very important and to be discussed later.

3.2. Primary Recrystallization Textures and Intensities of Coincidence Grains

Many investigations\textsuperscript{14–17,19) on the relationship between the primary and secondary recrystallization texture, especially, the sharpness of secondary recrystallization Goss and its intensity of coincidence boundaries in the primary texture, have been carried out. By applying vector method analysis, Harase \textit{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 $\Sigma 9$ (Goss texture) or $\Sigma 7$ (cube texture).\textsuperscript{19) 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;\textsuperscript{21)}

(a) $P_{CN}\Sigma 1$ (the product of $I_n$ (the intensity of nucleus orientation N) and $I_C\Sigma 1$ (intensity of $\Sigma 1$ coincidence ori-
It was reported, in case of Goss secondary recrystallization, that the nucleus orientation N should be smaller than certain values (J9 coincidence orientation) of Goss orientation and B8 (T), i.e., as the J9 coincidence orientation relation mutually, and J9 coincidence orientations for Goss and {411} are common. Therefore, in this case, not only Goss grain but also {411} could secondary-recrystallize due to the above-mentioned condition (c). However only Goss grains and dispersed Goss grains {110} have secondary-recrystallized, as already shown in Fig. 2. In addition, Table 4 shows the intensities (Ic) of Goss and {411} at both the surface and central layers quantitatively. The Ic of {411} are generally weak compared with one of Goss considering both the layers, i.e., due to the above-mentioned condition (b), this {411} might not secondary-recrystallize, even if Jc is very high. This can be explained by the condition of Harase’s proposal.

However, the B8-value difference in Fig. 1(b) between two materials (A, B), which mainly consists of the ratio of dispersed Goss grains in secondary recrystallized grains as shown in Fig. 2 and Fig. 4, cannot be explained yet. The reasons of this difference might be due to various possibilities. But all possibilities should be strongly connected with the primary textures because the quantity of the injected nitrogen (inhibitor intensity) and primary grain sizes (driving force) are the same. The relation between B8 values and other textural parameters have been investigated, expanding to the texture at the central layer.

As a result, two reasons may be proposed, one is the intensity of Jc of Goss and {411} which contain J9 coincidence orientations of Goss, i.e., as cold rolling reduction increases, the intensities of {111} and {411}, which are the major J9 coincidence orientations of Goss, increase.

Figure 6 shows intensity distributions of Jc of Goss in primary recrystallized specimens at surface layer and are characterized by Jc of Goss and {411} at the surface layer. In fact, the relation between Goss and {411} orientation is J9 coincidence orientation relation mutually, and J9 coincidence orientations for Goss and {411} are common. Therefore, in this case, not only Goss grain but also {411} could secondary-recrystallize due to the above-mentioned condition (b).

Table 4(a). Intensity of Goss ([110]001) and {411}(122) in the primary textures at the surface layer.

<table>
<thead>
<tr>
<th>Material</th>
<th>A</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduction</td>
<td>[110]001</td>
<td><a href="122">411</a></td>
</tr>
<tr>
<td>81.4%</td>
<td>0.701</td>
<td>1.076</td>
</tr>
<tr>
<td>84.8</td>
<td>0.483</td>
<td>0.233</td>
</tr>
<tr>
<td>87.6</td>
<td>0.271</td>
<td>0.219</td>
</tr>
<tr>
<td>91.3(91.7)</td>
<td>0.303</td>
<td>0.224</td>
</tr>
</tbody>
</table>

Table 4(b). Intensity of Goss ([110]001) and {411}(122) in the primary textures at the central layer.

<table>
<thead>
<tr>
<th>Material</th>
<th>A</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduction</td>
<td>[110]001</td>
<td><a href="122">411</a></td>
</tr>
<tr>
<td>81.4%</td>
<td>0.659</td>
<td>0.280</td>
</tr>
<tr>
<td>84.8</td>
<td>0.376</td>
<td>0.260</td>
</tr>
<tr>
<td>87.6</td>
<td>0.447</td>
<td>0.341</td>
</tr>
<tr>
<td>91.3(91.7)</td>
<td>0.477</td>
<td>0.350</td>
</tr>
</tbody>
</table>

Concerning the former reason, the deviation of {411} is sharper than one of {111} (112) around the normal direction axis. As the result, as {411} is stronger, the sharpness of Goss texture is enhanced. Figure 7 shows the relation between the intensity of Goss ([110]001) and {411} at the surface layer, and another is the Jc of dispersed Goss ([110]227) at the central layer.
intensity of \{411\}(148) and B8 and may explain this difference considerably seemingly. But as Goss grain grows at the expense of \(\Sigma 9\) coincidence grains, the textural feature of eroded grains (\(\Sigma 9\) coincidence grains of Goss orientation) could not be inherited. The reason has not been clarified yet.

Figure 8 shows intensity distributions of \(I_c\Sigma 5\) in primary recrystallized specimens at surface layer and is characterized by the intensity of dispersed Goss orientation (\{110\}(227)). In addition, Fig. 9 shows the quantitative relation between cold rolling reduction and the intensity of dispersed Goss orientation in primary recrystallization tex-

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**Fig. 5.** 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 91.3%.

(e), (f), (g) and (h): Material B with cold rolling reductions of 81.4, 84.8, 87.6 and 91.7%.
ture, and at the central layer the difference between material A and B can be found. Therefore, noticing the intensity of dispersed Goss orientation at the central layer, the latter reason is examined. Figure 10 shows the relation between B8 and the intensities of dispersed Goss orientation. At the surface layer (a), the difference between the relations still exits. However, as at the central layer (b) the considerably good correspondence of the relation for material A and B could be found, the texture not only at the surface layer but also at the other portion should be taken into account.

By the way, as the nuclei of secondary recrystallization of grain oriented silicon steel nucleate at the portion where
Fig. 7. Relation between intensity of $\{411\}$ and B8.

Fig. 8. Intensity distributions of $\{110\}S\Sigma5$ in primary recrystallized specimen at surface layer.
(a), (b), (c) and (d): Material A with cold rolling reductions of 81.4, 84.8, 87.6 and 91.3%.
(e), (f), (g) and (h): Material B with cold rolling reductions of 81.4, 84.8, 87.6 and 91.7%.
the intensity of the inhibitor substances, such as AlN, MnS and so on, deteriorates below a certain level, the dissolution and diffusion of inhibitor substances function as an important role. Therefore, if the secondary recrystallization temperature is high, this dissolution and diffusion happen in an instant. In fact, in case of the acquired inhibitor method, as the average of primary grain diameter is rather large (≈23 μm), the temperature of secondary recrystallization is higher than inherent inhibitor method.24) In addition, the interval between the start and the end of secondary recrystallization is shorter compared with the inherent inhibitor method.1,25,26) Accordingly, in case of the acquired inhibitor method, the viable nuclei of secondary recrystallization will nucleate not only at the surface layer but also at other portions of the sheets compared with the inherent inhibitor methods, i.e., the orientation of viable secondary nuclei will depend on the texture of other portions besides the surface layer.

The detailed analysis by using textures through the thickness should be carried out hereafter.

3.3. The Velocity of Σ5 and Σ9 Coincidence Boundaries

Concerning the velocity of grain growth, Nakayama et al. proposed the grain growth equation,27

\[ \frac{dR}{dt} = AME(1/Re - K/R - KZ/A) \]

where \( R \) = radius of secondary grain, \( A \) = form factor, \( M \) = mobility of a grain boundary, \( E \) = grain boundary energy, \( R_c \) = critical radius of primary grains, \( K \) = ratio of secondary grain boundary energy to primary grain boundary energy, \( Z \) = Zener term.
The total driving force consists of three components: the driving force derived from the grain boundary energy of primary grains, the retarding force from the shrinking force of the secondary grain, and the retarding force by the pinning effect of second phase particles.

Nakajima et al. investigated the grain boundary migration on coincidence grain boundary.28,29) On the other hand, Yoshitomi et al.26) proposed, by comparing the mobility (M) of $\Sigma 9$ and $\Sigma 5$ coincidence boundary, that the relative mobility might depend on the temperature, and at high temperature $M_{\Sigma 9}$ (mobility of $\Sigma 9$ coincidence grain boundary) would be as same as $M_{\Sigma 5}$.

Table 5 shows $Ic_{\Sigma 5}$ and $Ic_{\Sigma 9}$ of Goss and dispersed Goss orientation. Although $Ic_{\Sigma 5}$ of dispersed Goss of material B (91.6%) is highest, the secondary texture of dispersed Goss is very weak, and the size of sharp Goss secondary grains is larger than dispersed Goss as shown in Fig. 4. Therefore, the $\Sigma 9$ coincidence boundary can move much faster than $\Sigma 5$ coincidence boundary in this case.

Therefore, if $M_{\Sigma 9}$ would be as same as $M_{\Sigma 5}$, the difference of velocity might be caused by the grain boundary energy.27) Because in this study, $Z$ and $Rc$ in the above equation were controlled as same. In addition, at this high temperature (around 1323 K), as the mobility means physically the “self-diffusion coefficient”, $M_{\Sigma 9}=M_{\Sigma 5}$ and the reason of the velocity difference might depend on the energy for each coincidence grain boundary. The coincidence grain boundary is special in the grain boundary energy,30) especially $\Sigma 9$ coincidence grain boundary in the grain oriented silicon steel.31–34) In conclusion, the energy of $\Sigma 9$ coincidence boundary might be much lower than that of $\Sigma 5$.

3.4. Primary Recrystallization Texture Formation

Thus far, the relationship between primary and secondary texture has been discussed. In this section, the primary texture formation is discussed briefly. Primary textures depend on various metallurgical parameters, i.e., chemical compositions,35) texture36) and grain size37) before cold rolling, heat treatment before cold-rolling,38,39) roll diameter of cold rolling mill,40) temperature of cold rolling operation,41) cold rolling reduction,37,42) heating rate of annealing37) and so on. These parameters are related mutually.

Figure 11 shows the microstructure before cold rolling. Material A has coarse grains because of no $\alpha$-martensite transformation. This causes rather strong $\{110\}$, a little strong $\{100\}$ and a little weak $\{111\}$ in primary texture.

Figure 12 shows the intensity of Goss orientation in the primary texture. Larger grain size before cold rolling causes it stronger and higher cold rolling reduction causes it weaker. This is well-known in the field of primary recrystallization on flat steel products.37) Furthermore, in order to investigate the primary texture of grain oriented silicon steel, many studies have been carried out and reported by changing parameters such as cold-rolling temperature.41) But this kind of three dimensional analysis using such as Vector method has been very seldom.46)

In this study, it becomes clarified that the cold rolling reduction gives the similar effect to primary texture as that of C content. The increase of C content causes the grain size before cold rolling smaller. Therefore $Ic_{\Sigma 9}$ of Goss orientation (mainly $\{111\}$ connects $\langle 778 \rangle \{447\}$) increases as shown in Fig. 5 and Table 5, although the intensity of Goss orientation decreases as shown in Fig. 12. In addition, as cold rolling reduction increases, near $\{411\}$ ($\langle 148 \rangle$ connected with $\alpha$-fiber in bcc) also develops and Goss orientation decreases. As a result, because the preferable textural conditions are obtained, secondary recrystallized Goss orientation becomes sharper and its grain size becomes larger. But, in case of too high cold rolling reductions,16) Goss grains do not secondary-recrystallize any more because of small quantity of Goss nuclei in the primary texture according to Harase’s condition (b).21)

It will be summarized that the increases of C content and

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**Table 5**

<table>
<thead>
<tr>
<th>Material</th>
<th>$Ic_{\Sigma 5}$</th>
<th>$Ic_{\Sigma 9}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2.2mm</td>
<td>3.85 mm</td>
</tr>
<tr>
<td>B</td>
<td>2.2mm</td>
<td>4.105mm</td>
</tr>
</tbody>
</table>

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**Fig. 11.** Microstructures before cold rolling.

**Fig. 12.** Relation between cold rolling reduction and intensity of Goss.
cold rolling reduction enhance the $Ic\Sigma 9$ of Goss orientation, and secondary recrystallized Goss grains becomes sharper until the lower limit of the intensity of Goss orientation.

The preferable primary texture has become clear in case of acquired inhibitor method of grain oriented silicon steel. Hereafter the way of the formation of primary texture should be examined and the mechanism should be investigated with the aid of other steel products, e.g., mild steel, stainless steel and non-oriented electrical steel.

In addition, in case of the acquired inhibitor method of grain oriented silicon steel, as the primary texture, inhibitor condition and primary structure can be given separately, this kind of study can be easily carried out.

4. Conclusions

In order to investigate the relationship between primary and secondary recrystallization texture of grain oriented silicon steel, the specimens with various kinds of the primary texture were obtained by changing the cold rolling reduction and carbon content, and were secondary-recrystallized by the acquired inhibitor method. Furthermore in order to avoid the influence of inhibitor and estimate the pure effect of texture, the same final thickness was applied.

(1) For primary texture formation, C content and cold rolling reduction will function similarly, i.e., their increases will enhance the $Ic\Sigma 9$ of Goss orientation.

(2) The sharpness of Goss texture in the secondary recrystallization will depend mainly on the $Ic\Sigma 9$ of Goss orientation and partially on the texture besides other portions of the surface layer in the primary texture.

(3) Deviation from Goss orientation will be caused of the secondary recrystallized dispersed Goss orientation.

(4) $\Sigma 9$ coincidence boundary will move much faster than $\Sigma 5$ boundary. The reason might be that the grain boundary energy of $\Sigma 9$ will be much lower than that of $\Sigma 5$.

REFERENCES


