Influence of Primary Recrystallization Texture through Thickness to Secondary Texture on Grain Oriented Silicon Steel

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It is well known that the sharpness of secondary recrystallized Goss texture ([110](001)) is deteriorated by nitriding primary recrystallized sheet on the ordinary grain oriented silicon steel. The cause of this deterioration was investigated. It was found that secondary recrystallization of Dispersed Goss ([110](229)) caused the deterioration. From the surface to the central position in the primary recrystallization texture, the intensity of Goss Texture decreased and Dispersed Goss increased. Furthermore, nitriding increased the temperature of secondary recrystallization onset. As a result, the nucleation site of secondary recrystallization might shift to the central position where the circumstance that Dispersed Goss could grow easily is satisfied by the CSL model. This can be explained by using the CSL model with consideration of the intensity, S5 and S9 coincident orientation of Goss, and Dispersed Goss and assuming that S9 boundary is more mobile than S5 boundary.

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

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 the secondary recrystallization phenomenon. 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}(001) (Goss texture). Therefore, it is essential to enhance the sharpness of Goss texture.

The magnetic anisotropy of Fe-single crystal was discovered in 1926.2) In 1934, the principal production process on GO was invented by Goss.3–5) 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 Japan6–11) and the slab reheating temperature can be decreased from an extra high temperature to normal one,12) thus improving both quality and production technology.

The production technology can be classified into two categories.12) 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 disperse finely regardless of inhibitor kind. Another is the new nitriding technology, developed by Nippon Steel,12–14) 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”.

Nitriding technology consists of two methods.12) One is the nitriding by the reaction between the sheet surface and the atmosphere during secondary recrystallization annealing referred to as the “equilibrium nitriding method”. Another is the nitriding with NH3-containing atmosphere for short time after decarburization annealing referred to as the “non-equilibrium nitriding method”.

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 model12) (where coincident site lattice boundaries play a significant role) and the HE model (where high energy boundaries play a significant role15,16)). The CSL model has been employed for both inhibitor methods and the HE model has been mainly employed for inherent inhibitor method.

It is well known17–20) that the sharpness of Goss texture is deteriorated by nitriding the inherent material (ordinary GO). Yoshitomi et al.20) explained the cause by applying the CSL model as follows. The nitriding caused the secondary recrystallization temperature to decrease. At the lower temperature, the mobility of Σ5 coincident grain boundary is considered to be larger than that of Σ9 boundary. At the higher temperature they are considered to be the same level. Considering the product of the mobility for specific grain boundaries and the intensity of specific coincident grains,
dispersed Goss grain secondary recrystallizes more easily than Goss grain. In their study, the equilibrium nitriding method was applied.21)

The purpose of this study is to investigate the cause of the deterioration of Goss texture by considering the circumstances of secondary recrystallization nucleation site using the CSL model in the case of the non-equilibrium nitriding method of the ordinary GO.

2. Experimental Procedure

Table 1 shows the chemical composition of the specimens. The specimens, which were decarburized and primary-recrystallized, were prepared from ordinary commercial production22) of inherent inhibitor method, the main inhibitors being finely dispersed AlN and MnS. The thickness was 0.285 mm with cold rolling reduction of 87.6%. By non-equilibrium nitriding (ammonia) method, they were nitrided up to 5 content levels of nitrogen: 0.0077 (A: non-nitrided), 0.0119 (B), 0.0166 (C), 0.0206 (D), 0.0252 (E) mass%. The annealing separator, that mainly consists of MgO, was coated on the specimen surfaces. Two types of annealing were followed: one was full-cycle annealing in order to obtain the secondary recrystallization texture and the other was extraction annealing in order to investigate the process of secondary recrystallization.

2.1. Experiment 1 (Full-cycle Experiment)

The secondary recrystallization annealing was carried under a 25% N2 and 75% H2 atmosphere with a heating rate of 15 K/h up to 1473 K and maintained for 20 h at 1473 K under a 100% H2 atmosphere for purification. The specimens were 60 mm length (300 mm length) and the secondary (recrystallization) textures were obtained by back-reflection Laue diffraction method.

2.2. Experiment 2 (Extraction Experiment)

The secondary recrystallization annealing was carried under a 25% N2 and 75% H2 atmosphere with a heating rate of 15 K/h. The specimens were extracted at 8 temperature levels each 25 K from 1198 K to 1373 K. N and acid-soluble Al contents were analyzed, and the cross-sectional structure was observed. In order to clarify the secondary recrystallization behavior, B8 and W15/50 (core loss at 1.5 T and 50 Hz) were measured by SST (60 mm width, 220 mm length) instead of by eye observation. The difference of the length of SST was caused by the dimension of the extraction experiment apparatus.

2.3. Primary Recrystallization Texture

The primary textures were measured by X-ray diffraction method. Before all the experiments, the primary textures were obtained at 9 locations from one surface to another through the entire thickness by the complete {100} pole figures and analyzed by the three-dimensional texture analysis by Vector method.23) The thickness of the specimens were 60 μm. 9 specimens for one series were taken from each 10% position: 10.5, 21.1, 31.6, 42.1, 49.8, 57.9, 68.4, 78.9 and 89.5%. Three series were measured in order to improve the precision. The data was averaged at each position and turned down at the central position, i.e., the datum at the central position was the average of 3 data and the data at other positions were the average of 6 data. Furthermore, as Goss is the minor orientation, it was also estimated at the thickness of 10.5%, 49.8% and 89.5% positions by ODF, of which three pole figures were measured, namely \{110\}, \{100\} and \{211\}.

The intensity of any coincidence orientation, Si, for a nucleus orientation is expressed as S\text{is}(\{001\}), \text{dispersed }Goss (\{110\}(229)) and others orientation in primary textures, IA\Sigma 9, IC\Sigma 5, IA\Sigma 1 and P\Sigma 1 (the product of IN (the intensity of nucleus orientation N) and IA\Sigma 1 (the intensity of \Sigma 1 coincidence orientation)) were calculated.

3. Result and Discussion

3.1. Primary Recrystallization Texture

Figures 1, 2, and 3 show the three-dimensional orientation distributions of primary recrystallization texture, IA\Sigma 5 and IA\Sigma 9 at each position, respectively. No significant difference was found. The three-dimensional orientation distributions are characterized by \alpha-orientation (\{100\}(012)\sim\{411\}(148)\sim\{111\}(112)) and \gamma-orientation (\{111\}(011)\sim\{111\}(112)), that were named in this study, originated from \alpha-fiber (\{001\}(110)\sim\{112\}(110)\sim\{111\}(110)) and \gamma-fiber (\{111\}(011)\sim\{111\}(123)\sim\{111\}(112)), respectively.25–27) \gamma-orientation contains \{111\}(112), which is close to \Sigma 9 orientation for Goss. \alpha-orientation contains \{411\}(148), which is one of the main \Sigma 9 orientations for Goss.

\alpha-orientation at the surface position was weaker than at the central position. On the other hand, \gamma-orientation at the surface position was stronger than at the central position. This is caused by the texture of the hot rolling process.11) The distribution of IA\Sigma 5 was characterized by strong \{110\}(227) and the distribution of IA\Sigma 9 was characterized by strong \{411\}(122), \{447\}(110), \{110\}(223) and Goss, that were not so closely related to the thickness positions.

3.2. Full Cycle Experiment

Figure 4 shows the relationship between N-content and B8. B8 decreased (deteriorated) as N-content increased. This phenomenon has already been reported.17–20) B8 decreased drastically with a little addition of N (N = 0.0119%), but if N-content increased more, B8 did not decrease so much. To investigate the cause is the main theme of this study, and is discussed later.

Figure 5 shows the texture of secondary recrystallized specimens for each N-content. Non-nitrided condition gave

| Element | Mass% |
|---------|-------|
| O       | 0.001 |
| Si      | 3.23  |
| Mn      | 0.078 |
| S       | 0.026 |
| Al      | 0.029 |
| N       | 0.0072|
| Sn      | 0.07  |
| Cu      | 0.07  |
Fig. 1. Three dimensional orientation distributions of primary recrystallization texture (× random).
(a), (b), (c), (d) and (e): at the positions of 10.5, 21.1, 31.6, 42.1 and 49.8% thickness.

Fig. 2. Intensity distributions of $I_{C5}$ in primary recrystallized specimens.
(a), (b), (c), (d) and (e): at the positions of 10.5, 21.1, 31.6, 42.1 and 49.8% thickness.

Fig. 3. Intensity distributions of $I_{C9}$ in primary recrystallized specimens.
(a), (b), (c), (d) and (e): at the positions of 10.5, 21.1, 31.6, 42.1 and 49.8% thickness.
the sharp Goss orientation, but nitriding conditions gave larger misorientation. Figures 4 and 5 mean the same fact from the electrical and metallurgical points of view, respectively.

Figure 6 shows the macrostructures of secondary recrystallized grains. Secondary recrystallization was perfect even in case of low B8.

Table 2 shows the size classification of secondary recrystallized grains. Figure 7 shows secondary texture for each grain size classification obtained from Fig. 5. Larger class has sharper Goss orientation. This is the same phenomenon as the previous study, and the reason is that Σ9 boundary might move much faster than Σ5 boundary due to the difference of grain boundary energy.

Figure 8 shows the distribution of three misorientation angles from Goss. Three misorientation angles (α, β and γ) with respect to ND (Normal direction), RD (Rolling direction), and TD (Transverse direction) from Goss texture are defined as follows: ND(α) is the angle formed by the longitudinal (Rolling) direction and the projection of the [001] on specimen surface, TD(β) is the angle of rotation of the specimen about the [001] from the equiangular position, and RD(γ) is the tilt angle of the [001] out of specimen surface, respectively. The β and γ distributions with nitriding conditions were not so different from those with non-nitriding condition, and converged to zero (Goss). But α was dispersed widely. Therefore, the orientation of dispersed Goss was not defined but expressed as \{110\}(uvw): u=v, α≤37°).

### 3.3. Extraction Experiment

Figure 9 shows B8 and W15/50 at each temperature in the extraction experiment (all data were the average of 2 specimens). This indicates the secondary recrystallization behavior more accurately than eye observation because eye observation can cover only a small area of each specimen.

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**Table 2.** Size classification of secondary recrystallized grains.

| A       | B       | C       | D       | E       |
|---------|---------|---------|---------|---------|
| N: 0.0077% | N: 0.0119% | N: 0.0166% | N: 0.0206% | N: 0.0252% |

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Fig. 6. Macrostructure of secondary recrystallized grains.
On the contrary, this method can cover the entire specimen and give the average situation. B8 is sensitive to texture (orientation) and W15/50 is sensitive to grain size (grain growth). As at the same temperature, B8 and W15/50 began to change, it can be recognized that at the very temperature secondary recrystallization started. Namely, the temperature of secondary recrystallization outset (Tso) increased, as N content increased. This is different from the other studies in case of equilibrium method. To confirm the results, the same experiments were repeated twice. The results did not change.

Figure 10 shows the cross-sectional microstructure. At the surface layer, grain growth was observed as the temperature increased. In the case of higher N content, the grain growth was delayed. Figure 11 shows the grain size at surface layer quantitatively. As N content increased, the grain growth was delayed. Although, Yoshitomi et al. reported Tso decreased in case of equilibrium method, in this study (non-equilibrium nitriding method) Tso increased.

Figure 12 shows the change of N content and acid soluble Al. As N content increased, they were maintained at the higher level. This means that inhibition effect was kept

Fig. 7. {100} pole figures representig secondary recrystallized grains. (a), (b), (c) and (d): Grain size classification of D≤5 mm, D≤10 mm, D≤20 mm and D>20 mm.

Fig. 8. Distributions of deviation (α, β and γ) from Goss orien-taion. (a) α is the angle formed by the rolling direction and the projection of the [001] on specimen surface. (b) β is the angle of rotation of the specimen about the [001] from the equiangular position. (c) γ is the tilt angle of the [001] out of specimen surface.

Fig. 9. Secondary recrystal-lization behavior: Extraction temperature and magnetic properties (B8 and W15/50). (a) Extraction temperature and B8, (b) extraction temperature and W15/50.
Fig. 10. Microstructures in the extraction experiment (transverse section). (a) A (non-nitried, \(N=0.0077\%\)). (b) C \((N=0.0166\%\)). (c) E \((N=0.0252\%\)).

Fig. 11. Change of mean grain diameter at the surface layer.
However, I Goss was stronger than one of Goss, and on the contrary,  
Therefore, by applying ODF, the sharp Goss and nitriding conditions caused dispersed Goss crystallization was observed.  

Furthermore, by applying Vector method, the \( I_N \) (Goss) were already obtained in the inherent inhibitor method materials of Al-containing.\(^{32,33}\) In 2 of 3 cases, \( I_N \) (Goss) at the surface position was stronger than at the central position. Moreover, in case of two stages of cold rolling process, \( I_N \) of (Goss) at the surface position was stronger than one at the central position.\(^{34}\) 

On the other hand, the intensity of ND//{110} axis density through the thickness was already reported.\(^{35,36}\) Figure 14 shows the intensity of ND//{110} axis density calculated from Vector method. The feature was the same as the previous study, and the formation of Goss at the surface position originates from the shear stress at hot rolling.\(^{11}\) Therefore, as ND//{110} axis density includes the \( I_N \) (Goss), Figs. 13 and 14 do not contradict each other. 

The CSL model on G0 has become popular\(^{12,28,37-40}\) and many investigations on the relationship between the primary and secondary recrystallization texture, especially the sharpness of secondary recrystallized Goss and its intensity of coincidence boundaries in the primary texture, have been carried out. By applying vector method analysis, Harase et al.\(^{49}\) have proposed that Goss or cube secondary recrystallize 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). 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) 

\[
\text{(a) } P_{cN} \Sigma 1 \text{ (the product of } I_N \text{ (the intensity of nucleus orientation } N \text{) and } I_5 \Sigma 1 \text{ (intensity of } \Sigma 1 \text{ coincidence orientation)) \text{ of nucleus orientation } N \text{ should be smaller than certain values } (P_{cN} \Sigma 1_{cr}).} 
\]

\[
\text{(b) } I_N \text{ should be larger than certain critical values } (I_{Ncr}). 
\]

\[
\text{(c) Nucleus orientation } N \text{ should have a higher probability of coming in contact with mobile grain boundaries than other orientation.} 
\]

\[
\text{(d) } \Sigma 9 \text{ boundary might move much faster than } \Sigma 5 \text{ boundary due to the difference of boundary energy.}^{28} 
\]

3.5. Circumstance of Secondary Recrystallization

3.5.1. In Case of Non-nitriding

It is well known\(^{37}\) that the position of secondary nucleation is the around 20% depth of the total thickness and these nuclei erode the smaller grains and grow into central side in case of inherent inhibitor method. This is caused mainly by “inhibition effect gradient” at the surface position.\(^{35,31}\) Therefore, even if \( I_5 \Sigma 5 \) of dispersed Goss is stronger than one of Goss, as \( I_5 \Sigma 9 \) of Goss is stronger than one of dispersed Goss, Goss could secondary recrystallize more easily than dispersed Goss in the case of non-nitriding with basing the above-mentioned CSL model (\( \Sigma 9 \) boundary is more mobile than \( \Sigma 5 \) boundary). Namely, at the surface position, the circumstance of “Goss growth” might be prepared.

Furthermore, as the grain size is small (around 10 \( \mu \text{m} \))
compared with the thickness, this phenomena might be accelerated in the direction of the thickness, i.e., the driving force of grain boundary migration for secondary recrystallization might function more effectively in the direction of the thickness.

3.5.2. In Case of Nitriding

B8 is deteriorated by nitriding as already mentioned above (Fig. 3). Concerning this kind of phenomenon, Yoshitomi et al. proposed the mechanism that, in case of equilibrium nitriding method, the deterioration of B8 was caused by temperature-dependence of the mobility (M) for Σ5 and Σ9 boundaries. At the lower temperature, M(Σ5) is larger than M(Σ9), and at the higher temperature, M(Σ5) is nearly equal to M(Σ9). But it is unreasonable that at narrow range of Tso, such as 1223–1293 K, the relationship of M(Σ9) and M(Σ5) changes.

As already known, the position of secondary nucleation is the around 20% depth of the total thickness in case of inherent inhibitor method in case of non-nitriding. As shown in Fig. 11, by means of non-equilibrium nitriding the grain growth at the surface layer was “inhibited” and delayed until higher temperature. Therefore, the nucleation position of secondary recrystallization could shift into the central side and at a higher temperature, and nucleation might occur for a shorter time compared with a lower temperature and the selectivity for “Goss growth” might reduce.

The relative intensity of Goss against dispersed Goss de-
increased drastically at central side as Fig. 13. Even though the intensity of coincident orientations may be satisfied, the frequency of Goss grain is not sufficient. Due to the condition (b) of CSL model, dispersed Goss might secondary recrystallize more frequently instead of Goss. However, as this phenomenon is the probability process (the competitive phenomenon of the boundary migration), Goss can also secondary recrystallize partially.

Concerning the grain boundary migration of secondary recrystallization in the inherent inhibitor method, its velocity is slow and its behavior can easily be observed due to the lower Tso compared with the acquired inhibitor method. As its surrounding grains are still kept small and the nucleus of Goss grows at the cost of these small grains, the grain boundary migration might be influenced more by the texture circumstances.
In addition, Namajima et al.\(^{19}\) reported that in the case of the inherent inhibitor method, the deviation from Goss is accelerated by stronger inhibition force. However, the cause was not explained.

By non-equilibrium nitriding, the sharpness of Goss deteriorated. The reason might be that Tso increased and the nucleation position might shift to the central side. The temperature dependence of the relation for M(Σ5) and M(Σ9), as Y. Yoshimoto et al. proposed, is not necessary and to explain the cause it is enough to consider the CSL model including that Σ9 boundary is more mobile than Σ5 boundary.\(^{28}\) However, as the nitriding method of this study was different from that of the other studies, the cause for the sharpness deterioration of Goss could be different.

3.6. The Reason why Other Orientations, that had Strong \(i_{\Sigma 9}\), did not Secondary Recrystallize

So far, only the two orientations of Goss and dispersed Goss, that secondary recrystallized, were referred. Figure 3 shows the \(i_{\Sigma 9}\) distributions of each thickness. However, not only Goss but also \{411\}(122), \{447\}(110) and \{110\}(223) had strong \(i_{\Sigma 9}\). These orientations did not secondary recrystallize as shown in Fig. 5.

Figure 15 shows the \(I_{\Sigma 1}, I_{\Sigma 5}, I_{\Sigma 9}\) and \(P_{\Sigma} I_{\Sigma 1}\) of these orientations. Here, since dispersed Goss was not defined, half of the maximum value of \(e\) adopted as the representative value and it was defined with \{110\}(229), tentatively.

\(I_{\Sigma 1}, I_{\Sigma 5}, I_{\Sigma 9}\) and \(P_{\Sigma} I_{\Sigma 1}\) had similar aspects mutually other because \(I_{\Sigma 1}\) is also calculated by applying Brandon criterion.\(^{42}\) According to the CSL model, because \(I_{\Sigma 1}\) of \{411\}(122) and \{447\}(110) were strong, they will exceed the \(I_{\Sigma 1}\), which cannot be concretely specified for the time being. Furthermore, as \(I_{\Sigma 5}\) of \{411\}(122) and \{447\}(110) were weak compared with other orientations, they will have a disadvantage for secondary recrystallization.

However, \{110\}(223) had a quite similar aspect to Goss. Only with CSL model, the reason that \{110\}(223) did not secondary recrystallize cannot be explained. The reasons why only “special Σ9 boundary of Goss” can easily migrate cannot be found in the only CSL model. Other possible causes, for example the contribution of residual strain and the shape (that might be different by orientations), will be considered. For the future, the essential reasons why Goss and dispersed Goss can easily secondary recrystallize will be investigated in addition to the CSL model.

4. Conclusion

By non-equilibrium nitriding the ordinary GO, the sharpness of Goss orientation deteriorates. The reason was investigated. \(I_{\Sigma 9}\) of Goss in the primary texture decreased in the direction to the central position, and by nitriding, Tso increased. Therefore, the nucleation site of secondary recrystallization might shift to the central position where the circumstance that dispersed Goss grew might be easily satisfied. The temperature dependence of M(Σ5) and M(Σ9) is unnecessary, as proposed before, and it is enough to consider the \(I_{\Sigma 9}\) of Goss and dispersed Goss in the CSL model including that Σ9 boundary is more mobile than Σ5 boundary.

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