Evolution of Texture and Related Magnetic Properties in Fe–3%Si Steel during Single-step Annealing

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Cold rolled Fe–3%Si steel sheets underwent a single-step annealing process, which included a decarburizing treatment. Crystallographic texture and magnetic properties were investigated after cold rolling and annealing for various times. Most of the Goss oriented grains were situated inside the shear bands of (111)(112) oriented grains in the cold rolled samples. In the annealed samples, island grains were observed in the abnormally growing grains. The island grains had either low (<15°) or high (>45°) misorientations. The abnormally growing grains were surrounded by 25°–45° misorientation boundaries. The effects of crystallographic texture on magnetic properties were evaluated using a texture parameter, the ratio of the sum of Cube and Goss texture components fractions to γ fiber texture components fraction. The results show that iron losses decrease linearly with increasing texture parameter.

KEY WORDS: silicon steel; annealing; iron losses.

1. Introduction

The magnetic properties of silicon steels are improved by a control of crystallographic texture, grain size and composition. Grain Oriented (GO) Fe–3%Si steel is commonly used as core material most for transformers requiring high permeability and low iron loss. The favorable magnetic properties of GO Fe–3%Si steels are achieved as a result of their high resistivity and the preferred crystallographic orientation of grain. In GO steels most grains have a Goss, {110}/(001), orientation. The oriented grains improve the magnetic properties of the steel because the easy magnetization direction (001) of the grains is parallel to the magnetic field direction. Dunn (1949) found that the sharp Goss texture develops due to abnormal Goss oriented grain growth during secondary recrystallization annealing process. Despite the mass production of GO silicon steels with good magnetic properties, the mechanism of the Goss texture development is still not well understood.

Recrystallization has long been recognized as being both of technological importance and scientific interest. The origin of recrystallization texture has been explained by two theories based on the fact that the recrystallization texture is primarily derived from the crystallographic orientation of nuclei and the growth rate of these nuclei into the deformed matrix. The first theory, oriented nucleation theory, claims that nuclei of specific orientations originate more rapidly than those of other orientations and the orientation of these nuclei determine the final recrystallization texture of the material. The second theory, oriented growth theory, considers that nuclei are formed with many orientations, and those nuclei which have specific boundary characteristic with respect to the surrounding matrix will grow faster, and thus dominate the final texture. Both theories have been investigated by numerous experimental works and the modeling of the recrystallization kinetics, but neither theory can explain all of the observed phenomena clearly.

Various theories of preferential growth of Goss texture in GO Fe–3%Si steel have been proposed. They are essentially derived from either the oriented nucleation or the oriented growth theory. The studies in favor of the oriented nucleation theory are based on the stored energy of the deformed material. Nucleation starts in the grains having the highest stored energy. These grains then grow into the grains having lower stored energy. The studies based on the oriented growth theory focus on the role of the grain boundary misorientation. Specific coincidence site lattice (CSL) boundaries such as Σ9, Σ7, Σ11, Σ5 boundaries and boundaries with a misorientation angle between 20° and 45° have been related to the abnormal grain growth. In the oriented growth theories it is assumed that Goss oriented grains are surrounded by a larger number of such high mobility boundaries. Other mechanisms have been proposed such as the strain-energy-release maximization (SERM) model of Lee and Jeong but the mechanism of selective Goss texture development during the secondary recrystallization of FeSi steel has not yet been fully identified.

The manufacturing process of GO steel to achieve steel with a favorable texture with a sharp Goss texture is decided complicated. In industrial practice, the final annealing process, during which the Goss oriented grains grow abnormally, consists of two annealing processes. First, a primary recrystallization annealing is performed at 800–
900°C in a wet hydrogen atmosphere to decarburize the steel. The carbon content goes from 0.03% to 20–30 ppm during this process. A secondary recrystallization annealing follows at 1 150–1 200°C in a dry hydrogen atmosphere to achieve the preferred growth of Goss oriented grains. Goss oriented grains nucleate during the primary annealing process and grow selectively during the secondary annealing process.

In the present work, it was attempted to anneal cold rolled Fe–3%Si steels using a single-step annealing process which combined both primary and secondary annealing processes. In order to estimate the nucleation mechanism of Goss oriented grains the microtexture of cold rolled Fe–3%Si steel was investigated by means of Electron Backscatter Diffraction (EBSD) technique prior to the annealing process. Then, the single-step annealing process was carried out using various annealing times. The changes in room temperature texture and magnetic properties were measured as a function of the annealing time. In addition, two samples, with different grain sizes and major texture component intensities prior to cold rolling, were studied in order to investigate the effect of the starting grain size and texture component intensities on the texture development and magnetic properties after cold rolling and single-step annealing. The grain size and texture component intensities of the samples were controlled by a normalizing treatment before cold rolling.

The annealing texture development was analyzed in detail during the annealing of a Fe–3%Si steel performed at the scheduled temperature for various annealing times. In addition, influence of the texture on the magnetic properties in the annealed steels was quantified by means of a recently proposed texture parameter, the texture factor (TF).14)

2. Experimental Procedure

The steels used in the present study were two cold rolled Fe–3%Si steels having different texture intensities. The difference in texture intensities between two steels were achieved by different normalizing conditions after the hot rolling. Prior to cold rolling, the hot rolled steels were normalized at 1 100°C for 5 and 100 min. The 5 and 100 min normalized samples will be addressed as sample A and B, respectively. The average grain sizes at the surface and in the center of a metallographic cross section of the sample A were 52.0 μm and 73.4 μm, respectively. For sample B 58.0 μm and 90.5 μm were obtained.15) Both samples A and B were cold rolled to 0.29 mm in thickness equivalent to reduction of 87%. Following the cold rolling, the single-step annealing process shown in Fig. 1 was carried out. The process combines both a decarburization and a grain growth segment. The heat treatment was done at 1 100°C in a tube furnace under a controlled atmosphere of wet 5%H2–95%N2 and dry H2. The annealing time varied from 1 to 80 h.

The microstructure and microtexture of the cold rolled and annealed samples were examined by means of EBSD. The diffraction pattern data were obtained and analyzed using Orientation Imaging Microscopy (OIM 5.1) TSL software in order to estimate individual orientation and misorientation relationship between neighboring grains in the samples. To obtain a complete description of the sheet microtexture RD-TD, RD-ND and TD-ND sections were investigated, where RD, TD and ND are the rolling, transverse and normal directions, respectively. The macrotexture analysis of the annealed samples was carried out with a Rigaku D-Max 3A X-ray Diffractometer (XRD) using Mo Kα radiation of a wavelength of 0.709 Å. Orientation Distribution Functions (ODFs) were calculated from the {110}, {200} and {211} incomplete pole figures using the Tex-Tools software. In addition, magnetic property measurements of the annealed samples were performed on a BROCKHAUS MPG 100D using a single sheet test method. Iron loss (W17/50) and magnetic flux density (B10) were measured at 50 Hz and compared to standard in GO Fe–3%Si steel. The effect of texture on the iron loss was evaluated by using a recently proposed texture parameter, the texture factor (TF).14)

Texture factor = TF = \( \frac{V_{\{100\}(001)} + V_{\{110\}(000)}}{V_{\{111\}/ND}} \) ......(1)

Where \( V_{\{100\}(001)} \), \( V_{\{110\}(000)} \) and \( V_{\{111\}/ND} \) are the volume fraction of \{100\}(001) Cube oriented grains, the volume fraction of \{110\}(001) Goss oriented grains, and the volume fraction of grains with \{111\}/ND γ-fiber orientation, respectively. The texture factor thought to be closely related to the magnetization behavior of Cube, Goss and γ-fiber texture components. Cube and Goss texture components are well known to have good magnetic properties because the (001) directions of easy magnetization are easily oriented parallel to the applied field. γ-fiber components are considered to have undesirable magnetic properties. The ODFs calculated from the XRD pole figure data obtained for a large sample volume were used for the assessment of the orientation volume fractions \( V_{\{100\}(001)} \), \( V_{\{110\}(000)} \) and \( V_{\{111\}/ND} \) in the texture factor formula. The volume fraction of γ-fiber textures, \( V_{\{111\}/ND} \), was taken as the sum of the four major texture component of the γ-fiber (111)(1[10], (111)[21], (111)[01]) and (111)[112].

3. Results and Discussion

Figure 2 shows the grain size of the cold rolled samples A and B after different annealing times at 1 100°C. The mean linear intercept in the maximum principal strain direction (RD) of grains, which was parallel to the magnetic
field direction for the determination of the magnetic properties, was used as the grain size in the present work. The grain sizes of both samples A and B increased with increasing annealing time. The grain sizes increased continuously in the initial 20 h. Thereafter the grain size increased very slowly. In the early stages of the grain growth, the grain sizes of sample B were larger than the grain sizes of the sample A. The larger grain size in the sample B is very likely related to their large initial grains before cold rolling and the presence of coarse AlN precipitates in the steels due to the long time normalization of the hot rolled steel. After 10 h and at the later stage of the annealing process, the sample B had smaller grain sizes than those in the sample A. The previous study by Chang\textsuperscript{16)} has shown that normalizing of hot rolled steel leads to the coarsening of the MnS and AlN precipitates. Coarse precipitates provide only a weak barrier to primary and secondary recrystallization in GO electrical steel. From the previous work and the present observations, it is obvious that different sizes and dispersions of precipitate particles affect the driving force for secondary recrystallization in Fe–3%Si steel. Further work is definitely needed in this regard but the possible influence of precipitates size and distribution on the texture development and magnetic properties was not considered in the present work.

The macrotexture development in annealed Fe–3%Si steel is shown in Table 1 and Fig. 3. The volume fractions of γ-fiber texture components in both samples A and B decreased abruptly in the initial 10 h of annealing and remained approximately constant thereafter as can be seen in Fig. 3(a). This γ-fiber texture weakening is favorable for the magnetic properties in silicon steels. In contrast, the Goss texture component was considerably strengthened in the first 10 h of annealing and slightly weakened after 10 h annealing as shown in Fig. 3(b). The volume fraction profiles of γ-fiber and Goss texture along annealing time were dif-

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### Table 1. Crystal orientation volume percentages of major texture components in Fe–3%Si steel annealed at 1100°C.

| Texture  | Goss (001) | Cube (111) | γ-fiber (111) | Others |
|----------|-----------|------------|---------------|--------|
| Orientation | (011)[100] | (010)[100] | (111)[112] | (111)[110] |
| Sample A (5 min normalized steels) | | | | |
| Annealing time | | | | |
| 1 hr | 2.09 | 2.22 | 5.27 | 6.66 | 3.58 | 3.37 | 3.20 | 3.11 |
| 3 hrs | 4.54 | 2.11 | 6.20 | 5.08 | 3.48 | 3.69 | 2.78 | 2.86 |
| 10 hrs | 4.90 | 2.34 | 4.02 | 3.43 | 2.91 | 3.02 | 3.21 | 2.94 |
| 20 hrs | 4.86 | 2.23 | 3.84 | 4.10 | 2.90 | 2.94 | 3.12 | 2.96 |
| 80 hrs | 4.72 | 2.09 | 3.99 | 3.99 | 3.09 | 3.14 | 2.73 | 2.85 |

| Sample B (100 min normalized steels) | | | | |
| Annealing time | | | | |
| 1 hr | 2.06 | 2.28 | 7.69 | 6.21 | 4.46 | 4.54 | 2.61 | 2.82 |
| 3 hrs | 3.10 | 2.17 | 4.39 | 5.16 | 3.33 | 2.92 | 2.85 | 2.89 |
| 10 hrs | 5.20 | 2.35 | 4.01 | 3.88 | 2.95 | 2.86 | 3.17 | 3.02 |
| 20 hrs | 4.58 | 2.16 | 3.76 | 4.11 | 2.86 | 2.98 | 2.99 | 3.12 |
| 80 hrs | 4.01 | 2.14 | 4.06 | 3.82 | 2.80 | 2.70 | 3.14 | 2.58 |
These observations revealed that the orientation relation, a 35° rotation around the growth relationship between Goss and \{111\} texture in the cold rolled sample due to nearly the same fast process may have been caused by this stronger \{111\} served in sample A in the early stage of the annealing work,\(^{15}\) the \{112\}\{110\} rates in bi-crystal experiments.\(^5\) According to previous orientation relation of 27°/H\(^{20855}\) \{110\}\{112\}\{111\\} texture component as can be seen in Table 1. A strong \{112\}\{110\} rolling texture developed during cold rolling is known to transform to a sharp \{111\}\{112\} recrystallization texture due to their orientation relationship, a 35° rotation around the \{110\} transverse direction.\(^{17}\) This orientation relationship is close to the ideal orientation relation of 27° \{110\} found for high growth rates in bi-crystal experiments.\(^5\) According to previous work,\(^{15}\) the \{112\}\{110\} texture component in the cold rolled sample B had a 26% higher intensity than in sample A. As a result sample B developed a more pronounced \{111\}\{112\} orientation intensity. Furthermore, the difference in the amount of Goss texture components in the annealed steels was found to be related to the different intensities of \{111\}\{112\} texture components in the cold rolled steels. A 10% higher intensity of \{111\}\{112\} was obtained in cold rolled sample A.\(^{15}\) The stronger Goss texture observed in sample A in the early stage of the annealing process may have been caused by this stronger \{111\}\{112\} texture in the cold rolled sample due to nearly the same fast growth relationship between Goss and \{111\}\{112\} orientation, a 35° rotation around the \{110\} transverse direction. These observations revealed that the orientation relationship between deformed and recrystallized grains, which is a 35° \{110\} between deformed \{112\}\{110\} and recrystallized \{111\}\{112\} oriented grains, and, between deformed \{111\}\{112\} and recrystallized \{110\}\{001\} oriented grains, may play a role in the texture evolution in Fe–3%Si steel during annealing process.

The evolution of the microstructure and microtexture of Fe–3%Si steel during annealing was studied by EBSD. For better understanding of the nucleation mechanism of Goss oriented grains, the microtexture of the cold rolled Fe–3%Si steel was first investigated prior to the annealing process. Figure 4 shows image quality (IQ) and crystal orientation maps of the cold rolled steel. The IQ value is used to indicate the perfection of the crystal lattice and any distortion in the crystal lattice will reduce the IQ value. As a consequence, a low IQ value implies that the crystal lattice is locally more severely deformed. The dark areas in Figs. 4(a) and 4(b) have a low IQ value. These areas are more deformed, contain more dislocations and have higher stored energies. The majority of the Goss oriented grains were found in highly deformed areas and located in shear bands in \{111\}\{112\} oriented grains as shown in Figs. 4(b) and 4(c). A recent study by Dorner et al.\(^{18}\) has shown that Goss oriented regions exist either in microbands of the cold rolled steel or in shear bands within \{111\}\{112\} oriented grains after cold rolling of FeSi single crystal with an initial Goss orientation. In polycrystalline Fe–3%Si steel used in the present work, however, Goss grains were observed only in shear bands in \{111\}\{112\} oriented grains. This observation is in agreement with those made by Park and Szpunar\(^{19}\) after primary recrystallization annealing of Fe–2%Si steel. As Goss oriented grains in deformed and primary recrystallized steel are present in the same location, it is assumed that the Goss oriented grains observed in the present study may play an important role in Goss orientation nucleation phenomena during the annealing process.

Figure 5 shows the microstructures in the RD-TD surfaces of annealed Fe–3%Si steels. The images were produced by plotting reconstructed boundaries of the automated EBSD scans. Abnormal grain growth was investigated in sample A after annealing for 1 h at 1100°C, as shown in Fig. 5(a). It is clear from this observation that the primary recrystallization was completed and the secondary recrystallization started after less than 1 h annealing at 1100°C. The abnormally large grains were continuously observed in the annealed Fe–3%Si steel as shown in the Goss oriented grains in Figs. 5(b) and 5(c). In the sample annealed for 20 h, however, normal grain growth was found to be more predominant than abnormal grain growth. The
presented in annealed Fe–3%Si steels, and the all grains. The results are a grain size 30 times larger than an average grain size in the separately for the island grains, the large grains, which had misorientation angle distributions were studied between grain boundary mobility and boundary misorientations. In order to understand the correlation between these factors of annealed Fe–3%Si steels are shown in Table 1.

The characteristics of the grain boundaries around the island grains in growing grains and around the large growing grains were significant different from those of the other grains. The large growing grains were observed in the annealed Fe–3%Si steel, as discussed earlier. Inside the growing grains island grains, similar to the one indicated in Fig. 5(a), were occasionally detected. Island grains were generally present in those parts of the growing grains close to grain boundaries with a lower mobility compared to the other boundaries around the growing grain. The schematic of Fig. 6 shows how an island grain is generate inside a growing grain. A protruding grain such as to one indicated in Fig. 5(a) may possibly have become an island grain after further annealing. In order to understand the correlation between grain boundary mobility and boundary misorientation angle, misorientation angle distributions were studied separately for the island grains, the large grains, which had a grain size 30 times larger than an average grain size in the annealed Fe–3%Si steels, and the all grains. The results are presented in Fig. 7. The misorientation angle distribution for all the grains was similar to a Mackenzie plot for the case of uncorrelated, random orientation. The island grains and the large grains had strong intensities in specific misorientation angle ranges. Most of the island grains were surrounded by either low (≤15°) or high (>45°) angle grain boundaries. A large volume fraction of misorientation angles between 25° and 45° were detected around the large grains. These observations suggest that the 25°–45° misoriented boundaries have a high mobility, while low (≤15°) and high (>45°) misoriented boundaries have a low mobil-
sharply after a 10 h annealing. Beyond 10 h the iron loss remained constant or increased slightly. In contrast, texture factors of the steels increased in the initial stages of the annealing process and gradually decreased as the annealing process proceeded. The texture factor profiles along annealing time were nearly identical for both steel A and B even though the individual orientation profiles were different as discussed earlier. It may result in almost the same iron loss profiles along annealing time in the steels. Figure 9(b) represents the relationship between iron loss and texture factor (b).

4. Conclusions

The evolution of the texture and the magnetic properties of Fe–3%Si steel after a single-step annealing procedure combining both decarburization and abnormal grain growth, were investigated. The main conclusions of the present work are as follows:

1. Differences of texture component intensity in cold rolled Fe–3%Si steels were found to have an influence on the texture development during the recrystallization process. The cold rolled steel which had a high intensity for \{112\}(110) and a low intensity for \{111\}(112), developed a strong \{111\}(112) intensity and a weak \{110\}(001) intensity in the initial stages of the recrystallization annealing. The texture development is believed to be due to the orientation relationship between \{112\}(110) and \{111\}(112), and \{111\}(112) and \{110\}(001). In both case, the texture components are related by a 35° rotation around the \{110\} direction.

2. The Goss oriented grains are mainly found in shear bands in \{111\}(112) oriented grains in cold rolled Fe–3%Si steel.
(3) Grain boundary characteristics play an important role in the abnormal grain growth phenomenon in Fe–3%Si steels. Island grains were observed to have either low (<15°) or high (>45°) misorientations. On the other hand, large growing grains including Goss oriented grains were surrounded by intermediate 25°–45° misorientation boundaries.

(4) Abnormally large grains observed in the cross section of the annealed Fe–3%Si steel showed a gradual rotation of orientation. These local rotations were supposed to lead to subgrain growth during further annealing process. It may be related to the abnormal grain growth along the sample thickness in the Fe–3%Si steel.

(5) Iron losses of the annealed Fe–3%Si steel were shown to be affected related to a recently proposed texture parameter, the texture factor, which is expressed as the ratio of a sum of Goss and Cube texture to γ-fiber texture volume fractions. The result showed that iron losses were inversely proportional to the texture factor.

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