Distribution of \{1\bar{1}0\}<001> Oriented Grains in the Primary Recrystallized 3% Si–Fe Alloy*

By Jirou Harase** and Ryo Shimizu**

The distribution of \{1\bar{1}0\}<001> oriented grains in the primary recrystallized 3% Si–Fe alloy processed by the one-stage cold rolling method were investigated by SEM-ECC-ECP technique and the X-ray diffraction method. The main findings were in the following:

1. \{1\bar{1}0\}<001> oriented grains present below 30 \mu m from the surface of the primary recrystallized specimen were not among the largest grains nor were present as an aggregate. The frequency of the \{1\bar{1}0\} grains rotated about 0.35 rad around the ND axis had the highest frequency among the \{1\bar{1}0\} oriented grains and the frequency of an ideal Goss orientation is less than 1/3 of these orientations. Orientations of the extraordinarily large grains were similar to the major textural component of the primary recrystallized specimen.

2. Among the \{1\bar{1}0\} oriented grains, the Goss oriented grain had the highest frequency of coincidence oriented grains in the grains directly surrounding to these \{1\bar{1}0\} oriented grains and had a very low frequency of the \Sigma 1 boundary in relation to its orientation in the surface of the primary recrystallized matrix, while the large grains above mentioned had a rather higher frequency of the \Sigma 1 boundary in relation to its orientation.

3. The mechanism of the evolution of the Goss texture by secondary recrystallization can be explained, even if the Goss nucleus is not the largest at the completion of the primary recrystallization, by considering the special distribution of coincidence boundaries and their specific grain boundary migration characteristics associated with the intensity of the inhibitor.

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I. Introduction

Grain oriented silicon steels are characterized by the presence of a sharp \{1\bar{1}0\}<100> texture, i.e. the Goss texture\(^{(1)}\). The deviation angle of <100> axis of grains from the rolling direction is smaller than 10\(^{\circ}\) for commercially produced silicon steels. Although many investigations have been made in the decades to clarify the mechanism of the evolution of the Goss texture\(^{(1)}\)–\(^{(10)}\), it is not fully understood why such a sharp Goss texture is evolved by secondary recrystallization. One stream of the investigation has been focused on the Goss nucleus in the primary texture\(^{(9)}\)–\(^{(11)}\), another stream of the investigation has been associated with inhibitors for secondary recrystallization\(^{(9)}\)–\(^{(10)}\). The present authors investigated the distribution of Goss nucleus and the orientation of the grains having large grain size in the surface layer of the primary recrystallized specimen by SEM-ECC-ECP technique and discussed the formation mechanism of Goss texture by secondary recrystallization considering the intensity of the inhibitor and the distribution of the coincidence boundaries in the primary recrystallized specimen.

II. Experimental Procedure

The starting material was 2.3 mm thick hot rolled 3% silicon steel sheet with the following chemical compositions: C:0.04, Si:2.90, Mn:0.09, S:0.027, Al:0.026, N:0.007 mass%. The primary recrystallized specimen was prepared following the one-stage cold rolling...
method developed by Taguchi et al.\(^{12}\) as follows:

Hot Band (2.3 mm) → Annealing (1393 K–150 s) → W.Q (373 K boiling water) → Cold Rolling (0.3 mm) → Annealing (1039 K–150 s, dew point-333 K).

1. **Investigation by SEM-ECC-ECP technique**

The surface layer of the thickness of about 30 μm/one side was taken by chemical polishing (1/10 of the total thickness) and this newly made surface was utilized for the SEM-ECC-ECP investigation (specimen A). The grain size distribution of this surface was investigated by an image analyzer (Tospix II). The total number of grains utilized for the grain size distribution measurement were 789 grains. Then a square of 10 mm × 10 mm was scribed on this surface so that the one side of the square lay parallel to the rolling direction. Then grains with \{110\}<uvw> orientation were scanned by the ECP mode in this area at each 100 μm interval along the TD direction. As the average grain size of the specimen is about 8 μm and the pattern is generated from about 5–10 μm in diameter, 5–10% of the 10 mm × 10 mm square is scanned by ECP by scanning at each 100 μm interval. In the selection of patterns, all the grains of \{110\}<uvw> orientations in which \{110\} planes less than 0.17 rad deviated from either the TD or RD axis are chosen and for the rotation angle around the ND axis every angles were selected. When the \{110\}<uvw> grain was detected by ECP, its grain size was also investigated. 45 grains were arbitrarily chosen from the \{110\}<uvw> oriented grains detected by ECP and the orientations of all the grains surrounding these grains were investigated. The coincidence orientation relationship between the surrounding grains and these \{110\}<uvw> oriented grains were also investigated. Brandon’s condition\(^{13}\) is used for the criterion of the coincidence orientation relationship. Then grains larger than 20 μm in diameter were scanned by the ECC mode under magnifications of ×500 in the same manner as scanned by ECP mode. In this manner all the area of 10 mm × 10 mm can be scanned. The orientations of these grains were analyzed by ECP. Among these grains, 6 extraordinarily large grains were chosen and the orientation of the surrounding grains and their orientation relationship with these grains were investigated. The scanning electron microscope used was JSM-840. ECP was taken in the following condition; accelerating voltage: 35 kV, probe current: 9 × 10\(^{-9}\) A, working distance: 8 mm, rocking angle: ±0.14 rad.

2. **Investigation by X-ray measurements**

The specimen with 80 μm thick including surface was taken for the texture analysis by X-rays (specimen B). Transmission and reflection (100) pole figures were made from this specimen and the complete (100) pole figure was made from them. This pole figure was inverted by the vector method\(^{14}\) and the intensity distribution of \{110\}<001> grains rotated around the ND axis was obtained by the vector method. In order to compare the orientation measured by ECP with that measured by X-rays, the inverse pole figures seen from the ND and the RD direction were made by the vector method. The details of the method of making the RD inverse pole figure are shown elsewhere\(^{15}\).

III. **Results**

1. **Texture measurements by X-ray**

Figure 1(a) and (b) show the structure and the grain size distribution of specimen A. The structure is rewritten for processing by the image analyzer (Tospix II). The grain size distribution was obtained from these structure map consisting of 789 grains by the image analyzer. The average grain size is 7.5 μm and that no grains 5 times larger (=37.5 μm) than the average grain size is present in this distribution figure.

Figure 2(a) and (b) show (100) pole figure and the inverse pole figure calculated by the vector method of the specimen B. It can be seen that the (100) pole figure is a typical pole figure of a primary recrystallized 3% Si-Fe processed by the one-stage cold rolling method. The major textural components are
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Figure 3 shows the positions of \{110\}<uvw> oriented grains detected by ECP below 30 µm from the surface of the primary recrystallized specimen (specimen A). It can be seen that they are rather uniformly distributed. The total numbers of grains detected were 215. The maximum rotation angle of \{110\} grains from the TD and RD axis was 0.13 and 0.19 rad respectively. Among the \{110\}<uvw> oriented grains detected, grains in contact with each other were only three cases and all other \{110\}<uvw>

\{111\}\{112\} and that the \{110\}<uvw> is a minor component.

2. The distribution of \{110\} grains in the specimen A

Figure 3 shows the positions of \{110\}<uvw> oriented grains detected by ECP below 30 µm from the surface of the primary recrystallized specimen (specimen A). It can be seen that they are rather uniformly distributed. The total numbers of grains detected were 215. The maximum rotation angle of \{110\} grains from the TD and RD axis was 0.13 and 0.19 rad respectively. Among the \{110\}<uvw> oriented grains detected, grains in contact with each other were only three cases and all other \{110\}<uvw>
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orientated grains were present not in contact with each other.

Figure 4 shows grain size and the frequency of (110) grains around the ND axis classified by the rotation angle of each 0.087 rad obtained by ECP together with the intensity distribution obtained by the vector method. As the {110} planes include the planes rotated around the TD or RD axis up to 0.11 rad in the case of the vector method, the grains of {110} planes measured by ECP utilized for this analysis are selected to include the same planes as in the vector method. Frequency distribution obtained by the vector method and ECP is normalized so as to compare them. It can be seen from this figure that the shape of the intensity distribution by the vector method and by ECP is very similar suggesting that the grain size and the distribution of {110} oriented grains detected by ECP represents the overall characteristics of the distribution of the {110} oriented grains at the 30 µm below the surface layer of the primary recrystallized specimen. This result also suggests that the size of the grains having an ideal Goss orientation or nearly ideal Goss orientation (they are orientations of the secondary recrystallized specimen) is not particularly large, although they are slightly larger than the average grain size and they do not exist as colonies. Among the {110} oriented grains, the grain rotated about 0.35 rad around the ND axis has the highest intensity and that the intensity of an ideal Goss orientation is less than 1/3 of the maximum intensity.

3. Orientation and rotation relationship of the grains directly surrounding (110) oriented grains

45 grains were arbitrarily chosen from the {110} oriented grains detected by ECP. The orientation and rotation relationship of the grains directly surrounding these grains were investigated by ECP. The number of these grains was 390. Figure 5 shows orientation of 9 grains of nearly ideal Goss orientation and the surrounding grains. Although the number of the grains investigated is small, it can be seen from this figure that the orientations of the surrounding grains are similar to those surround-
ing large grains (Fig. 9). Figure 6 shows frequency of coincidence boundaries between the \{110\} oriented grain and its surrounding grains. The \{110\} oriented grains investigated are 45 grains as mentioned above. It can be seen from this figure that the frequency of \(\Sigma 5\), \(\Sigma 9\), \(\Sigma 19\) and \(\Sigma 45\) are relatively high (except \(\Sigma 1\)). Figure 7 shows the frequency of coincidence boundaries between the nearly ideal Goss oriented gains (deviation angle of the \(\langle 100\rangle\) axis is less than 0.087 rad) and their surrounding grains, and the \{110\} oriented grains largely deviated from the ideal Goss orientation (deviation angle of the \(\langle 100\rangle\) axis is larger than 0.28 rad) and their surrounding grains. Although the number of \{110\} grains utilized for this investigation are small (43 grains), it shows that nearly ideal Goss oriented grains have the highest frequency of coincidence boundaries.

4. Orientation and rotation relationship of the grains directly surrounding a large grain

Figure 8 shows orientation and grain size distribution of all the grains larger than 20 \(\mu m\) in diameter present in the same area scanned by ECP in specimen A. As the maximum grain size in the grain size distribution shown in the Fig. 1(b) is 20 \(\mu m\), the large grains detected by this method is considered to be extraordinarily large grains in this specimen. Among the
grains detected, no grains of nearly Goss orientation (deviation angle of \langle100\rangle axis is less than 0.17 rad) were present. Comparing with the inverse pole figure shown in Fig. 2(b), it can be seen that the orientation of these grains are mainly the major textural component of

![Graph](image)

Fig. 6. Frequency of coincidence boundaries surrounding \{110\}<uvw> grains present at the 30 µm below the surface of the primary recrystallized specimen (specimen A, R; random boundaries).

![Graph](image)

Fig. 7. Frequency of coincidence boundaries (3–51) directly contacted to nearly \{110\}<001> oriented grains and \{110\} grains rotated larger than 0.28 rad around the ND axis present 30 µm below the surface of the primary recrystallized specimen (specimen A).

![Graph](image)

Fig. 8. Orientation and size distribution of matrix grains larger than 20 µm in diameter present 30 µm below the surface of the primary recrystallized specimen (specimen A).
Among large grains detected, 6 especially large grains were arbitrarily chosen and orientation of the surrounding grains of these large grains and coincidence orientation relationship of these grains with these large grains were investigated. The number of the surrounding grains were 116 indicating that these large grains are surrounded by 19.3 grains in average. Figure 9 shows orientations of these large grains and surrounding grains. It can be seen that the orientation of these surrounding grains are similar as those surrounding Goss grains shown in Fig. 5.

Figure 10 shows the frequency of coincidence boundaries surrounding large grains.

It can be seen from this figure that the frequency of $\Sigma 9$, $\Sigma 19b$, $\Sigma 25b$ and $\Sigma 29b$ are relatively high. While the frequency of $\Sigma 1$ boundary of the surrounding grains of $\{110\}$ orientation was low (2.5%), the frequency of $\Sigma 1$ boundaries surrounding large grains are high (6%) as shown in Fig. 1. Figure 11 shows an example of a microstructure of a large grain and its surrounding grains and their orientations. The size of this large grain is approximately 40 $\mu$m (5 times larger than the average grain size) and it can be a nucleus of secondary recrystallization if the size effect alone is effective for the secondary recrystallization.

Fig. 9. An example showing (a) orientations of 6 large grains arbitrarily chosen and (b) 116 grains directly contacted to these grains present at the 30 $\mu$m below the surface of the primary recrystallized specimen (specimen A).
IV. Discussion

Many investigations have been carried out so far on the distribution of Goss grains in the primary recrystallized specimens (2)(6)-(8)(11). Especially on the two-stage cold rolling method (6), detailed studies were done by Inokuti et al. (6) utilizing the Kossel method. On the intensity distribution of Goss grains in the primary recrystallized specimen processed by one stage cold rolling process, Flowers and Heckler (11) reported that the intensity of {110}<001> oriented grains decreases with increasing deviation angle of <001> axis from the rolling direction through the investigation by the harmonic method (17). This is the same tendency with the present investigation by ECP or the vector method. Takashima et al. (2) investigated by micro-etch pit method the existence of {110}<001> oriented grains in the surface layer of primary recrystallized specimen processed by the one-stage cold rolling process and found that {110}<001> oriented grains are often found as colonies.

Fig. 10. Frequency of coincidence boundaries surrounding large grains present 30 μm below the surface of the primary recrystallized specimen (specimen A).

Fig. 11. An example showing (a) a microstructure of a large grain and (b) its surrounding grains and their orientations present 30 μm below the surface of the primary recrystallized specimen (specimen A), ○ indicates a large grain and × indicates surrounding grains.
Sakai et al. \(^7\) reported that \{110\}<001> oriented grains are the largest grains present in the surface layer of the primary recrystallized specimen processed by the one-stage cold rolling method. The present investigation is different from the results obtained by Takashima et al. \(^2\) or Sakai et al. \(^7\). It is not clear why the different results were obtained in the present investigation. One of the reasons may be attributed to the difference of the observed area. The actual amount of the grains having the Goss orientation present in the surface of the primary recrystallized specimen is very small and that the area observed in this experiment is restricted. The grain size of the secondary recrystallization is between 20 and 30 \(\text{mm} \) in diameter. It means that the probability of the presence of the potential nucleus for the Goss secondary is null or only one in the area investigated in the present experiment if a secondary grain originates from the one nucleus having the largest grain size in that area in the primary recrystallized specimen. Therefore there is a probability that the potential Goss nucleus was not present in the area investigated by the present experiment.

The following two additional experiments were carried out to investigate that the potential nucleus for secondary recrystallization should always be the largest grains at the primary recrystallized stage. If the potential nuclei of the Goss secondaries are the largest grains, the sharpness of secondaries might be decreased when the specimen size is smaller than the grain size of the secondary recrystallized grain that might grow when the specimen size is large enough.

Figure 12\(^{18}\) shows the effect of the specimen width on the sharpness of Goss secondaries utilizing the similar specimen with the present experiment. The details of the secondary recrystallization annealing condition is shown elsewhere\(^{19}\). It shows that the sharpness of Goss secondaries does not depend on the specimen width. It was found out in some of the secondary grains that orientations are slightly different from place to place within one grain\(^{20}\). This suggests that a nucleus of a secondary recrystallized grain might not always originate from only one nucleus grain. These two additional experiments suggest that the potential nucleus of Goss secondaries is not necessarily the largest grains in the primary recrystallized matrix.

Recent finding by Makita et al. \(^{21}\) in FCC alloys also show that the grains having an orientation identical to the secondary recrystallized grains in the primary matrix are nearly the same in grain size as that of the surrounding grains. The reason that the large grains at the completion of the primary recrystallization cannot be a viable nucleus of secondary

![Fig. 12. Effect of the width of the primary recrystallized specimen on the sharpness of Goss secondaries.](image-url)
recrystallization might be considered as follows. The present authors indicated that the Σ1 boundary is not mobile in the grain growth process of 3%Si–Fe\(^{22}\). It has also been shown that the Σ1 boundary is not mobile and works as an inhibitor for grain growth in FCC alloys\(^{23}\).

The present investigation shows that large grains present at the primary recrystallized stage has a higher frequency of directly contacting with the Σ1 boundary compared with Goss oriented grains. In order to know the probability of these grains to contact with the Σ1 boundary in the course of grain growth of these grains, the frequency of the coincidence boundaries corresponding to these 6 large grains in the primary recrystallized matrix were investigated. The orientations of the 507 grains in the primary recrystallized specimen processed by a similar condition to that in the present experiment were utilized as matrix grains for this investigation. (111)[112], (001)[010] and an ideal Goss orientation are also investigated for comparison. (111)[112], (001)[010] are one of the major texture components of the primary texture and an ideal Goss orientation is the major secondary textural component. It can be seen from Fig. 13 that these 6 grains have a higher frequency of the Σ1 boundary compared with an ideal Goss orientation, and also that (111)[112], (001)[010] have a higher frequency of the Σ1 boundary compared with an ideal Goss orientation.

These results indirectly suggest that grains having these orientations have a higher probability of grain growth inhibition during grain growth in the course of the secondary recrystallization annealing than the Goss orientation. Recently several researchers\(^{22}\)(24)-(26) have reported that the coincidence boundaries play an important role in the secondary recrystallization of 3% Si–Fe as is the case with FCC alloys\(^{21}\)(22)(27). It has been shown that an ideal Goss orientation has the highest probability of contacting with the coincidence oriented grains in the course of grain growth\(^{24}\), and also that coincidence boundaries are more mobile than random boundaries in the grain growth process of the same material\(^{22}\). It has also been shown that the secondary recrystal-

![Fig. 13. Frequency of the Σ1 boundary and the coincidence boundaries in the primary recrystallized matrix corresponding to 6 extraordinary large grains and an ideal Goss orientation and the two major orientations ((111)[112], (001)[010]).](image)
tensity level of the inhibitor at which the coincidence boundary could migrate in preference to other boundaries, resulting in the formation of viable nucleus of secondary recrystallization. Based on this mechanism, the size of the nucleus is not the prime necessity for the evolution of the secondary recrystallization texture.

V. Conclusion

(1) \{110\}<001> oriented grains present below 30 µm from the surface of the primary recrystallized specimen processed by the one-stage cold rolling method were not among the largest grains nor were present as an aggregate.

(2) The orientation of the extraordinarily large grains present below 30 µm from the surface of the primary recrystallized specimen processed by the one-stage cold rolling method was on the whole similar to that of the major textural component of the primary recrystallized specimen.

(3) Among the \{110\} oriented grains, the Goss oriented grain had the highest frequency of coincidence oriented grains in the grains directly surrounding to these \{110\} oriented grains and had a very low frequency of the \{111\} boundary in relation to its orientation in the surface of the primary recrystallized matrix, while the large grains above mentioned had rather higher frequency of the \{111\} boundary in relation to its orientation.

(4) The mechanism of the sharp Goss texture formation by secondary recrystallization can be explained successfully, even if the Goss nucleus is not the largest at the completion of the primary recrystallization, by considering the special distribution of coincidence boundaries and their specific gain boundary migration characteristics associated with the intensity of the inhibitor.

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