Influence of grain size before cold rolling on development of crystallographically equivalent main texture component in recrystallization texture of 3% Si-Fe

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Abstract. 3%Si-Fe sheet produced by high cold rolling reduction over 80% has strong (111)<112> recrystallization texture. This orientation has two crystallographically equivalent orientations such as (111)[1 -2 1] and (111)[-1 -1 2]. However, there are a few previous works in which these orientations are investigated separately. In the present study, the recrystallization behavior of the crystallographically equivalent orientations of (111)<112> was investigated using two samples with different grain sizes before cold rolling. Both recrystallized samples had strong (111)<112> component texture. The sample with a small grain size before cold rolling had almost the same fraction of each equivalent orientations, while the sample with a large grain size had substantially different fractions of each equivalent orientations. Spatial distribution of two crystallographically equivalent orientations in the sample with a small grain size before cold rolling was almost homogeneous, however that in the sample with a large grain size before cold rolling was inhomogeneous. It is considered that these differences are explained by the assumption that a certain large grain before cold rolling will generate only single crystallographically equivalent orientation after cold rolling and annealing.

1. Introduction
Texture control is very important for improving mechanical or magnetic properties of several kinds of steel products. Therefore, rolling and annealing conditions are usually controlled precisely. For example, (111)//ND (normal direction of a sheet plane) component in the texture of final product provides good deep drawability [1, 2]. Therefore, this component is suitable for steel sheets for automobile. In the case of electrical steels, since (100) direction can be magnetized easily, this direction is aligned with an excitation direction on the steels. Texture control is applied not only to final process but also to intermediate processes. In grain-oriented electrical steel sheets, (111)<112> component is needed on a primary recrystallization texture in order to obtain a strong (110)<001> (Goss) texture after secondary recrystallization. There are many studies which attempt to explain a reason why (111)<112> component is important for the strong Goss texture development [3-13]. For example, Hayakawa et al. proposed a mechanism that grain boundaries having misorientation between 20° and 45° in primary recrystallized sheet play an important role in a selection of secondary recrystallization textures [3-6]. Since (111)<112> orientation has 35° of misorientation toward Goss orientation, the (111)<112> component in the primary recrystallization texture is highly effective for Goss texture development in a final product. Since (111)//ND component, including (111)<112> orientation, is important as described above, there are many studies which are trying to clarify a formation mechanism of this component. Abe et al.
mentioned that recrystallized grains with \{111\} were preferentially formed in initial grain boundary regions [14]. Senuma et al. [15] and Inagaki [16] obtained almost same results independently that \{111\} recrystallized grains were formed from grain boundary regions of deformed grains. Furthermore, other results [17,18] demonstrating that small initial grain size gave rise to a strong \langle111\rangle//ND recrystallization texture implied that the nucleation would occur on initial grain boundaries. However, Vanderschueren et al. showed that \langle111\rangle//ND nuclei did not generate only at grain boundaries, they also formed in the interior of deformed \gamma\text{-fiber} grains [19]. Also, Barnett confirmed that nuclei having near \{554\}<225> orientation, which is considered to be one of \langle111\rangle//ND component, generated by in-grain shear banding [20]. In view of rolling reduction on recrystallization texture, a high cold rolling reduction seems to result in a \{111\}<112> texture development during recrystallization and grain growth [21, 22], whereas \{111\}<110> orientation become major in the case of a relatively low rolling reduction [14, 23].

\{111\}<112> orientation has two crystallographically equivalent orientations such as \{111\}[1 -2 1] and \{111\)[-1 -1 2] in rolling process. However, there are a few previous works in which these orientations are investigated separately. In the present study, the generation behavior of the crystallographically equivalent orientations of \{111\}<112> was investigated using two samples with different grain sizes before cold rolling.

2. Experiment

3.1 mass%Si-0.1mass%Mn-Fe alloy ingot was hot-rolled to 2.3 mm thickness after heating to 1473 K (1200 °C). Then the two hot-rolled samples were heated to 1173 K (900 °C) and 1373K (1100 °C) respectively for 30 seconds under nitrogen atmosphere in order to obtain different recrystallized grain size. Both heated samples were cold-rolled to 0.23 mm thickness with the cold rolling reductions rate of 90%. Then an annealing was performed on the cold rolled samples at 943K (670 °C) without soaking time and at 1073K (800 °C) for 10 seconds under nitrogen atmosphere. The samples subjected to the annealing at 943 K (670 °C) were hardly recrystallized, whereas the samples subjected to the annealing at 1073 K (800 °C) were fully recrystallized. This procedure is shown in Table 1.

An optical microscope was used to obtain the microstructure of the samples before cold rolling in order to evaluate their grain size. The orientation distribution function (ODF) of the fully recrystallized samples was calculated from 110, 200 and 211 pole figures measured by X-ray diffraction (XRD) method using Rigaku RINT-2000. XRD measurement was performed on the center layer of the samples. The ODF calculation was performed using the discrete method [24] with orthorhombic lattice system. In this paper, ODF representation of $\phi_2 = 45^\circ$ cross section in Bunge notation [25] is used.

| Ingot with 3.1 mass%Si-0.1mass%Mn-Fe |
|-------------------------------------|
| Heating at 1473K (1200°C) for 1hour |
| then hot rolling to 2.3mm           |
| Soaking for 30 seconds              |
| at 1173K (900°C)                   |
| <sample A>                         |
| Cold rolling to 0.23mm              |
| <sample Ac>                        |
| Annealing                           |
| at 943K (670°C)                    |
| without soaking time               |
| <sample A-1>                       |
| at 1073K (800°C) for 10 seconds    |
| <sample A-2>                       |
| at 1373K (1100°C)                  |
| <sample B>                         |
| Cold rolling to 0.23mm              |
| <sample Bc>                        |
| Annealing                           |
| at 943K (670°C)                    |
| without soaking time               |
| <sample B-1>                       |
| at 1073K (800°C) for 10 seconds    |
| <sample B-2>                       |

Table 1. Experimental procedures of this study.
Microstructures and grain orientations of the annealed samples were investigated by electron backscatter diffraction (EBSD) method using scanning electron microscope (SEM, JEOL JSM-7001F) and EBSD data analysis software (TSL OIM Analysis 7). EBSD measurement was carried out on the surface (perpendicular to normal direction) of the fully recrystallized samples and the cross section (perpendicular to transverse direction) of the samples subjected to the annealing at 943 K (670 °C).

3. Results and discussion

The microstructures of the heated samples before cold rolling are shown in figure 1. Both samples are fully recrystallized and have relatively equiaxed grains. Sample A subjected to heat treatment at 1173 K (900 °C) has small grain size, sample B subjected to heat treatment at 1373 K (1100 °C) has huge grain size.

Figure 2 shows textures of cold-rolled (sample Ac, Bc) samples. The cold-rolling textures have strong α-fiber and {111}<110> components, which are well known as major components in a deformation texture in the rolling process [26, 27], and are very similar each other. It is generally accepted that the initial grain size has little influence on cold-rolling texture [14, 26]. Same tendency was also observed in this study. In the recrystallization texture in sample A-2 and B-2, both textures had main component of near {111}<112> orientation, which is frequently observed in recrystallization textures of low carbon steels and silicon steels.

Figure 3. Orientation Image Map of Variant A (colored in red) and B (colored in green) with 15° tolerance. a) sample A-2, b) sample B-2.
\{111\}<112> orientation has two crystallographically equivalent orientations (111)[1 -2 1] and (111)[-1 -1 2] in rolling process. In order to investigate spatial distributions of each crystallographically equivalent orientations separately, crystal orientation analysis was performed on the fully recrystallized samples by EBSD. The result is shown in Figure 3. In this analysis, two crystallographically equivalent orientations were named as Variant A and B. Variant A and B are defined as the orientations represented as Euler angles \((\phi_1, \Phi, \phi_2) = (30, 55, 45)\) and \((90, 55, 45)\) in ODF space, respectively. Figure 3(a) shows Image Quality (IQ) map of sample A-2. Recrystallized grains having Variant A and B were colored in red and green respectively with 15° tolerance and are placed uniformly. In the case of sample B-2, however, spatial distribution of Variant A and B is inhomogeneous as shown in Figure 3(b).

In order to evaluate spatial distribution precisely, nearest neighbor analysis was applied [28]. In this analysis, the degree of spatial dispersion in the distribution is evaluated on minimum distances between two points with a numerical value called as the nearest neighbor index (NNI), which is calculated using a following equation,

\[
\text{NNI} = \frac{Ad}{Ed}
\]

where \(Ad\) is the average of nearest neighbor distance defined as,

\[
Ad = \frac{1}{n} \sum_{i=1}^{n} di
\]

where \(n\) is the number of points and \(di\) means the nearest neighbor distance of \(i\)-th point. \(Ed\) is the expected value of the average nearest distance in random distribution. \(Ed\) is defined as,

\[
Ed = \frac{1}{2\sqrt{n/S}}
\]

where \(S\) is the area that the points exist.

NNIs of the grains with Variant A on both sample A-2 and B-2 were calculated with above equations using the measured EBSD data including grain positions and measurement area. In this analysis, the number of intended grains of Variant A is approximately 280 in both samples. The calculated NNIs were 0.943 and 0.868, respectively. According to the nearest neighbor analysis, a distribution is

\[\text{Figure 4. IPF maps and IQ maps of sample A-1 (a) and c)) and B-1 (b) and d). (TD cross section)}\]
classified three types such as random, clustered and dispersed patterns [29]. When NNI is near 1, the
distribution of points is almost random pattern. When NNI is lower than 1, the distribution is clustered.
Consequently, it is statistically determined that the grain distribution of Variant A on sample A-2 had
almost random pattern and that on sample B-2 was clustered.
In order to explain a reason why the spatial distributions of the recrystallized grains were different with
the grain size before cold rolling, microstructures and their crystal orientation distributions in deformed
state, which was prior to recrystallization, were investigated using sample A-1 and B-1 by EBSD method.
Figure 4 shows inverse pole figure (IPF) maps and IQ maps obtained with the EBSD measurement on
the cross section of the samples. Each IPF is parallel to the normal direction (ND). There are many
elongated deformed band structures toward the rolling direction (RD) in sample A-1 as shown in Figure
4 (a) and (c). On the other hand, sample B-1 has a few elongated deformed band structures as shown in
Figure 4 (b) and (d). Thickness of them is very high, which is considered to reflect the grain size before
cold rolling. IPF maps (in Figure 4 (a) and (b)) display orientations of these deformed band structures
for both samples. Orientation of these band structures mainly consist of <111>/<ND (colored in blue)
and <100>/<ND (colored in red), which are well known as popular components in a heavily cold-rolled
specimen [26, 27]. Many previous works have revealed that recrystallized grains that have {111}<112>
orientation mainly generate in deformed grains with <111>/<ND orientation [30-33]. Hence, deformed
grain with <111>/<ND orientation in the sample which had large grain size before cold rolling in Figure
4 (b) were examined in detail. Figure 5 shows the particular region of Figure 4(b) with <111>/<ND.
There are many small band-like areas with low IQ value (shown with arrows in figure 5 (a)), which
implies strain accumulation. Many of these areas have Variant A as shown in figure 5 (b). Moreover,
the deformed band structure has Variant B. In order to investigate the orientation relationship between
the small band-like area and the deformed band structure, these orientations in selected areas shown
with a rectangle in Figure 5 are represented by pole figures in Figure 6. These orientations have a
common <211> axis as shown near the center of the 211 pole figure. This fact implies that crystal
orientation rotation around the <211> common axis would occur. Similar orientation rotation
relationship has been reported by Muraki et. al [31].
Since the many small band-like areas had the same orientation of Variant A, this orientation rotation
probably occurred in the entire region of the deformed band structure shown in figure 5. Assuming the
small band-like areas are main candidates of recrystallization nuclei, recrystallized grains with Variant
A will dominate after the annealing. Hence, the special distribution differences of crystallographically
equivalent orientations of $<111>\{112\}$ can be explained by the assumption that a certain large grain before cold rolling will generate only single crystallographically equivalent orientation after cold rolling and annealing.

**Conclusion**

The recrystallization behavior of the crystallographically equivalent orientations of $\{111\}<112>$ was investigated using two samples with different grain sizes before cold rolling. Both recrystallized samples had strong $\{111\}<112>$ component texture. But spatial distributions of two crystallographically equivalent orientations of $\{111\}<112>$ in the recrystallized samples were different. It is considered that these differences are explained by the assumption that a certain large grain before cold rolling will generate only single crystallographically equivalent orientation after cold rolling and annealing.

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