Secondary Recrystallization of Grains with Cube Orientation for Pure Iron Tape

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Abstract. The formation mechanism of secondary recrystallized grains with cube orientation has been investigated for a pure iron tape prepared by a three-stage cold-rolling method. The crystal orientation before the final cold-rolling affected the appearance of secondary recrystallized grains with cube orientation. The {0 1 1} {<1 0 0>} orientation before the final cold-rolling led to the formation of secondary recrystallized grains with cube orientation on the tape surface.

1. Introduction

Secondary recrystallization has been used for controlling the crystal orientation of silicon steel sheets. The secondary recrystallization of grains with {0 1 1} {<1 0 0>}, Goss orientation, is well known for silicon steel sheets with 3% silicon. The secondary recrystallized grains with Goss orientation align {<1 0 0>} axis of grains to parallel to the rolling direction (RD) for the silicon steel sheet, which is fabricated by rolling and annealing. Pure irons and silicon steels, which are soft magnetic materials, have anisotropy of magnetization. The {<1 0 0>} axis is the direction most easily magnetized as compared with the {<1 1 0>} and {<1 1 1>} axes [1]. The easily magnetization direction for the sheet with Goss orientation is one direction which is parallel to the RD on the sheet surface. On the other hand, the sheet with {0 0 1} {<1 0 0>}, cube orientation, has two easily magnetization directions on the sheet surface, which are parallel to the RD and the transverse direction (TD). The cube oriented silicon steel is attractive as a core material with a low iron loss in transformers. It is difficult to develop the cube orientation in silicon steel with a bcc structure. The fabrication of silicon steel sheet with cube orientation has been attempted by various methods based on rolling and annealing processes. The cube oriented silicon steel has been fabricated using a cross cold-rolling technique, which the silicon steel was rolled alternately along two perpendicular directions for the cross rolling process [2]. The cube oriented silicon steel has been prepared by the cross rolling of 3% Si steel containing AlN [3]. The 3% Si steel sheet with cube orientation has been fabricated by the manganese removal and decarbonization of steel with a chemical composition of Fe-3Si-1.0Mn-0.05C [4].

From the viewpoint of manufacturing industrial products of the silicon steels with cube orientation, it is desirable to develop the cube orientation on the silicon steel sheet by the simplest method possible. It is important to clarify the mechanism of secondary recrystallization of the grains with cube orientation for metals with bcc structure. In this paper, the secondary recrystallization of grains with cube...
orientation has been reported for a pure iron tape prepared by a three-stage cold-rolling method. The pure iron with bcc structure was selected for investigating the essential mechanism of the secondary recrystallization in pure iron with few impurities. The initial orientation before final cold-rolling have been pointed to be important for development of secondary recrystallization of grain with cube orientation for the silicon steel [4]. The relationship between initial orientation before final cold-rolling and the secondary recrystallization of grains with cube orientation was investigated for the pure iron.

2. Experimental procedures
For the fabrication of iron tape with a cube orientation, an iron ingot was prepared by arc melting. The chemical composition of the iron metal used in this study is shown in Table 1. Rectangular rod specimen with dimensions of approximately W8×T12mm² was cut into from the iron ingot. For the third-stage cold-rolling process, the reduction rates in thickness were 15% for first rolling, 75% for second rolling and 92% for third rolling, which was final rolling. The annealings were performed at 1323K×10.8ks for the first intermediate-annealing, at 1473K×10.8ks for the second intermediate-annealing, and at 1163K×3.6ks for the final annealing. All of the annealings were performed in a vacuum. The thickness of tape fabricated is ca. 0.2mm. The surface of the iron tape was electropolished with a solution of perchloric acid and ethanol (= 1:9) for EBSD observation. The orientation of the iron tape was analyzed using an SEM-EBSD system (JEOL JSM-6360 equipped with a TSL MSC-2200 EBSD camera). The orientation characteristics of the iron tape were analyzed using TSL orientation imaging microscopy (OIM) analysis software (version 6.1).

| C    | P    | S    | Si   | Mn   | Cu   | O    | N    | H    | Fe   |
|------|------|------|------|------|------|------|------|------|------|
| Pure iron |<0.0020 |<0.0005 |<0.0005 |<0.0005 |<0.0005 |<0.0005 |<0.0005 |<0.0005 |<0.0005 |Bal.  |

3. Results
The {0 0 1} pole figure and IPF map for tape surface of the iron tape (sample-1) fabricated by the third-stage cold-rolling process are shown in Figure 1. The white and black lines in the IPF map denote low-angle grain boundaries with angles < 15°, and high-angle grain boundaries with angles ≥ 15°, respectively. The pole figure shows that the orientation of the tape surface is the cube orientation. The coarse grains with a grain size over ca. 1mm formed on the tape surface shown in Figure 1 (b). These results show that the secondary recrystallized grains with cube orientation forms on the tape surface of iron tape. The grain with cube orientation for secondary recrystallization grows in a vacuum atmosphere according to the following the growth rate (G) of grain for pure iron:

\[ G = M \left( \frac{γ_0}{r} + \frac{2Δγ_s}{t} \right) \]

where \(M\), \(γ_0\), \(r\), \(Δγ_s\) and \(t\) are the mobility of the grain boundary, the grain boundary energy, the average primary grain radius, the average difference in the surface energy (\(γ_0\)) between the \{0 0 1\} plane and the other \{h k l\} planes, and the tape thickness [5]. For the secondary crystallization of grains with cube orientation in a vacuum, the grains with \{0 0 1\} plane grow by the driving force of grain growth related to the grain boundary energy and the surface free energy of \{0 0 1\} plane, which is based on the \(Δγ_s\) during the annealing in the vacuum atmosphere. Vacuum annealing leads to growth of grains with \{0 0 1\} orientation, because the surface energy of the \{0 0 1\} plane become low in vacuum atmosphere.
It has been pointed out that the cube orientation for secondary recrystallization is related to orientation of tape surface before final cold-rolling. The crystal rotation for specimen with the orientation of {0 1 2}<1 0 0> and {0 1 4}<1 0 0> by the final rolling and annealing results in the formation of cube orientation on the sheet surface for the Fe-3%Si sheets [4, 6]. The cube oriented pure iron tape was fabricated by the final rolling and annealing of the specimen with an orientation of {2 1 1 26}<3 1 8 1> shown in Figure 2. The (2 1 1 26) plane is a near-(0 1 2) plane with a misorientation of ca. 5° from the (0 1 2) plane. The crystal rotation occurred by final cold-rolling and annealing for the pure iron tape, which is the crystal rotation from {0 1 2} <1 0 0> to {0 0 1} <1 0 0>, is similar to those for the Fe-3%Si sheets.

Figure 1. (a) {0 0 1} pole figure and (b) IPF map for tape surface of iron tape (sample-1) fabricated by the three-stage cold-rolling method.

Figure 2. {0 0 1} pole figure for pure iron tape (sample-1) before final cold-rolling.
An iron tape (sample-2) was fabricated by the cold-rolling and annealing of the specimen with an orientation of $\gamma$-fibers shown in Figure 3, which are ND/\langle 4 \ 5 \ 6 \rangle and ND/\langle 2 \ 1 \ 1 \rangle. The cold-rolling with a reduction rate in thickness of 89% and annealing at 1073K/$\times$3.6ks in a vacuum were performed for the specimen. The \{1 1 1\} pole figure and IPF map for tape surface of the iron tape (sample-2) fabricated are shown in Figure 4. The recrystallized grains with an orientation of $\gamma$-fiber (ND/\langle 1 \ 1 \ 1 \rangle) grew on the tape surface. The grains with a grain size of less than 300$\mu$m formed on the tape surface. For the specimen with the orientation of $\gamma$-fibers before the cold-rolling, the origin of grains with cube orientation for secondary recrystallization is unlikely to be formed in the rolled structure of iron tape. These results show that the crystal orientation before final cold-rolling affects the formation of secondary recrystallized grains with cube orientation. In the secondary recrystallization for pure iron, the \{0 1 2\}/\langle 1 \ 0 \ 0 \rangle orientation before cold-rolling resulted in the formation of origin of grains with cube orientation in the rolled structure of the tape. The origin of grains with cube orientation grows to secondary recrystallized grains mainly by a driving force of the surface free energy of \{0 0 1\} plane under the vacuum annealing.

![Figure 3. \{0 0 1\} pole figure for specimen (sample-2) of pure iron before cold-rolling.](image)

![Figure 4. (a) \{1 1 1\} pole figure and (b) IPF map for tape surface of iron tape (sample-2) fabricated by the cold-rolling and annealing of specimen with the orientation of $\gamma$-fibers of ND/\langle 4 \ 5 \ 6 \rangle and ND/\langle 2 \ 1 \ 1 \rangle.](image)
4. Conclusion
The formation mechanism of secondary recrystallized grains with cube orientation was investigated for a pure iron tape prepared by the three-stage cold-rolling method. The crystal orientation before the final cold-rolling affected the appearance of secondary recrystallized grains with cube orientation. The \{0 \bar{1} 2\} <1 0 0> orientation before the final cold-rolling led to the formation of secondary recrystallized grains with cube orientation on the tape surface.

Acknowledgments
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