Investigation on the primary and secondary recrystallization textures of rare earth micro-alloyed grain-oriented silicon steel

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Abstract. The primary and secondary recrystallization textures of rare earth micro-alloyed grain-oriented electrical steel was examined by using electron backscattered diffraction (EBSD) techniques. The results show that the primary recrystallization texture comprises \{111\}\{112\}, \{111\}\{110\}, \{411\}\{148\}, and \{012\}\{001\} components. The beneficial texture components of primary recrystallization increase with increasing rare earth addition after decarburizing annealing. During secondary recrystallization, the proportion of Σ9 boundaries are reduced. The sample containing 5ppm rare earth starts secondary recrystallization at 900°C, and the sample containing of 17ppm rare earth starts secondary recrystallization at 950°C. The addition of rare earth can delay the process of secondary recrystallization.

Key words: rare earth, grain-oriented silicon steel, secondary recrystallization, texture

1. Introduction

Grain-oriented silicon steel uses a secondary recrystallization phenomenon to obtain a single \{110\}<001> texture. In order to develop a perfect secondary recrystallized structure, fine dispersed precipitates are often used as inhibitors. The inhibitor inhibits the normal growth of the primary recrystallized grains by pinning, and causes grains having the \{110\}<001> orientation to grow abnormally during secondary recrystallization. Therefore, texture is one of the key contents of research on grain-oriented silicon steel [1-3]. Adding trace rare earth elements to steel can improve the strength, toughness and corrosion resistance of steel [4-7]. Therefore, the development of high quality rare earth microalloyed steel is one of the methods to improve the performance of steel. The purpose of this paper is to investigate the primary and secondary recrystallization textures of rare earth micro-alloyed grain-oriented silicon steel.

2. Experimental Methods

The composition of the experimental steel is shown in Table 1. The manufacturing process of the grain-oriented silicon steel is as follows: smelting→ forging→ slab heating at 1250°C→ hot rolling→ pickling→ first cold rolling→ decarburization annealing→secondary cold rolling→ magnesium oxide coating → high temperature annealing. The decarburization annealing is performed in a wet H₂ and N₂ mixed atmosphere at a temperature of 820 °C for 4 minutes. The high temperature annealing process is conducted at 1200 °C at a heating rate of 15 °C/h in a 25% H₂ + 75% N₂ atmosphere, and then purified...
at 1200℃×6h in 100% high purity H₂, and finally cooled. The microstructure of grain-oriented silicon steel was observed by a Zeiss optical microscope. The primary and secondary recrystallization textures of the rare earth micro-alloyed grain-oriented electrical steel was examined by using electron backscattered diffraction (EBSD).

| specimen | C  | Si  | Mn  | P  | S  | Al  | Cu  | Nb  | Ce  |
|----------|----|-----|-----|----|----|-----|-----|-----|-----|
| No. 1    | 0.037 | 2.99 | 0.25 | 0.008 | 0.020 | 0.016 | 0.524 | 0.008 | 0.0005 |
| No. 2    | 0.038 | 3.04 | 0.25 | 0.007 | 0.016 | 0.021 | 0.524 | 0.008 | 0.0017 |

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|----------|----|-----|-----|----|----|-----|-----|-----|-----|
| No. 1    | 0.037 | 2.99 | 0.25 | 0.008 | 0.020 | 0.016 | 0.524 | 0.008 | 0.0005 |
| No. 2    | 0.038 | 3.04 | 0.25 | 0.007 | 0.016 | 0.021 | 0.524 | 0.008 | 0.0017 |

Figure 1. EBSD grain unique color maps of the specimens obtained after decarburization annealing at 820℃
(a) sample No. 1; (b) sample No. 2

3. Results and Discussion

Fig. 1 shows the EBSD grain unique color maps of the specimens obtained after decarburization annealing at 820℃. The typical primary recrystallization texture components are {110}<112>, {111}<110>, {411}<148>, {012}<001>, {001}<100>. The content of Goss ({110}<001>) in primary recrystallization texture is very small, mainly distributed around {111}<112> and {111}<110>. During the cold rolling process, the Goss residual crystal exists in the form of cell structure in transition zones in {111}<112> grains. During decarburization annealing, the Goss texture preferentially nucleates on the {111}<112> grain boundary. The {111}<112> texture not only provides a nucleation site for the Goss texture, but also provides a matrix for the abnormal growth of the Goss texture. The {111}<112> texture in the γ fibre and the {411}<148> texture in the α fibre have close to the Σ9 CLS boundaries with respect to the Goss texture, which is beneficial to the abnormal growth of Goss grains during high temperature annealing. It is also found that {012}<001> texture is a texture that is conducive to the abnormal growth of Goss grains during high temperature annealing.

The area fractions of primary orientations in the primary recrystallized structure are listed in Table 2. The fractions of the Goss texture of the two samples are not very high, and the fraction of sample No. 2 is higher than that of sample No. 1. The sum of the four favorable textures {111}<112>, {111}<110>, {411}<148>, {012}<001> of sample No. 2 is higher than that of sample No. 1. Moreover, the fraction of {110}<112> texture in sample No. 2 was lower than that of sample No. 1. {110}<112> texture severely affects the formation of Goss texture during the high temperature annealing of oriented silicon steel, so it is of great significance to inhibit the texture of {110}<112> during the
production of oriented silicon steel. It can be seen from the above analysis that the addition of rare earths is more conducive to the abnormal growth of the Goss grains during the high temperature annealing of the grain-oriented silicon steel.

Table 2 Area fractions of primary orientations in the primary recrystallized structure (%)

| specimen | {001} | {110} | {112} | {111} | {110} | {001} | {100} | {112} | {111} | {012} | {001} | {411} | {148} |
|----------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| No. 1    | 1.3   | 0.854 | 13.5  | 4.16  | 2.32  | 7.94  | 20.2  | 1.39  | 8.73  | 10.9  |       |       |       |
| No. 2    | 1.13  | 0.793 | 11    | 5.6   | 4.26  | 8.76  | 17.3  | 3.36  | 12.1  | 14.3  |       |       |       |

Figure 2. Microstructure of sample No. 1 at different temperature during annealing
(a) 650 °C; (b) 850 °C; (c) 900 °C; (d) 950 °C;

Figure 3. Microstructure of sample No. 2 at different temperature during annealing
(a) 650 °C; (b) 850 °C; (c) 900 °C; (d) 950 °C
Fig. 2 shows the microstructure of sample No. 1 during annealing at 650 °C, 850 °C, 900 °C, 950 °C, respectively. It can be seen that sample No. 1 did not undergo secondary recrystallization at 650 °C and 850 °C, and the crystal grains were fine and uniform. When the temperature is raised to 900 °C, a large grain appears on the edge of the sample, indicating that secondary recrystallization has occurred. As the temperature increases, the abnormally grown Goss grains continuously engulf the small grains around them, and the abnormally grown grains are uneven and the grain boundaries are extremely irregular.

Fig. 3 shows the microstructure of sample No. 2 during annealing at 650 °C, 850 °C, 900 °C, 950 °C, respectively. It can be seen that sample No. 2 did not undergo secondary recrystallization at 650 °C, 850 °C and 900 °C. A large grain appeared on the edge of the sample at 950 °C, which proved that it began to undergo secondary recrystallization. Comparing the secondary recrystallization starting temperature of No. 1 and No. 2 samples, it can be seen that the addition of rare earth to the oriented silicon steel will delay the occurrence of secondary recrystallization.

The EBSD grain unique color maps of sample No. 1 at 900 °C and sample No. 2 at 950 °C during annealing is presented in Fig. 4. As can be seen, the Goss grains are elongated along the RD, and growth of the Goss grain along the ND is being blocked by many equiaxed grains. The sizes of most equiaxed grain are 20-100 μm. The equiaxed grains texture components are {110}<112>, {111}<110>, {011}<120>, {411}<148>, {012}<011>. The secondary recrystallization is induced by special grain boundaries between {111}<112> and Goss grains. Goss grains prefer to swallow {111}<112> primary grains but have difficulty consuming {411}<148> primary grains during high temperature annealing, despite their similar Σ9 relationships [8].

![Figure 4. EBSD grain unique color maps of sample No. 1 at 900 °C (a) and sample No. 2 at 950 °C (b) during annealing](image)

| Temperature/°C | Sample No. 1 | Sample No. 2 |
|---------------|--------------|--------------|
|               | Σ 3 | Σ 5 | Σ 7 | Σ 9 | Σ 3 | Σ 5 | Σ 7 | Σ 9 |
| 850           | 4.25 | 0.49 | 0.33 | 1.14 | 3.75 | 0.45 | 0.72 | 0.52 |
| 900           | 3.55 | 0.48 | 0.27 | 0.39 | 5.07 | 0.36 | 0.44 | 0.62 |
| 950           | 3.02 | 0.59 | 0.31 | 0.79 | 4.12 | 0.28 | 0.40 | 0.40 |

Table 3 shows the proportions of CSL boundaries at different temperatures during annealing. No. 1 sample undergoes secondary recrystallization at 900 °C, and the proportion of the Σ9 grain boundary
which is favorable for the abnormal growth of Goss grains, is significantly reduced. Sample No. 2 undergoes secondary recrystallization at 950 ℃, and the proportion of Σ9 grain boundary was also significantly reduced. It is indicated that Σ9 grain boundaries contribute to the growth of Goss grains, and they are preferentially consumed. The coincidence site lattice theory holds that the energy of CSL grain boundaries is lower than those of other large angle boundaries. During the temperature rise of high temperature annealing, the pinning force of the CSL grain boundary by the inhibitor is smaller than those of other large angle grain boundaries. When the inhibitor begins to coarsen and decompose, these grain boundaries will be out of pinning earlier, which will help the Goss grains to grow abnormally, especially the Σ9 grain boundary plays a very important role in the process of Goss grain growth.

4. Conclusion
The primary recrystallization texture of rare earth micro-alloyed grain-oriented silicon steel comprises {111}<112>, {111}<110>, {411}<148>, and {012}<001> components. Adding rare earth can improve favorable texture components of primary recrystallization texture. The sample containing of 5ppm rare earth starts secondary recrystallization at 900℃, and the sample containing of 17ppm rare earth starts secondary recrystallization at 950℃. The addition of rare earth can delay the process of secondary recrystallization.

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