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Microstructure and texture evolution of ultra-thin grain-oriented silicon steel sheet fabricated using strip casting and three-stage cold rolling method

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ABSTRACT

A 0.1 mm-thick grain-oriented silicon steel sheet was successfully produced using strip casting and three-stage cold rolling method. The microstructure, texture and inhibitor evolution during the processing was briefly analyzed. It was found that Goss texture was absent in the hot rolled sheet because of the lack of shear deformation. After normalizing, a large number of dispersed MnS precipitates with the size range of 15–90 nm were produced. During first cold rolling, dense shear bands were generated in the deformed ferrite grains, resulting in the intense Goss texture after first intermediate annealing. The microstructure was further refined and homogenized during the subsequent cold rolling and annealing processes. After primary recrystallization annealing, a homogeneous microstructure consisting of fine and equiaxed grains was produced while the associated texture was characterized by a strong γ-fiber texture. Finally, a complete secondary recrystallization microstructure consisting of entirely large Goss grains was produced. The magnetic induction B_8 and iron loss $P_{10/400}$ was 1.79 T and 6.9 W/kg, respectively.

1. Introduction

Grain-oriented silicon steel is mainly used in transformers as a result of its excellent magnetic properties originating from sharp {110} < 001 > preferred orientation (Goss texture) [1,2]. Nowadays, with increasing demand of high-frequency electrical appliances, ultra-thin (≤0.1 mm) grain-oriented silicon steel has drawn great attention because of its outstanding high-frequency performances [3]. However, although the processing has been well improved [4,5] since Goss first proposed the manufacturing route of grain-oriented silicon steel in 1934 [6], it is still difficult to produce the ultra-thin products by conventional processing. That is because the secondary recrystallization of ultra-thin sheets may become unstable due to the instability of inhibitors, giving rise to the formation of small grains [7,8]. Thus, up to now, people have to fabricate the ultra-thin sheets mainly by a complicated tertiary recrystallization process [9,10]. In this process, 0.18–0.35 mm-thick conventional or high-permeability grain-oriented silicon steel products are used as raw materials. After removing the glass film and dielectric film, the sheets are subjected to cold rolling and annealing in which sharp Goss texture forms due to lower surface energy of {110} planes [9]. People have made great efforts in stabilizing the secondary recrystallization of ultra-thin sheets to produce them by a simpler way.

Strip casting is based on the concept originally proposed by Bessemer [11], in which thin strips can be directly produced from the molten metal. The recent progress in strip casting makes it possible to produce grain-oriented silicon steels by a simpler way. Recently, Liu et al. [12] and Song et al. [13,14] investigated the microstructure, texture and inhibitor evolution along the processing and successfully fabricated 0.23–0.27 mm-thick strip-cast grain-oriented silicon steel sheets. It was found that strip casting exhibited great advantages in producing effective inhibitors and desired primary recrystallized structures, leading to the complete secondary recrystallization microstructures [12–14]. Thus, it is reasonable to deduce that more effective inhibitors together with more suitable primary recrystallization structures may be produced by sufficiently controlling the microstructure, texture and inhibitor evolution. As a result, it may be possible to stabilize the secondary recrystallization and thus produce the ultra-thin grain-oriented silicon steel sheets using strip casting process. However, the corresponding investigations have not yet been reported.

In this work, an ultra-thin 0.1 mm-thick grain-oriented silicon steel sheet was successfully fabricated by a novel process. This process included the strip casting, hot rolling, normalizing, three-stage cold rolling with intermediate annealing, primary recrystallization annealing and secondary recrystallization annealing. The microstructure, texture and inhibitor evolution along the processing was briefly investigated.

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2. Experimental

The chemical composition (wt%) of the tested steel was 3.2 Si, 0.055 C, 0.085 Mn, 0.027 S and balance Fe. A 2.9 mm-thick as-cast strip was produced using a laboratory vertical twin-roll strip caster and quenched by cold water. During casting, a relatively low melt superheat was employed to produce a solidification microstructure mainly consisting of equiaxed grains. The experimental details of this process have been reported previously [15,16]. The as-cast strips were reheated to 1130 °C, hot rolled to 2.4 mm with 17.2% reduction in one pass, cooled in air for a few seconds and quenched by cold water. The sheets were then subjected to the normalizing in which they were first soaked at 1130 °C for 2 min, cooled to 930 °C in air, soaked at 930 °C for 2 min and quenched in boiling water. After this, the sheets were first cold rolled to 1.1 mm with 54.2% reduction and followed by first intermediate annealing at 830 °C for 5 min. Then, they were second cold rolled to 0.45 mm with 59.0% reduction and subjected to second intermediate annealing at 830 °C for 5 min. Subsequently, the sheets were third cold rolled to the final thickness of 0.10 mm with 77.8% reduction and subjected to primary recrystallization annealing at 830 °C for 5 min in a wet atmosphere of 75% H_2 and 25% N_2. Finally, the sheets were subjected to secondary recrystallization annealing in which they were heated from 800 °C to 1200 °C at a rate of 15 °C/h in a 75% H_2 and 25% N_2 atmosphere and soaked at 1200 °C for 20 h.

The microstructures on the rolling direction (RD) and normal direction (ND) section were etched with a 4% nital and examined using a Leica optical microscope. The secondary recrystallization microstructures on the RD and transverse direction (TD) section were revealed by 10% hydrochloric acid. For electron backscattered diffraction (EBSD) measurements, the samples were wet ground using silicon carbide papers, mechanically polished and electropolished with a 14% perchloric acid alcohol solution using a Buehler ElectroMet 4 polisher. The EBSD system was installed on a Zeiss Ultra 55 scanning electron microscope (SEM) and the EBSD maps were collected at 20 kV using a 70° tilt. The textures were measured based on X-ray diffraction across the thickness. The measured layer is defined as a parameter S=2a/d, where a and d are the distances from the center and sheet thickness, respectively. The orientation distribution functions (ODFs) were calculated from three incomplete pole figures {110}, {200} and {211} by the series expansion method (I_{max}=22). The size distribution of the inhibitors was determined using SEM and a Tecnai G2 F20 transmission electron microscope. The chemical composition of inhibitors was verified by means of energy dispersive X-ray spectroscopy. The magnetic properties of the samples sheared to 100 mm×30 mm were measured by using a tester.

3. Results

The microstructure of the as-cast strip was composed of equiaxed ferrite grains and martensite, see Fig. 1a. The associated texture was almost random throughout the thickness. This kind of solidification microstructure and texture has been attributed to the experimental conditions during strip casting [17].

The hot rolled microstructure was mainly characterized by the deformed ferrite grains, many colonies consisting of small equiaxed ferrite grains and pearlite and martensite, Fig. 2a. A quite weak texture was observed on the surface, Fig. 3. A near λ-fiber texture (< 001 > //ND) evolved in the S=0.5 layer and a weak α-fiber texture (< 110 > //RD) dominated in the center layer. After normalizing, the microstructure was composed of ferrite, pearlite and carbides (Fig. 2b). It was found that the normalized sheet showed a more dispersive distribution of pearlite and it had a similar texture to that of hot rolled sheet.

After first cold rolling, an inhomogeneous deformation microstructure was produced, Fig. 2c. Some ferrite grains exhibited dense shear bands and deformation bands while other grains showed very few substructures. The associated texture was characterized by a strong α-fiber texture (< 110 > //RD) and γ-fiber (< 111 > //ND) texture, Fig. 4. A very sharp texture gradient evolved across the thickness, i.e., the intensity of texture decreased remarkably from the surface to the center. After first intermediate annealing, an inhomogeneous recrystallization microstructure was produced, see Fig. 2d. As shown in Fig. 5, a relatively strong Goss texture evolved in the S=0.5 and S=0 layers. In the surface layer, a near cube texture ({001} < 100 >) and a weak Goss component were observed.

After second cold rolling, a relatively homogeneous deformation microstructure was produced, Fig. 2e. As shown in Fig. 6, the texture was characterized by a strong α-fiber texture, a medium γ-fiber texture with the maximum near {111} < 112 > component and a λ-fiber texture, which was similar to first cold rolled texture. The texture gradient was no longer as sharp as that in Fig. 4. After second intermediate annealing, a relatively uniform recrystallization microstructure was produced (Fig. 2f), which was finer and more homogeneous than that in Fig. 2d. As shown in Fig. 7, the associated texture was characterized by a medium γ-fiber texture and Goss texture, differing from first intermediate annealed texture in which γ-fiber texture was absent.

After third cold rolling, a homogeneous deformation microstructure was observed, as shown in Fig. 2g. The associated texture was characterized by a strong γ-fiber texture, a medium α-fiber texture and λ-fiber texture, see Fig. 8. After primary recrystallization annealing, a uniform microstructure consisting of fine ferrite grains was produced, as shown in Fig. 2h. It was measured that the average grain size was only 11.5 µm. As shown in Fig. 9, the texture was characterized by a medium α-fiber texture (from {118} < 110 > to {332} < 110 >) and a strong γ-fiber texture with the maximum at {111} < 112 > component.

Only a few MnS precipitates were observed in as-cast strip. After hot rolling, many fine MnS precipitates evolved along grain boundaries and inside the grains. After normalizing, a large number of fine and dispersed MnS precipitates formed, see Fig. 10. Most precipitates ranged in the size from 15 nm to 90 nm and they may act as effective inhibitors due to strong Zener drag effect originating from the large volume fraction and small size [18].

After secondary recrystallization annealing, a microstructure consisting of large Goss grains together with a sharp Goss texture was produced, see Fig. 11. The magnetic induction B_8 and iron loss $P_{10/400}$ of the sheet was 1.79 T and 6.9 W/kg, respectively. The microstructure and texture in Fig. 11 indicated that a stable secondary recrystallization evolved during the final annealing, significantly differing from the previous reports.

4. Discussion

4.1. Initial microstructure and texture of the as-cast strip

The melt superheat during strip casting has a critical effect on the initial solidification structures [17]. In case of relatively low superheat in this work, rapid heterogeneous nucleation tends to occur near the roll surfaces in a large undercooled region. The heterogeneous nuclei may move towards the liquid by convection motion to promote the formation of an equiaxed grain structure [19]. Thus, a microstructure consisting of equiaxed δ-ferrite grains with random orientations was produced just after solidification. Then, some austenite precipitated from the parent δ-ferrite during air cooling and finally transformed into martensite during water quenching. It is known that austenite may obey Kurdjumov-Sachs or Nishiyama-Wasserman relationships with respect to its parent δ-ferrite during δ→γ transformation [20,21]. Martensite may also obey these relationships with its parent austenite [22]. However, giving that the volume fraction of martensite was below 10% due to limited δ→γ transformation, the transformation texture