



Research articles

Ultra-thin grain-oriented silicon steel sheet fabricated by a novel way: Twin-roll strip casting and two-stage cold rolling



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ABSTRACT

0.05–0.15 mm-thick ultra-thin grain-oriented silicon steel sheets were successfully produced by a novel processing route including strip casting, hot rolling, normalizing, two-stage cold rolling with intermediate annealing, primary recrystallization annealing and secondary recrystallization annealing. The evolutions of microstructure, texture and inhibitor along the processing were briefly investigated. The results showed that the initial Goss orientation originated due to the heterogenous nucleation of δ -ferrite grains during solidification. Because of the lack of shear deformation, only a few Goss grains were observed in the hot rolled sheet. After the first cold rolling and intermediate annealing, Goss texture was enhanced and distributed in the whole thickness. A small number of Goss grains having a high fraction of high energy boundaries exhibited in the primary recrystallization annealed sheet. A large number of fine and dispersed MnS and AlN and a few co-precipitates MnS and AlN with the size range of 10–70 nm were also observed. Interestingly, a well-developed secondary recrystallization microstructure characterized by 10–60 μ m grains and a sharp Goss texture were finally produced in the 0.05–0.15 mm-thick ultra-thin sheets. A magnetic induction B_8 of 1.72–1.84 T was obtained. Another new finding was that a few $\{2\ 3\ 0\}\langle 0\ 0\ 1\rangle$ and $\{2\ 1\ 0\}\langle 1\ 2\ 7\rangle$ grains also can grow up abnormally because of the high fraction of high energy boundaries and the size and number advantage, respectively. These non-Goss grains finally deteriorated the magnetic properties of the ultra-thin sheets. In addition, low surface energies of $\{hk0\}$ planes may also contribute to the abnormal growth of Goss, $\{2\ 3\ 0\}\langle 0\ 0\ 1\rangle$ and $\{2\ 1\ 0\}\langle 1\ 2\ 7\rangle$ grains.

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1. Introduction

Ultra-thin grain-oriented silicon steel is mainly used as the core material in the electrical appliances working at high frequencies (≥ 400 Hz) because of its excellent high-frequency magnetic properties [1–4]. Although the manufacturing process of grain-oriented silicon steel has been well developed, the difficulties in terms of stable secondary recrystallization remain to be overcome when producing the ultra-thin sheets. This is thought to be related with the rapid coarsening and decomposition of the inhibitors during secondary recrystallization annealing, leading to the deteriorated pinning effects. Hence, an incomplete secondary recrystallization microstructure containing many fine non-Goss grains was usually produced [5,6]. Thus, it is difficult to produce grain-oriented silicon steel sheet thinner than 160 μ m by the conventional secondary recrystallization process [4]. Nowadays, ultra-thin grain-oriented silicon steel sheet has to be produced based on tertiary recrystallization [7–9]. In this process, the 0.30 mm-thick grain-oriented

silicon steel product is cold rolled to the required thickness and subjected to the tertiary recrystallization annealing for several hours in order to obtain sharp Goss texture. Considering that the current process is quite complicated, the researchers have been making great efforts to establish a simple process to produce the ultra-thin sheet.

Up to now, twin-roll strip casting is a novel process which can fabricate 1–5 mm-thick steel sheets directly. Because of its superiority of controlling the solidification microstructure and shorting the manufacturing process, it has been applied in producing the low-carbon steel [10–12], stainless steel [13–16] and non-oriented silicon steel [17–20]. Recently, Liu et al. [21–23] have successfully produced the 0.23–0.27 mm-thick grain-oriented silicon steel sheets based on twin-roll strip casting process. The evolutions of the microstructure, texture and inhibitor along the processing route were investigated in detail. It was found that the formation of precipitates was remarkably suppressed due to the sub-rapid solidification and the subsequent water quenching. Thus, compared with the conventional process, a large number of desired fine inhibitors could be more easily obtained by normalizing. And more effective inhibitors are very useful for forming stable and fine

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primary recrystallization microstructure. Thus, in this way, the secondary recrystallization of ultra-thin grain-oriented silicon steel may be stabilized, giving rise to the formation of complete secondary recrystallization microstructure.

In the present work, 0.05–0.15 mm-thick ultra-thin grain-oriented silicon steel sheets were successfully fabricated based on strip casting and secondary recrystallization. The evolutions of the microstructure, texture and inhibitor along the processing route were investigated with much attention to the mechanism of abnormal grain growth in ultra-thin grain-oriented silicon steel.

2. Experimental procedure

The chemical composition (wt%) of the tested steel was 3.2 Si, 0.055 C, 0.085 Mn, 0.027 S, 0.008 N, 0.005 Als and balance Fe. A 2.9 mm-thick as-cast strip was produced using a laboratory vertical twin-roll strip caster with low melt superheat and quenched by cold water, as described in detail in previous reports [19,20]. 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 hot rolled 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 normalized sheets were first cold rolled to 0.9 mm with 62.5% reduction and followed by the intermediate annealing at 830 °C for 5 min. Then, they were cold rolled to three final thicknesses, i.e., 0.15 mm with 83.3% reduction, 0.10 mm with 88.9% reduction, and 0.05 mm with 94.4% reduction. Then, the second cold rolled sheets were primary recrystallization annealed at 830 °C for 5 min in a wet atmosphere of 75% H₂ and 25% N₂. Finally, these 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 N₂ atmosphere and soaked at 1200 °C for 20 h.

Metallographic specimens were machined, polished and etched with 4% nital. A Leica optical microscope was applied on longitudinal sections as defined by the rolling direction (RD) and the normal direction (ND). The secondary recrystallization microstructures on the RD and transverse direction (TD) section were revealed by 10% hydrochloric acid. The crystal orientation maps were determined by the OIM 4000 Electron Backscatter Diffraction (EBSD) system equipped at FEI Quanta 600 Scanning Electron Microscope (SEM). 5 regions (800 μm × 150 μm, 800 μm × 100 μm and 800 μm × 50 μm, respectively) on the RD-ND section of each thickness primary recrystallization annealed sheet were scanned to calculate and analyze the grain boundary character distribution (GBCD). The fractions of special boundaries were defined as the parameter $f = n/m$, where n and m represented the length of high energy (HE) boundaries or coincidence site lattice (CSL) boundaries surrounding the grains with certain orientations and the total boundaries, respectively. The inhibitors were observed using a TECNAI G2 F20 Transmission Electron Microscope (TEM). In order to investigate the size distribution of inhibitors, 40–50 regions (3.2 μm × 3.2 μm) were randomly examined using an image analyzer. The chemical composition of inhibitors with the number higher than 30 was verified by means of energy dispersive X-ray spectroscopy (EDX). The texture was examined through the thickness by measuring three incomplete pole figures {1 1 0}, {2 0 0} and {2 1 1} in Bruker D8 Discover X-ray diffractometer. 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. Three specimens with 100 mm × 30 mm were cut from the secondary recrystallization annealed sheets of each thickness for the measurement of magnetic properties. Magnetic inductions at 800 A/m (B₈) and iron losses at 1.7 T, 50 Hz (P_{17/50}); 1 T, 400 Hz (P_{10/400}); 1.5 T, 400 Hz (P_{15/400}); 1 T, 1000 Hz (P_{10/1000}) were measured in a single sheet

tester. Then three series of magnetic properties data were obtained for every annealed sheet and used to calculate the average value in the present work.

3. Results

3.1. Evolution of microstructure along the processing route

The microstructure of the as-cast strip was composed of equiaxed ferrite grains and martensite, see Fig. 1a. After hot rolling with limited reduction, the microstructure was mainly composed of large deformed ferrite grains and the colonies of small equiaxed ferrite grains, pearlite and martensite, see Fig. 1b. The normalized sheet showed coarser microstructure than the hot rolled sheet, see Fig. 1c. After first cold rolling, a remarkably inhomogeneous microstructure composed of pearlite, carbides and elongated ferrite grains with dense in-grain shear bands and deformation bands was produced, see Fig. 1d. After intermediate annealing, the sheet showed a recrystallization microstructure composed of inhomogeneous ferrite grains, see Fig. 1e. After second cold rolling, a homogeneous deformation microstructure was produced, see Fig. 1f. The primary recrystallization annealed sheets showed a fully recrystallized microstructure composed of fine ferrite grains, see Fig. 1g.

3.2. Evolution of texture along the processing route

The texture of the as-cast strip was almost random through the whole thickness, see Fig. 2. After hot rolling, a near λ -fiber ($\langle 0 0 1 \rangle // ND$) texture and a weak α -fiber ($\langle 1 1 0 \rangle // RD$) texture evolved in the $S = 0.5$ layer and the $S = 0$ layer, respectively, while a quite weak texture appeared in the $S = 1.0$ layer, see Fig. 3. After subsequent normalizing, the main texture components enhanced and Goss texture with the intensity of 4.84 appeared in the $S = 0$ layer, see Fig. 4. After first cold rolling, the associated texture was mainly characterized by strong α -fiber texture and medium γ -fiber ($\langle 1 1 1 \rangle // ND$) texture with the maximum at $\{1 1 1\} \langle 1 1 0 \rangle$ component through the whole thickness, see Fig. 5. After intermediate annealing, a medium Goss texture evolved through the whole thickness, and a weak near cube texture ($\langle 0 0 1 \rangle \langle 1 0 0 \rangle$) appeared in the surface, see Fig. 6. It was found that the intensity of Goss texture was decreased from 5.95 in the $S = 1.0$ layer to 3.26 in the $S = 0$ layer. After second cold rolling, the texture was mainly characterized by a strong α -fiber texture, medium λ -fiber texture and strong γ -fiber texture, see Figs. 7a–9a. It was found that the densities of both the α -fiber texture and the γ -fiber texture in the $S = 0$ layer have increased by nearly 2 times with the thickness decreasing from 0.15 mm to 0.05 mm. After primary recrystallization annealing, the texture was characterized by medium α -fiber texture and strong γ -fiber texture with the maximum $\{1 1 1\} \langle 1 1 2 \rangle$ component, see Figs. 7b–9b. The density of the γ -fiber texture was found to increase by nearly 60% as the sheet thickness reducing from 0.15 mm to 0.05 mm.

3.3. Inhibitors of the primary annealed sheet

It was found that the inhibitors in the primary recrystallization annealed sheets are mainly MnS and AlN and a few co-precipitates of MnS and AlN, see Figs. 10 and 11. The densities of inhibitors in the 0.15 mm-thick, 0.10 mm-thick and 0.05 mm-thick sheets were 5.20/μm², 5.89/μm² and 3.12/μm², respectively. The corresponding average sizes of inhibitors were 26.36 nm, 28.72 nm and 51.65 nm, respectively. In the 0.15 mm-thick and 0.10 mm-thick sheets, a large number of fine inhibitors with a diameter of 10–40 nm were observed, see Fig. 11d. In contrast, the size of inhibitors in the 0.05 mm-thick sheet was range of 10–70 nm.

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