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Inhibitor induced secondary recrystallization in thin-gauge grain oriented silicon steel with high permeability



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ABSTRACT

Thin gauge grain-oriented (GO) silicon steel with high permeability was successfully processed by a simple way based on strip casting process, without hot rolling and decarburization annealing. The primary annealed sheet exhibited unique characteristics of homogeneous precipitation because of near-rapid solidification, and fine-grained recrystallized microstructure with relatively strong Goss texture and pronounced {111} (112) texture, which was responsible for the subsequent Goss abnormal growth. MnS, (Nb,V)N and AlN precipitates provided strong pinning effect on grain boundaries at different stages during the final annealing process, which is referred as "sequential inhibition behavior". The thin gauge GO silicon steel experienced complete secondary recrystallization induced by inhibitor, and exhibited sharp Goss texture together with significantly superior magnetic properties (magnetic induction $B_8 = 1.98$ T; iron loss $P_{1.7/50} = 0.7$ W/kg).

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1. Introduction

GO silicon steel is one of the most widely used as core material in transformers and exhibits excellent magnetic properties which are closed related to the sharpness $\{110\}$ (001) (Goss) texture [1]. In the past decade, the focus of research in grain-oriented (GO) silicon steel was aimed at increasing magnetic induction and reducing iron loss without increasing the production cost, which has significant contribution in reducing electrical consumption and protecting the global environment [2,3]. Thin gauge GO silicon steel is an effective approach to meet the requirements of core material for high-frequency transformers, which has obvious advantages of high saturation magnetization over nanocrystalline materials, even though there are some difficulties associated with processing, particularly in terms of formability and stable secondary recrystallization [4-6]. It is generally difficult to produce grain-oriented silicon steel sheets of thickness less than ~160 µm by secondary recrystallization induced by inhibitors, because the annealing atmosphere and surface condition have an obvious influence on the ripening of inhibitor [7]. Recent attempts to solve the dilemma have led to two primary approaches. The first approach is based on the retention of Goss texture, where secondary recrystallized silicon steel was rolled to less than 0.10 mm thick and subsequently annealed in vacuum to obtain sharp Goss texture [8,9]. The second approach involved multi-stage cold rolling with intermediate annealing and high temperature annealing of pure Fe—Si hot rolled sheet. The driving force for selective grain growth of (110) [001] is the difference in surface energy between (110) and other planes, and is known as surface energy-induced tertiary recrystallization [10,11]. Unfortunately, these techniques are far from industrial applications on a large scale due to high production cost, the environmental impact and the limited product size of products fabricated in above approaches.

While a significant effort is being made to develop an appropriate process, a cost-effective process to produce thin gauge GO silicon steel with superior magnetic properties has not yet been developed and requires further research. In this regard, the novel advanced and energy-efficient process, strip casting technology, provides a promising possibility to produce steel that eliminates continuous casting, high temperature reheating, and multi-pass hot rolling, adopted during conventional processing [12–14]. Strip casting progress has attracted significant attention to manufacture steels over the world and shows a great potential advantage in texture control over conventional process [15]. Most of the corresponding investigations focus on the fabrication technique of non-oriented or regular thickness GO silicon steel in the recent years [16–19]. Moreover, thin gauge GO silicon steel processed by strip casting process has not been explored to the best of our understanding.

In the present study, a 0.08–0.15 mm thick GO silicon steel with high permeability was successfully processed by strip casting, and involved

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Fig. 1. Schematic diagram of the present process.

normalizing, two-stage cold rolling with intermediate annealing, primary recrystallization and secondary recrystallization annealing. The evolution of microstructure, texture and effect of inhibitor are elucidated during different processing stages.

2. Materials and experimental procedure

The schematic diagram of the present process is shown in Fig. 1. The GO silicon steel of 2.5 mm thickness with nominal chemical composition (in weight %) of 0.005% C, 2.9% Si, 0.2% Mn, 0.02% Al, 0.05% Nb, 0.03% V, 0.02% S, 0.018% N, was produced by a vertical type laboratory twin-roll caster with melt superheat of 30 °C, and subsequently cooled by cold water to room temperature. Next, the as-cast strip was normalized at 1050 °C for 5 min and the normalizing strip was first cold rolled to 0.55 mm in thickness with a reduction of ~78% and subjected to intermediate annealing at 950 °C for 5 min, followed by secondary cold rolling to 0.08–0.15 mm thickness with reduction range of 73%–85%. The cold rolled sheets were further annealed at 830 °C for 5 min in an atmosphere of 100% N₂ for primary recrystallization. Lastly, the primary annealed sheet was heated to 1200 °C at a rate of 20 °C/h for secondary recrystallization in an atmosphere of 75% H₂ and 25% N₂ and then annealed at 1200 °C for 10 h in 100% H₂ for purification.

The evolution of microstructure, texture, and precipitation of ~0.08 mm thickness GO silicon steel was studied in detail. The microtexture of the specimens was studied along the longitudinal sections defined by the rolling direction (RD) and the normal direction (ND), using electron backscatter diffraction (EBSD) facility attached to a Zeiss Ultra 55 field emission scanning electron microscope. The precipitates were studied via transmission electron microscope (TEM) equipped with an energy dispersive X-ray spectroscopy. The magnetic properties of secondary annealed specimens were measured using a single sheet tester in the rolling direction with 100 mm length and 30 mm width.

3. Results and discussion

The as-cast strip was characterized by coarse equiaxed grains of ~200–350 μ m (Fig. 2(a)) through thickness, and initial solidification texture consisted of relatively strong {100} fiber (Fig. 2(b)). In the case of rapid solidification with low melt superheat (30 °C), rapid heterogeneous nucleation occurred along the solid/liquid interface during the early stage of solidification. Hence, grains with (100) direction had the advantage to preferentially grow with respect to the vertical heat flow direction over the other oriented grains, leading to the initial texture, referred as selective growth mechanism for body-centered cubic structure [20]. As a result, the initial solidification microstructure was supposed to be a consequence of combination of heterogeneous nucleation and selective growth under the 30 °C melt superheat condition. Compared with the conventional hot-rolled sheet, the as-cast strip exhibited coarser grains and much weaker through-thickness texture gradient, which is quite similar to the previous results by Raabe [21].

Given that it is difficult to manifest an ideal secondary recrystallization when the thickness of the product sheet is reduced, thus, the evolution of precipitates is considered to be a key metallurgical factor in processing GO silicon steel with high permeability. The evolution of inhibitor in the present process was quite distinct from that of the conventional process, as shown in Fig. 3. Only few coarse MnS and (Nb,V)N particles with a diameter of ~40-150 nm preferentially precipitated at the grain boundaries in the as-cast strip (Fig. 3(a)). This implied that nucleation and growth of precipitates was effectively suppressed because of rapid cooling rate, leading to highly supersaturated solid solution matrix [19]. A number of particles of size ~50-150 nm were observed at grain boundaries and some intragranular (Nb,V)N precipitates of ~30-80 nm were newly formed in normalized band, as shown in Fig. 3(b). After the first stage of cold rolling and intermediate annealing, fine precipitates were nucleated and dispersed in the matrix (Fig. 3(b)), where spherical particles are MnS of diameter ~ 60-100 nm and rod-like are



Fig. 2. (a) EBSD orientation map and (b) orientation distribution function (ODF) section at constant $\phi_2 = 45^\circ$ of the as-cast strip.

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