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Effect of annealing atmosphere on secondary recrystallization in thin-gauge grain-oriented silicon steel: evolution of inhibitors

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ABSTRACT

Thin-gauge cold-rolled grain-oriented (TG-CRGO) silicon steels were fabricated using the acquired inhibitor method, in which the behaviors of inhibitor are strongly influenced by the final annealing atmosphere and differ from that of the TG-CRGO silicon steel. In this study, macrostructures, grain orientations, and evolution of inhibitors in two gauges of thickness (0.23 and 0.18 mm) of steels were studied and compared in five different annealing atmospheres. The second-phase particles in samples were observed by field-emission scanning electron microscopy. Distribution densities and mean diameter of particles were statistically calculated to illustrate the effect of Zener factor. Results indicated that the inhibitor behaviors in the two thickness samples are similar with the change of N_2 proportion in annealing atmosphere, i.e., nitrogen loss, decomposition, coalescence, and ripening of particles would be strongly suppressed with the increase of N_2 content. However, in comparison with the 0.23-mm samples, the nitride precipitation, decomposition and inhibitor coarsening of the 0.18-mm specimens are more likely to occur at a lower temperature because of the thinner thickness. Furthermore, the value of Zener factor implies that for thinner samples, more N_2 should be employed when nitrides are used the main inhibitor; this condition would inhibit the development of undesirable deviated Goss grains. This study showed that the 0.23-mm sample could achieve the best magnetic properties (B_8) at 1.92 T in the atmosphere containing 25% N_2. The 0.18-mm sample could achieve the best magnetic properties (B_8) at 1.93 T in the atmosphere containing 75% N_2.

1. Introduction

Grain-oriented silicon steel is an important soft magnetic metallic material that has excellent magnetic properties in terms of magnetic induction intensity (***B***) and iron loss ($\boldsymbol{P}_T$). It is mainly used for making power transformer's core. High magnetic induction (***B***) can be obtained through preparing polycrystalline sheets with a strong preferential orientation of Goss ({011} < 100 >) by complex and fine craft. Meanwhile, low iron loss ($\boldsymbol{P}_T$) can be obtained by reducing the inclusions, crystal defect, and internal force, improving the surface quality, or using domain refining techniques. With the development of industrial production technology of oriented silicon steel, the methods for increasing the occupancy of Goss grains to improve the magnetic induction and iron loss properties have become increasingly difficult. Thickness thinning of silicon steel can significantly reduce iron loss (mainly eddy current loss), thus becoming a main trend in development of future technology. However, production of thinner steels requires more cold rolling deformation, which leads to microstructural and textural deformation different from thick-gauge silicon steel. The considerable difference, particularly in the significant reduction in proportion of Goss orientation component, would cause less competitive advantage of abnormal Goss grains growth during the final annealing process [1], thus posing challenges for industrial production. In addition, the inhibitors near the surface layer ripen and decompose easily as the thinner thickness of steels during secondary recrystallization, which would facilitate weakening of inhibitions, thus causing negative effect on the abnormal Goss growth [2]. Accordingly, fully understanding the evolution behavior of the inhibitors in the thin-gauge oriented silicon steel is necessary to control the abnormal growth of Goss grains.

The technology of the preparation of inhibitors for the production of high magnetic induction oriented silicon steel (Hi-B type) can be classified into two categories [3–7], namely, the inherent inhibitor method of high-temperature (> 1573 K) and medium-temperature (1473 K to 1573 K) slab reheating technology, and the acquired inhibitor method of low-temperature (1373 K to 1473 K) slab reheating

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technology [8]. The high slab heating temperature of the inherent inhibitor method will result in high energy consumption, high cost, and low rate of finished product, etc. Thus, the acquired inhibitor method of low temperature (1373 K to 1473 K) slab reheating technology become the focus of the research and development of industrial production technology. Comparison between the original alloy composition of the inherent inhibitor method and the acquired inhibitor method revealed a considerable difference. The precipitates in sheets of the acquired inhibitor method would be less in the hot rolling process, and could not satisfy the needs for secondary recrystallization. Therefore, increasing the nitriding treatment process is necessary to obtain enough nitride particles and to increase the inhibitions.

Final annealing is the key step of the development of secondary recrystallization, during which the distribution and transformation of nitride particles play a decisive role in the abnormal growth of Goss grains. Thus, studying the evolution behavior of the nitride in the final annealing process is important to improve the magnetic properties of Hi-B steel and reveal the mechanism of the formation of Goss grains in secondary recrystallization. Numerous studies [9–15] have focused on the effect of inhibitor types, inhibitor content, and annealing atmosphere on the secondary recrystallization in the final annealing process. For the inherent inhibitor method, with a higher H_2 content in the final annealing atmosphere, the secondary recrystallization temperature drops, the secondary recrystallization time shortens, the final grain size of the oriented silicon steel becomes finer, and the magnetic induction decreases [8]. Furthermore, the AlN precipitation is continuous during the final annealing process and is strongly suppressed by high N_2 partial pressure, which raises secondary recrystallization temperature [9] to obtain a sharp Goss texture and high magnetic induction [9,16]. However, study also shows that the secondary recrystallization temperature would be lower, and AlN is more likely to decompose at a lower temperature, leading to better magnetic properties at higher H_2 partial pressure [12]. In comparison with the inherent inhibitor method, few studies on the acquired inhibitor method have been reported, which mainly focus on the conversion of nitride in the heating process [17], effect of different nitrogen content on secondary recrystallization [11], and change of AlN inhibitors under different atmosphere conditions at 920 °C with different soaking times [18]. The systematic research on the evolution behavior of the inhibitors in the final annealing process is rarely reported. Particularly, the mechanisms of evolution of the inhibitor behavior in different annealing atmospheres and during the heating process of the plate with different thicknesses remain unclear. Currently, experimental analysis of inhibitors has not been reported; thus, a theoretical basis for the acquired inhibitor method cannot be provided to control the inhibitor behavior and the formation of Goss texture through adjusting N_2 partial pressure in final annealing process.

The evolution behaviors of the inhibitors are sensitive to the final annealing atmosphere and strongly influenced by temperature. In this study, the differences of conversion of particles in the 0.23-mm and 0.18-mm samples under different temperatures and atmospheres were compared. In addition, the particle sizes and distribution densities were measured and the effect of ripening behavior of particles on the formation of Goss texture was analyzed as the thickness of plates reduced. The results provide a practical basis for the improvement of the preparation technology and the mechanism of Goss texture formation of thin-gauge cold-rolled grain-oriented (TG-CRGO)steels.

2. Experimental procedure

Industrial hot rolled plates of the low temperature method with 2.23 mm thicknesses were used as raw materials, which mainly contained 0.05% C, 3.0% Si, 0.1% Mn, 0.005% S, 0.03% Al, 0.006% N, 0.03% Sn, and the balance Fe (mass fraction, wt%). After normalizing, two kinds of different thickness gauges of 0.23 and 0.18 mm sheets were prepared by once cold rolling process, and then the cold-rolled sheets were treated by decarburizing annealing, nitriding, and MgO coating. Then, the annealing treatment was conducted under five different atmospheric conditions. The annealing institution is shown in Fig. 1, which is similar to our previous study, Effect of annealing atmospheres on secondary recrystallization in thin-gauge grain-oriented silicon steel: microstructures and textures. In the range of 1223 K to 1298 K, the samples were taken out from the annealing furnace every 25 K to observe the changes of particles in different atmospheres (mixture gas of H_2 and N_2) during the final annealing process. Given that the abnormal Goss growth occurs near the surface layer [15], in which the particles change most dramatically, the study of the particle changes in this region was the most significant. Accordingly, the interrupting samples were grinded to the surface region, followed by polishing and erosion treatment. Then the particles were observed by scanning electron microscopy (LEO-1450) and transmission electron microscopy (G2 F30 Tecnai) with energy-dispersive spectroscopy (EDS). In addition, the magnetic induction of samples with size 30 mm (TD)×300 mm (RD), was measured with an MPG200D soft magnetic measuring instrument (BrockHaus Measurements) under the a magnetic field of 800 A/m. The orientation of the final two samples was analyzed by Oxford HKL Channel5 electron backscatter diffraction.

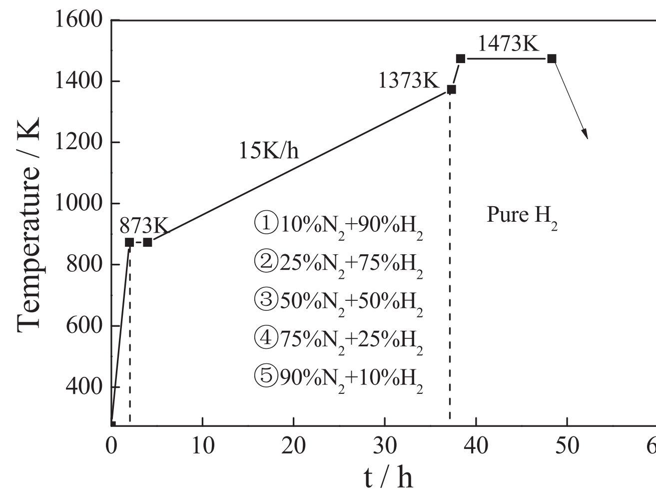

Fig. 1. Schematic diagram of annealing experiment with five different atmospheres.

3. Results and discussions

3.1. Magnetic performance

As shown in Fig. 2, all the specimens of the two kinds of steel in five different annealing atmospheres were found to undergo secondary recrystallization completely. Based on the properties B_8 and {200} pole figure of specimens in different annealing atmospheres, the 0.23-mm and 0.18-mm thick specimens obtained the sharpest Goss texture and the best properties, which were 1.92 T and 1.93 T in the atmosphere containing 25% N_2 and 75% N_2 (volume fraction, vol%), respectively, as shown in Table 1. In other atmospheres, a number of grains appeared in the specimens, which deviated from Goss orientation more than 7° (named as deviated Goss grains in this paper), leading to lower magnetic inductions.

3.2. Observation of second-phase particles

Two different precipitates, mainly nitrides and MnS, occur in the nitride samples. However, the particle number per unit area of MnS is much less than that of the nitrides because of the slab composition, and MnS rarely plays the function of inhibitors during low temperature method [19]. After the nitriding process, the nitrides were mainly Si_3N_4