극박 3%규소강에서 Mn이 황의 편석 거동 및 자성특성에 미치는 효과

Effects of Mn on Sulfur Segregation and Magnetic Induction in Thin-gauged 3%Si-Fe Strip

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Abstract

Effects of addition of manganese and final reduction on segregation behavior of sulfur and final mangetic induction during final annealing have been investigated in the 300 ppm sulfur-contained 3% silicon-iron alloy strips with or without manganese. At the same concentration of sulfur, lower final reduction is favorable for final Goss texture. This is because the probability that the initial Goss grains survive under the highly segregated sulfur atmosphere and grow selectively within the segregated sulfur-free time range becomes higher. In the case of 3% silicon-iron with manganese, much lower magnetic induction was obtained, although the weak final reduction of 30% is given to the alloy, comparative to the 40%. This is because MnS particles acted as an reducer in the primary grain size.

Key Words : Mn, Sulfur Segregation, Magnetic Induction, 3%Si-Fe, Surface-energy-induced Selective Growth

1. Introduction

In 3% Si-Fe alloys containing sulfur [1-4], the sulfur segregates to the sample surface and grain boundaries during final annealing, and then is lost to the vacuum or H_2 atmosphere through evaporation or H_2S reaction. While a high surface concentration of sulfur is present, there is a strong tendency for grains with {001} or {111} surface orientation to grow. If all of Goss grains are lost within the time range of highly segregated sulfur during annealing, the final material has poor orientation and poor magnetic properties. If some of the Goss grains survive, they grow to dominate the final Goss texture after the sulfur is lost, resulting in a strong Goss texture and high magnetic properties. Furthermore, the bad effects of high sulfur concentration can be offset by reducing the final reduction from 60 to 40%. This reduces the grain size after initial recrystallization, which enables some of the Goss grains to survive. Effects of addition of manganese and final reduction on segregation behavior of sulfur and final magnetic induction during final annealing are investigated in this study.

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2. Experimental Procedure

Two kinds of 100μm thick 3%Si-Fe strips, which contain 300 ppm bulk content of sulfur and additional manganese, were prepared through vacuum induction melting, hot- and three-stage cold-rolling processes. In the alloy without manganese, final reduction of 60%

was mostly given to the alloys. Final reduction of 40% was also given to the 300 ppm sulfur-contained alloy in order to investigate the difference in recrystallization kinetics with final reduction. An intermediate annealing was given to the alloy at 800℃ for 1.8 ks. The alloy with manganese was given to the final reduction of 30% followed by an intermediate annealing at 1000℃ for 3.6 ks under a vacuum of 6 x 10^{-6} Torr. Most of final annealing was carried out at 1200℃ under the same vacuum condition in the case without manganese and under hydrogen atmosphere in the other case. Texture was analyzed with ODF (orientation distribution function) and an etch-pit method. Surface segregation behavior on the strips was investigated with an ion-sputtering technique in an AES (Auger electron spectroscope) after final annealing and fast cooling. The primary beam energy was 2 keV. The differential peak heights were obtained every 15 seconds during ion-sputtering. The silicon, carbon and oxygen peaks around 92, 272 and 503 eV were chosen. In order to minimize the contamination effect from air, the peak height of S 150 eV were normalized with the peak height, 90, of Fe 703 eV that was obtained after ion sputtering for 4~8 min. Magnetic induction (B_{10}, Tesla) was measured with a DC-fluxmeter under a magnetic field of 1000 A/m.

3. Results and Discussion

During final annealing of the strip with final reduction of 40%, the convex profile of segregated sulfur corresponded to the trough of magnetic induction

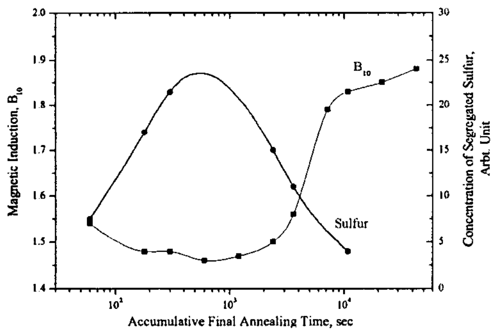

Fig. 1. Correlation between interfacial segregation of sulfur and magnetic induction in the 3% silicon-iron strips without manganese.

during final annealing at 1200℃. Such a segregation behavior of sulfur, which shows a maximum, arises from the competition between segregation and evaporation of segregated sulfur.

The effect of final reduction on magnetic induction of the 3% silicon-iron alloy without manganese is shown in Fig. 2. The saturation in magnetic induction after final vacuum annealing at 1200℃ was much higher in the case of final 40% reduction.

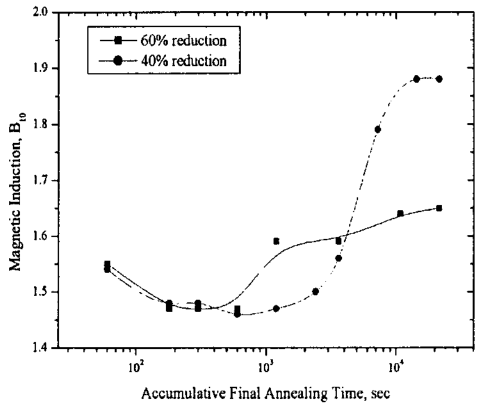

Fig. 2. Effect of final reduction on magnetic induction of the 3% silicon-iron alloy without manganese.

Under a fixed final reduction (final reduction of 60%), Goss texture after final annealing for 30 sec was not observed in the strip containing 300 ppm bulk content of sulfur which shows a relatively higher concentration of surface-segregated sulfur, as shown in Fig. 2. However, this strip with final reduction of 40% showed a weak Goss texture. At the lower final reduction, grain size at an initial annealing time t is generally larger, because the growth rate of grain is higher than the nucleation rate. At the same concentration of surface-segregated sulfur, the surface-energy- induced selective grain growth rate is, therefore, higher in case of smaller average grain size [4]. Based on present results, as the degree of final reduction increase, the simplified evolution of initial texture after final annealing changes from strong {011}<100> + weak {111}<uvw> to {001}<uvw> + {111}<uvw> and to strong {111}<uvw>.

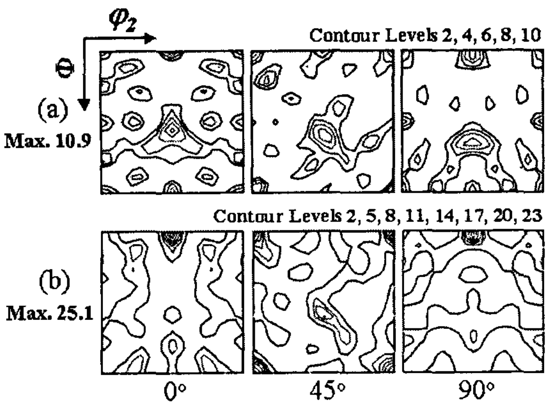

Fig 3. The difference in ODF of the 3% silicon-iron strips without manganese after final vacuum annealing: (a) the 40% cold-rolled strip annealed for 3.6 ks and (b) the 60% cold-rolled strip annealed for 2.4 ks.

This results in the lower magnetic induction in the case of final 40% reduction, as shown in Fig. 2. Figure 4 shows changes in concentration of segregated sulfur and magnetic induction with final annealing time at 1200℃ in the 3% silicon-iron strips with manganese.

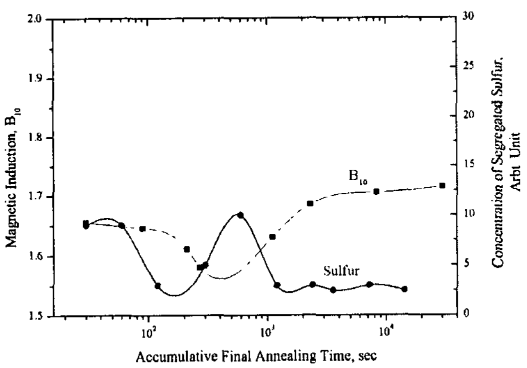

Fig. 4. Changes in concentration of segregated sulfur and magnetic induction with final annealing time at 1200℃ in the 3% silicon-iron strips with manganese.

During final annealing, the change in magnetic induction with final annealing time was similar to that in the case of final reduction of 60%. However, the segregation behavior of sulfur was prominently different from the case in the alloy without manganese. For supporting the results of Fig. 3 and 4, a modified relation of surface-energy-induced selective grain growth rate G(t) given by

$$G(t) = M(t) \cdot \left[\frac{\gamma_G}{r(t)} + \frac{2\Delta\gamma_S}{d} + C(t) \right]$$

where $M(t)$ is the grain boundary mobility, γ_G is the grain boundary free energy, $r(t)$ is the average grain size which means, strictly speaking, the average grain radius excluding the selectively growing grains, d is the sheet thickness. $C(t)$, Zeners term, acts as a negative driving force, which is generally related to inclusions at grain boundaries or the concentration of segregated sulfur which varies with final annealing time and has a strong effect on grain boundary movement. Considering $\Delta\gamma_S / \gamma_G$ = 0.03 [5], the surface energy difference is responsible for selecting the grain which will grow, and is not the main controlling factor in the growth rate.

The larger primary grain size is, generally, favorable for final Goss texture after final annealing.

The size and distribution of MnS particles, which precipitated from the matrix during an intermediate annealing, determines the primary grain size in the case of 3% silicon-iron alloy with manganese, while higher final reduction (e.x., final reduction of 60%) is responsible for the smaller primary grain size. As a result, the lower magnetic induction in the alloy strips with Mn after final annealing can be attributed to the MnS particles which act as an reducer in the primary grain size, although the final reduction of 30% is relatively weak, comparative to the 40%.

4. Conclusions

In the manganese-free 3% silicon-iron alloy strips, higher magnetic induction than 1.9 Tesla was obtained, as the final reduction was reduced. In the manganese-contained 3% silicon-iron alloy strips, much lower magnetic induction was obtained, because the relatively smaller primary grain size was caused by the size and distribution of MnS particles. Evaporation-controlled surface segregation of sulfur was observed in two alloy strips, resulting in a maximum of segregated sulfur.

However, the segregation concentration in the manganese-contained 3% silicon-iron alloy strips was relatively low and the behavior was very different from the other case. This is because the segregation kinetics is governed by the MnS precipitation kinetics as well as the evaporation kinetics.

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