Effects of Anti-phase Boundary on the Iron Loss of Grain Oriented Silicon Steel

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We present a systematic analysis of the iron loss behavior of grain oriented silicon steels containing different Si contents using transmission electron microscopy. When the silicon content changed in the range of 3-6.5 wt%, the iron loss showed a convex profile and the maximum iron loss was observed in 5.2 wt% silicon steel. This maximum iron loss should be ascribed to the formation of antiphase boundaries that acted as pinning centers in the magnetic domain wall.

KEY WORDS: grain oriented silicon steel; iron loss; anti-phase boundary; magnetic domain wall.

1. Introduction

Silicon steel has been widely used in electrical appliances and devices, such as transformers and motor cores, because of its excellent soft magnetic properties and low cost. In recent years, however, further reductions in the magnetic loss in silicon steel have been sought in order to improve efficiency in electrical appliances. It is well known that the magnetic properties of silicon steel are strongly dependent on the strip thickness, silicon concentration, grain size, and crystallographic texture.1,2) Among these factors, the increase in silicon content is one of the most effective ways to reduce eddy current loss, resulting in efficiency improvements in high frequency transformers and motors. Furthermore, the magnetostriction value of the silicon steel is reduced to almost zero when the silicon content reaches 6.5 wt%, which can result in a sharp reduction in hysteresis loss.3–6) Nevertheless, the application of 6.5 wt% silicon steel to transformers and motor cores has been limited because it is too brittle for use in conventional rolling techniques.7,8) The brittleness of high silicon steel stems from the formation of two different ordered phases, B2 (FeSi) and D03 (Fe3Si),3,4,7–9) which are known to alter the magnetic properties of the steel.10,11) However, the specific relationship between the ordered phases and the magnetic properties in high silicon steel is still unclear. One of the key challenges in the use of high silicon steel is, therefore, to understand how ordered phases contribute to the magnetic loss properties.

As previously mentioned, the brittleness of high silicon steel makes it difficult to use conventional rolling techniques in fabrication. To overcome this problem, several methods such as chemical vapor deposition process were suggested.12) The suggested methods require complex and long procedures so a more simplified fabrication method is strongly demanded. In this study, SiO2 textiles were used to control the amount of silicon in thin-gauged grain oriented (GO) silicon steel. Further, the iron loss behaviors of the GO silicon steels with high silicon content were investigated by increasing the silicon content to 6.5 wt%.

2. Experimental Work

Silicon steels (3 wt% Si) containing 58 ppm sulphur were used as starting materials to prepare GO silicon steels, the detailed compositions of which are provided in Table 1. Thin-gauged strips 0.15 mm in thickness were prepared through the hot- and multi-stage cold-rolling processes described in detail in a previous paper.13) Figure 1 shows angular relationship between the <001> crystal direction and the rolling direction in a fabricated 3 wt% silicon steel. As shown in the figure, the deviation angles were less than 10° in almost grains. The angular relationship did not after annealing process. Each annealed strip was inserted between SiO2 textiles and was then heat-treated at 1 200°C under a 6N hydrogen atmosphere in a quartz tube. Silicon content was controlled through the diffusion of silicon generated from SiO2 decomposition. After the annealing process, the strips were air-cooled. The silicon content and impurity levels of the strips were measured using inductively coupled plasma spectrometer.

Table 1. Chemical composition of the 3 wt% silicon steel.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0067</td>
<td>2.97</td>
<td>0.0058</td>
<td>balance</td>
</tr>
</tbody>
</table>
plasma mass spectroscopy (ICP-MS, Spectro, flame modula eop). The microstructures of the strips were analyzed using X-ray diffractometry (XRD, Rigaku, D/MAX-2500) with Cu Kα radiation and transmission electron microscopy (TEM, Jeol, JEM 3010 and 2010). Iron and hysteresis losses were measured using MPG 100D, the hysteresis loops of single sheet testers made by BROCKHAUS Ltd. DC were measured at an excitation induction of 1.0 T, and hysteresis losses were calculated by multiplying the DC hysteresis losses. A classical eddy current loss was calculated using the Benford equation with a resistivity of 46 μΩ and a density of 7.67 g/cm³. Anomalous loss associated with domain wall motion was calculated by subtracting the hysteresis loss and the classical eddy current loss from the total iron loss.

3. Results and Discussion

**Figure 2** shows the changes in silicon content of GO silicon steels as a function of the annealing time. As shown in the figure, the silicon content increased approximately linearly with annealing time. This indicates that silicon was successfully supplied by the decomposition of SiO₂ textiles and the diffusion of decomposed silicon. Moreover, control in silicon content of high silicon steels was successfully achieved simply by annealing with the textiles.

XRD patterns of the 3.0, 5.2, 6.0, and 6.5 wt% silicon steels are shown in **Fig. 3**. The separation of a Fe (110) peak in the 5.2 wt% silicon steel was clearly observed, and the separation angle increased with the silicon content. In order to clarify the results of the additional phases, XRD peaks were curve-fitted using a Lorentzian function. As a result, the Fe (110) peak in the 5.2 wt% silicon steel was shown to be separated into B₂ (110) and Fe (110). In addition, B₂ (110) peaks were also separated into D₀₃ (220), B₂ (110), and Fe (110) in 6.0 and 6.5 wt% silicon steels, and the intensity of the D₀₃ peak increased in the 6.5 wt% silicon steel. In addition, the peak positions of α-Fe shifted to lower
Through the above observations, the formation of the ordered phases resulted in a decrease in the silicon content of α-Fe.

In order to observe the microstructural evolution, selected area diffraction (SAD) patterns and two-beam and Lorentz images of silicon steels were obtained and are shown in Figs. 4 and 5. As shown in Fig. 4, the ordered phases of B2 and D0₃ are clearly observed in the [011] SAD patterns of high silicon steels. According to the diffraction patterns, only a disordered Fe phase existed in 3.0 wt% silicon steel, while the B2 phase was initially formed in 5.2 wt% silicon steel, and the D0₃ phase finally appeared in 6.0 wt% silicon steel. The intensities of the ordered phases generally related to the phase content as the intensity increased with increasing silicon content. These microstructural changes are in accord with previously reported XRD patterns and the binary Fe-Si phase diagram.¹⁵)

As shown in Fig. 5, the ordered phases resulted in the formation of anti-phase domains. Two beam images show that the smoothly curved boundaries have a (a/2)<111> type displacement vector, so called B2-type 1/2<111> anti-phase boundaries (APBs).¹⁶) These B2 anti-phase domains and the APBs were observed in all samples except the 3.0 wt% silicon steel. The size of the anti-phase domains in the 5.2 wt% silicon steel was about 5–20 nm, which then increased to a few hundred nm in the 6.5 wt% silicon steel. Thus, the densities of the APBs decreased with increasing silicon content. Lorentz micrographs show that the sizes of the magnetic domains were much greater than the sizes of the antiphase domains, and magnetic domain walls (white lines in the figure) cut across the APBs.

Figure 6 shows the iron loss in high silicon steels measured at 60 Hz and 1.0 T. In order to clarify the effect of the silicon content on the loss mechanism, the iron loss was separated into three different losses. The maximum and minimum iron losses were found in the 5.2 wt% and 6.5 wt% silicon steels, respectively. The hysteresis loss was peaked in the 5.2 wt% silicon steel, and was drastically decreased in the 6.5 wt% silicon steel. The hysteresis loss was caused by the movement of the magnetic domain wall, which is strongly related to microstructures such as APBs, stress, grain boundaries, and impurities.¹⁷) In particular, the presence of the APBs should affect the magnetic domain wall movement in this material. In general, APBs act as two-dimensional defects which should oppose the displacements of the magnetic domain walls. Thus, pinning of the magnetic domain walls by the APBs is expected to deteriorate the soft magnetic properties of the silicon steels by decreasing permeability and increasing coercive force.¹⁷) Therefore, the abrupt
increase in the hysteresis loss of the 5.2 wt% silicon steel was closely related to the presence of B2 APBs. In addition, the decrease in hysteresis loss with further increase in silicon content may have been caused by a decrease in the B2 APBs density. As expected, the eddy current loss decreased with increasing silicon content due to increased resistivity; however, the anomalous loss did not exhibit a linear dependency on the silicon content. The minimum anomalous loss was observed in the 5.2 wt% silicon steel (0.266 W/kg), and the maximum was 0.329 W/kg in the 6.0 wt% silicon steel. As shown in the figure, the hysteresis loss was the most important factor in the iron loss of high silicon steels, although the anomalous loss fluctuated with silicon content. In particular, the minimum iron loss was obtained in the 6.5 wt% silicon steel, partially due to the low density of APBs. Thus, it is critical to decrease the density of APBs through proper annealing in order to minimize the iron loss in high silicon steel.

4. Conclusions

Microstructures and iron loss behaviors of silicon steel were investigated in the silicon content range of 3 to 6.5 wt%. TEM micrographs showed that B2-type <111> APBs were first observed in the 5.2 wt% silicon steel, and the average size of the antiphase domains increased with increasing silicon content. In addition, Lorentz micrographs showed that magnetic domains cut through these APBs. The hysteresis loss of the silicon steels sharply increased with the presence of APBs. With increasing silicon content, both the area of APBs and the hysteresis loss decreased. These results strongly indicate that APBs act as magnetic domain wall pinning sites. Therefore, control of the sizes of antiphase domains is critical for reducing iron loss in high silicon steels.

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