Magnetic Domain Structures in Electrical Steel Sheets
Studied by Lorentz Microscopy and Electron Holography

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Magnetic domain structures of doubly oriented and non-oriented electrical steel sheets, the former of which has two perpendicular magnetic easy axes in the sheet plane, have been observed by Lorentz microscopy and electron holography. In the demagnetized state, the doubly oriented electrical steel sheet shows straight lines of domain walls with homogeneous distribution of lines of magnetic flux. On the other hand, non-oriented electrical steel shows irregular distribution of domain walls with curved lines of magnetic flux. Through the in situ observation of magnetization process of these electrical steel sheets, it has been found that domain walls in doubly oriented electrical steel sheets move continuously, while domain walls in non-oriented electrical steel sheets move discontinuously. It has been also clarified that the domain walls in non-oriented electrical steel sheets are pinned at the precipitates which are considered to consist of Al2O3, SiO2, MnO and TiO2. These differences in the movement of domain walls are briefly discussed with their magnetic properties.

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1. Introduction

Electrical steel sheets have been widely used for various devices such as magnetic shield, magnetic cores of motors and transformers, etc. The electrical steel sheets are therefore soft magnetic materials, for which high permeability and low magnetic core loss are the most important requirements to be met, and they are generally classified into two groups in terms of magnetic anisotropy. One is the so-called grain-oriented electrical steel sheet that has the (110)(001) or (100)(001) crystallographic texture and is highly anisotropic with one or two magnetic easy axes lying in the sheet plane since the easy axes in Fe–Si lie along the (100) directions. The other is the non-oriented steel sheet that, as the word implies, has almost no texture and is isotropic. In addition, the grain-oriented steel sheet with the (100)(001) texture is often designated “doubly oriented” electrical steel sheet since it has two perpendicular easy axes in the sheet plane. The purpose of this research is to analyze the magnetic domain structures of the thin foils prepared from the doubly oriented and non-oriented electrical steel sheets by Lorentz microscopy and electron holography, in order to understand their magnetic properties and to help improve them.

So far, magnetic domain structures have been studied by many researchers via Kerr microscopy. However, the analysis by Kerr microscopy is limited to the surface region, and the data obtained by this technique are rather macroscopic although the information on the domain structure in bulk materials can be obtained. In order to make clear the more microscopic relation between microstructures and domain structures, transmission electron microscopy (TEM) may be a useful technique. In this study, in addition to the conventional TEM, Lorentz microscopy and electron holography observations are therefore carried out to investigate the detailed domain structure of the electrical steel sheets. Furthermore, for understanding the detailed magnetic properties, in situ observation of domain wall migration is also performed by applying external fields in foil planes.

We studied two types of electrical steel sheet, i.e. doubly oriented electrical steel sheets and non-oriented electrical steel sheets, as aforementioned. The doubly oriented electrical steel sheet has been particularly used to obtain the thin foils having the (100) plane parallel to the foil plane, in which the typical 90° and 180° magnetic domain walls and their movement caused by external magnetic fields should be observed in in-situ experiment. As for the latter non-oriented electrical steel sheet, in which the distribution of domain walls will be more complicated and the presence of small precipitates are expected, our attention has been focused on the effects of the precipitates on the domain wall migration as well as the domain structure.

2. Experimental Procedures

The thin foil specimens were prepared from two kinds of electrical steel sheet, a doubly oriented electrical steel sheet made in a laboratory and a commercial non-oriented electrical steel sheet. The doubly oriented electrical steel sheet had a grain size of about 500 µm and a strong preferred orientation of (100)(001). The core loss $W_{15/50}$ of this material was 0.90 W/kg at 1.5 T and 50 Hz when the sheet thickness was 0.35 mm. The detailed fabrication process of doubly oriented electrical steel sheet has been reported elsewhere. The grains size of the non-oriented electrical steel sheets was about 20 µm. This material contains more sulfur than the first one in order to improve workability by the presence of precipitates of MnS and so on. The sizes of precipitates are generally controlled in order to keep good magnetic properties. The core loss $W_{15/50}$ of this material was 7.53 W/kg at 1.5 T and 50 Hz when the sheet thickness
was 0.5 mm. Thin-foil specimens for TEM observations were prepared by jet electropolishing using an electrolyte consisting of 20 vol% HNO$_3$–80 vol% CH$_3$OH. The surfaces of TEM specimens were parallel to the surface of the steel sheets.

A holographic electron microscope, a JEM-3000F electron microscope which was equipped with a field emission gun, a biprism and a magnetically shielded objective lens$^5$ was used to take Lorentz micrographs and electron holograms by which domain walls and lines of magnetic flux can be observed, respectively. A specially designed TEM specimen holder$^6$ was used in order to apply the magnetic field in the specimen plane in the TEM. In this holder, the magnetic field produced by a coil can be conducted to a specimen through yokes, and the field can be increased up to tens of thousands A/m.

3. Results and Discussion

In Fig. 1(a), a Lorentz microscope image of a doubly oriented electrical steel sheet is presented. Since the image has been observed under the Fresnel mode, domain walls appear to be bright lines or dark bands depending on the focus condition. It is noted that the distribution of domain walls is strongly affected by the specimen shape. Figure 1(b) shows a hologram obtained at the same area as Fig. 1(a). Most of the interference fringes outside the specimen are straight, while the interference fringes inside the specimen, especially at the lower left region deviate largely from straight lines due to the strong magnetic field of the specimen. Figure 1(c) shows a reconstructed phase image obtained from the hologram in Fig. 1(b) through the Fourier transform operation.$^7$ The arrows in the figure indicate the direction of lines of flux. In the reconstructed phase image, the phase change ($\phi_{x,y}$) of incident electrons is represented by the image intensity ($I_{x,y}$) being linear to $\cos \phi$. This phase change is mostly caused when the incident electrons transmit through the magnetic field inside the specimen,$^8$ and, therefore, the direction of observed stripes corresponds to that of magnetic flux inside the specimen.

It is noted that in the lower left part of the specimen, there exists a $90^\circ$ domain wall which well corresponds to the observation of the Lorentz microscopy image in Fig. 1(a). On the other hand, in the vacuum region at the top right of Fig. 1(c), the sharp stray field can be seen.

Figure 2 shows Lorentz microscope images of a doubly oriented electrical steel sheet observed at the different region where the external magnetic field is applied along the direction indicated by an empty arrow in Fig. 2(b). It is noted that domain walls are parallel to the [100] and [010] directions of the electrical steel sheet. In Figs. 2(a)–(c), the electric currents of 0, 15 and 32 mA were applied to the excitation coil of the specimen holder, respectively. These electric currents correspond to the magnetic fields of 0.0, 2.4 and 5.1 kA/m at the specimen position. With the increase of external magnetic field in the film plane, some of the domain walls which are nearly parallel to the horizontal line disappear. It is noteworthy that the movements of domain walls are continuous and smooth, indicating the core loss of doubly oriented electrical steel sheets is quite low. Comparing with the magnetic properties such as coercivity, the magnetic field which can produce the domain wall movement
seems to be high. One of the reasons for the discrepancy is that some of external magnetic flux may be absorbed by the surrounding thick region. It should also be noted that the surface pinning effect may be attributed to the discrepancy.

Figures 3(a) and (b) show in-situ Lorenz microscopic observation of a non-oriented electrical steel sheet with the external magnetic fields of 0.0 and 2.2 kA/m, which were applied from the right to the left by a small electroromagnet in the specimen holder. In both images, domain walls appear to be curved lines showing the sharp difference from those of a doubly oriented electrical steel sheet. Figures 3(c) and (d) show reconstructed phase images obtained at the same area and under the same external magnetic field as in Figs. 3(a) and (b), respectively. From the reconstructed phase images, it is seen that not only the domain walls but also the lines of magnetic flux curve gradually. The feature of fluctuated distribution in lines of magnetic flux and their movement is quite different from those of doubly oriented electrical steel sheets. It is considered that the fluctuated distribution of lines of magnetic flux under the external magnetic field results from the difference in crystallographic orientations and the effect of precipitates. It is also seen that the direction and the density of lines of magnetic flux fluctuate from place to place in Fig. 3(d). It is considered that some of the magnetic flux is out of the film plane due to the external magnetic field applied, resulting in the inhomogeneous density of magnetic flux projected along the incident beam. Through this in situ experiment, the movements of domain walls were found to be discontinuous being different from that of a doubly oriented electrical steel sheet.

Figure 4 shows Lorentz microscope images of a non-oriented electrical steel sheet observed at the different region where the external magnetic field is applied along the direction indicated by an arrow. In Figs. 4(a)–(c), the electric currents (magnetic fields) in the electromagnet on the specimen holder are 0 mA (0.0 kA/m), 27 mA (4.3 kA/m) and 30 mA (4.8 kA/m), respectively. At this area, the domain walls do not move at all with the electric current up to 27 mA (4.3 kA/m). However beyond 27 mA (4.3 kA/m), the domain
walls start to move discontinuously. At 30 mA (4.8 kA/m), the direction of domain walls are parallel to the direction of the external magnetic field as shown in Fig. 4(c). Further, above 30 mA, the domain whose magnetization direction is parallel to the external magnetic field grows discontinuously. The arrow in Fig. 4 indicates a large precipitate inside the specimen, while arrowheads indicate a black band which corresponds to a domain wall passing through this precipitate. The position of the black band moves around the precipitate with the increase of external magnetic field, and thus the domain wall is considered to be pinned at the precipitate. As shown in Fig. 5, where EDS spectrum obtained from a similar precipitate in the specimen is presented, the precipitate is considered to contain Al$_2$O$_3$, SiO$_2$, MnO and TiO$_2$. It is considered that one of the causes for the domain wall pinning is due to the reduction of the magnetostatic energy inside the precipitates.

4. Conclusion

The results obtained by Lorentz microscopy and electron holography on oriented and non-oriented electrical steel sheets are summarized as follows. Doubly oriented electrical steel has straight domain walls. The magnetization direction and domain walls are basically along the axis of easy magnetization (100), and the domain walls move continuously with increase of external magnetic field. On the other hand, non-oriented electrical steel shows irregular distribution of domain walls with curved lines of magnetic flux. The domain wall was found to be pinned at a precipitate of Al–Si–Mn–Ti oxide. Discontinuous movement of domain walls in non-oriented electrical steel sheet is considered to result from the pinning effect caused by the reduction of the magnetostatic energy inside the precipitates.

REFERENCES