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Study on the Surface Magnetic Domain Structure of Thin-Gauged 3% Si-Fe Strips using Scanning Electron Microscopy with Polarization Analysis

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Scanning Electron Microscopy with Polarization Analysis (SEMPA) was used to image the surface magnetic domain structure of the 100 μm thick 3% Si-Fe sheet. The thin-gauged 3% Si-Fe strips with magnetic induction (B_{10}) from 1.98 to 1.57 Tesla were prepared via conventional metallurgical processes including melting, hot- and cold-rolling, intermediate annealing and final annealing. Using SEMPA, it was observed that the B_{10} (1.98 T) Tesla sample was almost composed of 180° stripe domains which are parallel to rolling direction. On the other hand the 3% Si-Fe sheet with B_{10} (1.57 T) Tesla was composed of large 180° stripe domains that are slanted about 30° to the rolling direction and complex magnetic domain structures like tree and zigzag pattern. The 180° stripe domains, which covered a major part of the sample, had (110)<001> Goss texture parallel to the rolling direction. The domain walls between 180° stripe domains were the conventional Bloch type walls. On the other hand, the 90° domains, which covered minor part on edge of the sample, were observed in (200) grains. The domain walls between 90° domains were the Neel type walls. In high magnification, the elliptical singularity at the Neel walls was clearly observed.

1. Introduction

Due to the important soft magnetic properties, 3% Si-Fe sheets have been extensively studied and currently used as core material of large transformers, large rotating machines and pole transformers where energy losses during magnetization are of critical concern. To reduce the energy losses of 3% Si-Fe sheets, the domain modification by the application of uniaxial tensile stress, mechanical scribing and laser scribing has been achieved [1, 2]. Many experimental and theoretical studies have been conducted on the thin-gauged Si-Fe sheets due to their good magnetic properties. However, there are few studies on the domain structure of the thin-gauged Si-Fe sheets. Therefore, it is very important to observe the domain of the Si-Fe sheets precisely to understand mechanism of energy loss.

It is well known that the energy losses are significantly decreased with decreasing thickness of Si-Fe sheets [3]. The (110)[001] texture of thin-gauged Si-Fe sheets are developed through a tertiary recrystallization process [4]. The tertiary recrystallization of the thin-gauged Fe-Si sheets is induced by the difference of a surface energy between (110) grains and the other grains caused by sulfur segregation on surfaces [5, 6].

In this study, 100 μm thick Si-Fe sheets with various magnetic properties were prepared by changing reduction rate during cold rolling, and their surface magnetic domain structure was studied by using Scanning Electron Microscopy with Polarization Analysis (SEMPA) [7]. The magnetic domain was also investigated with Bitter method, and compared with the results observed with SEMPA.

2. Experimental Procedures

A 3% Si-Fe ingot was prepared from electrolytic iron of high grade and extremely purified silicon through vacuum induction melting. Table 1 lists the chemical compositions of the alloy.

The ingot was hot-rolled to 2.5 mm plates after holding at 1200°C for 1 hr. The plates were pre-annealed at 800°C for 4 hr and were reduced to 100 μm through three-step cold rolling with reduction ratios of 80% - 50% - 60% [8]. In each step of the cold rolling, the reduction ratio/pass was changed from 1% to 20% in order to investigate the effect of deformation history on the magnetic property of the final thin-gauged sheets. The total reduction ratio was the same, irrespective of the number of cold rolling pass. Intermediate annealing was performed at 800°C for 30 min,

Table 1. Chemical compositions of the Fe-Si alloy (wt. %)

C	Si	Mn	S	N
0.006	2.92	<0.001	0.006	0.001

and final annealing was given to thin strips at 1200°C for 1 hr. All the intermediate and final annealing treatments were carried out in a vacuum of 6×10^{-6} Torr. Texture and microstructure of the strips were analyzed by using XRD and optical microscopy. Magnetic properties of the strips were measured with a DC loop hysterisisgraph (TRF-5AH 1, Toei Co. Japan).

Scanning Electron Microscopy with Polarization Analysis (SEMPA) was used to image the surface magnetic domain structure of the 100 μm thick 3% Si-Fe sheet in ultra-high vacuum (base pressure $<2.0\times10^{-10}$ Torr) in order to maintain clean surface condition during the measurements. The electron beam at 7.5 keV is scanned across the surface of the sample and secondary electrons which carry the magnetic information are extracted and spin analyzed by the spin-polarimeter. We detected two orthogonal in plane components of the spin polarization simultaneously. All of SEMPA images used for this study are 128×128 square pixels. The dwell time per pixel was 70 ms for a total image acquisition time of 20 min. In this experiment, instrumental asymmetries were eliminated by using an Ag foil attached to the same sample holder.

3. Results and Discussion

Fig. 1 shows changes in magnetic induction, B_{10}(T), of final thin strips with reduction ratio/pass in the cold rolling process. The B_{10}(T) values increased to a maximum value of 1.98 T at the reduction ratio/pass of 10% and then decreased with increasing reduction ratio/pass. These results agree with our previous measurements [9].

Fig. 2 shows the domain images of the sample with B_{10}(1.98 T), observed with SEMPA (b, d) and the Bitter method (a, c). The images in Fig. 2(a), (b) and (c), (d) correspond to the middle and the edge parts of sample (5 mm×100 mm), respectively. In the middle part of the B_{10}(1.98 T) sample (Fig. 2a, b), only 180° stripe domains whose domain walls are parallel to the rolling direction are observed. Whereas the edge parts of the B_{10}(1.98 T) sample (Fig. 2c, d) are composed of 180° and 90° domains. From XRD experiments, it was confirmed that the 180° and 90° domains are on (110) and (200) planes, respectively.

The black or white image corresponds to one component of the measured surface magnetization, the white areas pointing right. It is clearly seen that there are 180° stripe domains in Fig. 2(b) and 90° domains in Fig. 2(d). During surveying the domain wall structure of the B_{10}(1.98 T) sample, we found very interesting domain wall structures

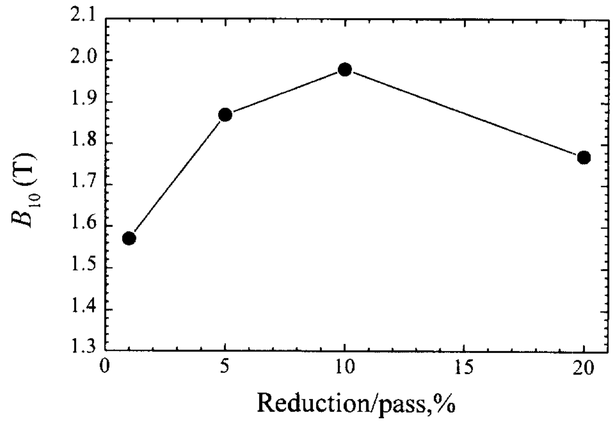

Fig. 1. A changes in B_{10}(T) with reduction ratio/pass of thin-gauged 3% Si-Fe strips finally annealed at 1200°C for 1 hr.

at parallelogramic area in Fig. 2(d).

Fig. 3 shows the magnetization angle of the domains at parallelogramic area in Fig. 2(d), derived from the two measured orthogonal in-plane components of magnetization. From Fig. 3, it is observed that the Neel walls at the place where the magnetization is reversed by about 90° across the straight edges of walls, and the Bloch walls at the place where the magnetization is reversed by about 180°. Also the elliptical singularity at the triple junction of the domains were clearly observed. This is the first observation of the 90° Neel walls and the elliptical singularity in thin-gauged 3% Si-Fe sheets.

Fig. 4 shows the domain images of the sample with B_{10}(1.57 T), observed with SEMPA (b, d) and the Bitter method (a, c). There are large 180° domains, whose domain walls are slanted about 30° to the rolling direction Fig. 4(b), and 90° domains in the middle part Fig. 4(a). It was observed that the domain structures near the edge part of the sample was more complicated than around the middle part of the same sample.

Higher magnification images (×1000) of the areas marked with A and B in Fig. 4(d) are shown in Fig. 5(a) and (b), respectively. From area A, small tree pattern (Fig. 5(a)) was observed while zigzag domain boundaries (Fig. 5(b)) were seen in area B. The formation of the tree pattern has been explained as a slight inclination of the surface to (100) planes and the branches were oriented so that they pointed in the downhill direction [10]. When the inclination between surface and crystal planes becomes relatively large, the branches of the tree pattern become thick and close together, until finally the outlines of the end portions of the branches remain [10]. Therefore, the zigzag domain structure shown in Fig. 5(b) can be attributed to a relatively larger inclination from the (100) planes.

The domain structure between the middle part and the edge part of the thin gauged Si-Fe sheets is quite different as can be seen in Fig. 2 and Fig. 4. This may be attributed to different strain between these 2 parts. For sample

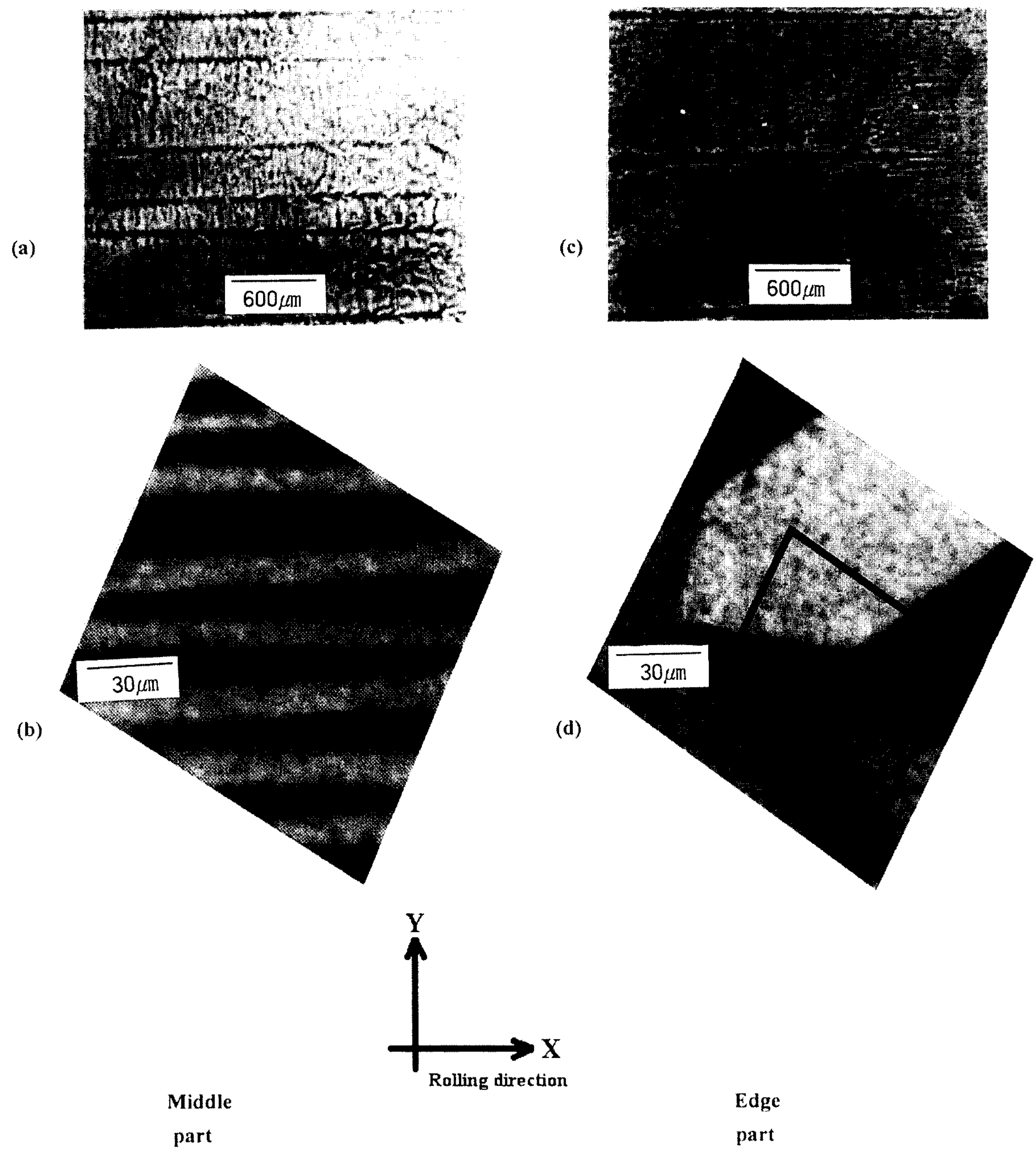

Fig. 2. Bitter (a), (c) and SEMPA (b), (d) domain images of B_{10}(1.98 T) sample.

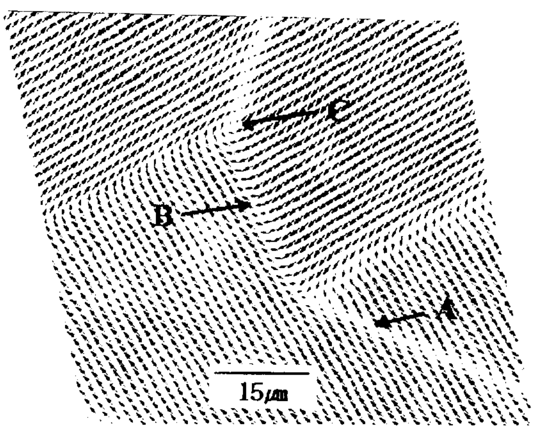

Fig. 3. Magnetization angle of the domains at parallelogramic area in Fig. 3(b). (A: Bloch wall, B: Neel wall, C: Elliptical singularity)

preparation, we cut the samples to strip shape (5×100 mm^2) by cutter and the shear strain might be stored at the edge parts heavily. This may lead the different domain structure between the middle part and the edge part of the thin gauged Si-Fe sheets.

The difference in the domain structure between the sample of B_{10}(1.98 T) and B_{10}(1.57 T) might be the main reason for the difference in magnetic properties shown in Fig. 1.

4. Conclusions

The B_{10}(T) value of 100 μm thick 3% Si-Fe sheets increased with reduction ratio/pass during the cold rolling

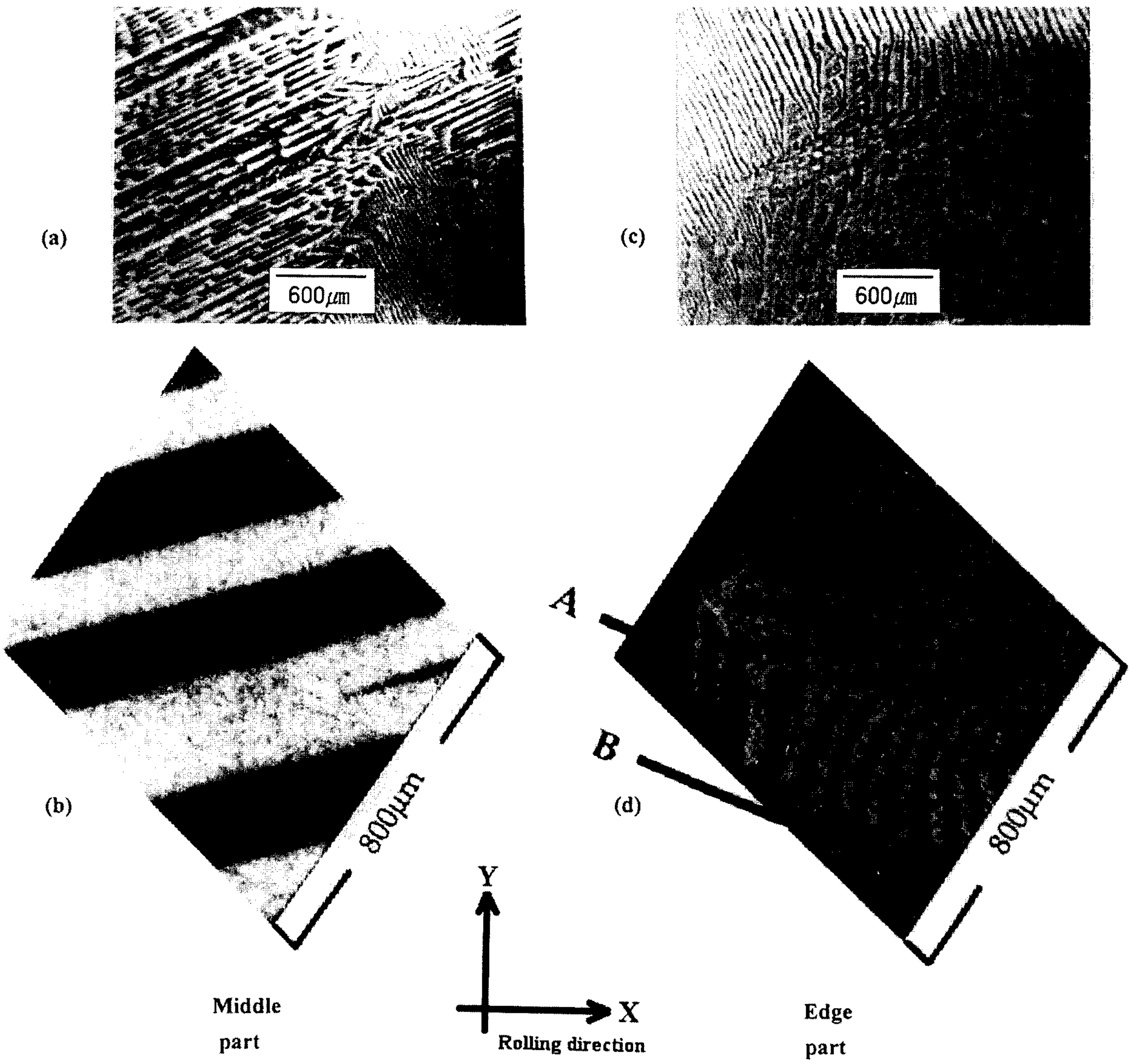

Fig. 4. Bitter (a), (c) and SEMPA (b), (d) domain images of B_{10}(1.57 T) sample.

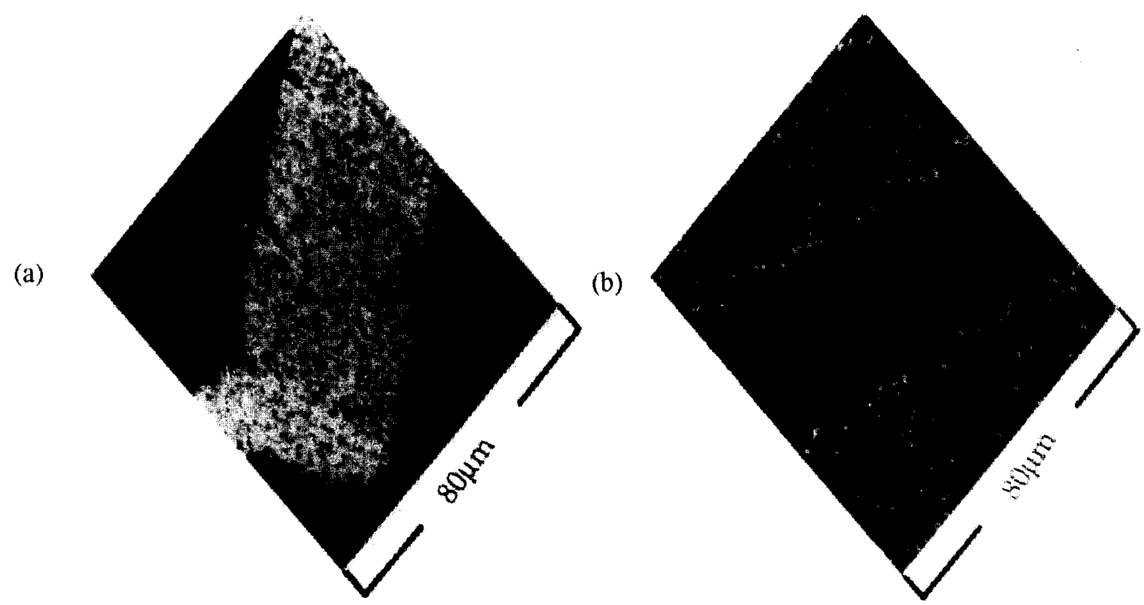

Fig. 5. (a) The θ images of the small tree type domain structure, area A in Fig. 4(a). (b) The θ images of the zigzag domain structure, area B in Fig. 4(a).

process and reached the maximum value of 1.98 Tesla at the reduction ratio/pass of 10%, where after it decreased with the reduction ratio. From observation of magnetic domains by Bitter method, it was found that the B_{10}(1.98 T) sample was composed of 2 kinds of domains: 180° stripe domains and 90° domains. The 180° stripe domains whose domain walls are parallel to the rolling direction, covered a major part of the sample. On the other hand, the 90° domains were observed in (200) grains at the edge of the sample. From analysis by using SEMPA, it was found that the Neel walls occur at the place where the magnetization is reversed by about 90° across the straight edges of walls, and the Bloch walls at the place where the magnetization is reversed by about 180°. Also the elliptical singularity at the triple junction of the domains was clearly observed. For the Si-Fe sheets with B_{10}(1.57 T), large 180° stripe domains are slanted about 30° from the rolling direction in the middle part. Tree-type and zigzag pattern domain structures were observed near the edge of the B_{10}(1.57 T) sample.

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References

[1] J. W. Shilling and G. L. Houze, IEEE Trans. **Mag-10**, 195 (1974).
[2] A. L. Von Holle and J. W. Schoen, J. Appl. Phys. **55**, 2124 (1984).
[3] E. T. Stephenson, J. Appl. Phys. **55**, 2142 (1984).
[4] J. L. Walter and C. G. Dunn, Trans. AIME **215**, 465 (1959).
[5] N. H. Heo, K. H. Chai and J. G. Na, J. Appl. Phys. **83**, 1 (1998).
[6] N. H. Heo, K. H. Chai, J. G. Na and J. S. Woo, Scripta Mater. (in press)
[7] M. R. Scheinfein, J. Unguris, M. H. Kelly, D. T. Pierce and R. J. Cellota, Rev. Sci. Instrum. **61**, 2051 (1990).
[8] N. H. Heo, Y. J. Kim and J. G. Na, Scripta Mater. (in press)
[9] Y. Lee, A. R. Koymen, N. H. Heo, J. G. Na and J. S. Woo, Scripta Mater. (in press)
[10] H. J. Williams, R. M. Bozoth and W. Shockly, Phy. Rev. **75**, 155 (1994).