



## Research articles

## Effect of Al content on magnetic domains of {100} grains in electrical steels

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## ABSTRACT

Magnetic domains of {100} grains in electrical steels with  $1.85 \leq \text{Al} \leq 6.54$  wt% were observed by magneto-optic Kerr microscopy. The characteristics and motion of magnetic domains were correlated with hysteresis loss, anomalous loss and permeability. As Al contents increased, domain wall energy decreased, so the magnetic domain size of {100} grains decreased. In steel with Al 6.54 wt%,  $90^\circ$  domain walls appeared, so the complexity of domain structures increased. Addition of Al caused increase in hysteresis loss. Anomalous loss decreased until Al 4.68 wt%, then saturated, despite high resistivity and small domain size. External magnetizing force  $\vec{H}$  required to induce maximum permeability  $\mu_{\text{max}}$  increased drastically at Al 6.54 wt%. These changes of magnetic properties may be caused by pinning of  $90^\circ$  walls during magnetization. As domain size decreased to form closure domains, the complexity of reorganization of magnetic domains increased, and they were interrupted by pinning  $90^\circ$  walls.

## 1. Introduction

Non-oriented (NO) electrical steel is a soft magnetic material that is widely used in rotating machineries that require high magnetic flux densities and low core losses. Core loss in NO electrical steel can be separated into hysteresis loss, classical eddy-current loss and anomalous loss. Magnetic properties in electrical steels, especially hysteresis loss and anomalous loss are affected by the structure and behavior of magnetic domains under external fields [1–5]. Earlier work has focused on investigations of single crystals of iron with the {100} plane parallel to the surface [6–8]. However, NO steel requires randomly oriented crystallographic texture, {100} <0uv>, and the optimal average grain size for minimization of core losses [9,10]. The domains are severely affected by the neighboring domains, so to develop NO steel that has low core loss, the magnetic domain structure of {100} <0uv> in polycrystalline electrical steel should be investigated.

The addition of Si can decrease eddy-current loss in NO steel. However, Si degrades the cold workability of high silicon steels, so the Si content is limited to 3.5 wt%. To reduce eddy current loss further, Al can be added to Si steels up to [Si + Al] contents of 4.5 wt% [11]. Many researchers have investigated magnetic properties of Al-added electrical steels [11–15]. In Fe–Al binary electrical steels, Al content affects the magnetic properties such as core loss, texture, magnetic flux density, permeability and magnetic domain structure [16]. However, the correlation between the magnetic domains and the core losses of Fe–Al binary electrical steels has not been studied in detail.

In this work, the structure and dynamic motion of {100} magnetic domains of Fe–Al NO steels were observed under external fields. The correlation between the magnetic domain structure of {100} and core losses was studied.

## 2. Experimental procedures

## 2.1. Sample preparation

Fe–Al binary electrical steels containing  $0.53 \leq \text{Al} \leq 9.65$  wt% were prepared (Table 1) by vacuum melting. The as-cast ingots were reheated at  $1100^\circ\text{C}$  for 2 h for homogenization, then hot-rolled to a thickness of 2.0 mm. After hot rolling, they were annealed at  $1000^\circ\text{C}$  for 5 min to remove elongated grains. The oxidized layers of annealed samples were removed by 35 wt% HCl etchant. Then the samples were cold-rolled to a thickness of 0.35 mm. The cold-rolled samples were annealed at  $1000^\circ\text{C}$  for 1 min, 3 min, 5 min, 10 min and 60 min in  $\text{H}_2$  atmosphere to control the average grain size. The cooling rate was about  $55^\circ\text{C/s}$ . For observation of magnetic domains without residual stress, samples were polished with 20 nm colloidal silica for 2 h, then annealed at  $750^\circ\text{C}$  for 2 h in Ar atmosphere.

## 2.2. Method of analysis

{100} magnetic domains in steels with  $1.85 \leq \text{Al} \leq 6.54$  wt% were investigated by magneto-optic Kerr microscopy using a ZEISS Axio

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**Table 1**  
Chemical composition of steels.

Al (wt%)	Si (wt%)	C (ppm)	S (ppm)	N (ppm)	P (ppm)	Ti (ppm)	Fe (wt%)
0.53	0.0028	280	15	140	< 10	< 10	bal.
1.85	0.0043	220	16	11	< 10	< 10	bal.
2.64	0.014	20	10	2	< 10	< 10	bal.
4.68	0.011	44	9	1	< 10	< 10	bal.
6.54	0.0069	17	9	22	< 10	< 10	bal.
9.65	0.0040	20	9	1	< 10	< 10	bal.

Imager D1m optical microscope. The magnetization process of {100} magnetic domains was observed under an AC field at a frequency of 0.1 Hz. A peak external field of 1080 Oe was exerted to the specimens. The crystallographic orientation of grains and their grain sizes were measured by Electron Back-scattering Diffraction (EBSD). Supplementary domains, which hinder domain wall motion, can be created by a small deviation angle from the exact {100} crystallographic plane [7,17]. Thus, to observe magnetic domains, only {100} grains within 2° were selected.

Core losses were measured using a Brockhaus Instrument Model MPG 100, coil system 50 mm × 50 mm single-strip tester [16]. Core loss was measured from 20 Hz to 200 Hz in magnetic flux density of 1.5 T. Core loss and magnetic flux density were calculated by averaging the values of the longitudinal and transverse directions. Hysteresis loss and anomalous loss were obtained using the loss separation method. After excluding theoretical eddy-current loss [18] from the total loss, the other losses were separated by regression vs. frequency. Textures of the recrystallized sheets were measured using a Bruker D8-discover X-ray diffractometer. Orientation distribution functions (ODFs) were obtained from pole figures, then surface area fractions of {110}, {200}, {111} planes on the recrystallized samples were calculated using the texture coefficient [19]. The effect of texture on loss or magnetic induction was considered by using the A-deviation angle parameter [20].

### 3. Results and discussion

#### 3.1. Magnetic domain size and structure of {100} grains in demagnetized Fe-Al

The effect of Al contents on demagnetized {100} magnetic domains were investigated by Kerr-microscopy (Fig. 1). {100} grains within 2° are shown as the grains enclosed by lines. Low angle grain boundaries whose misorientation is < 15° were investigated to consider magnetostatic energy generated by the neighboring grains. At all Al contents, the {100} grains have low angle grain boundaries of similar length and misorientation angle. The domain size of Fe-Al decreased as Al contents increased (Fig. 2). This trend can be explained by the change of magnetic anisotropic constant  $K_1$  and magnetostriction  $\lambda_{100}$ . Magnetic anisotropy constant  $K_1$  is an intrinsic parameter due to the directional dependence of magnetic properties in a material. Magnetostriction  $\lambda_{100}$  is the fractional change of length in a [100] direction when a single crystal is magnetized to saturation in a [100] direction. At Al ≤ 4.68 wt %, magnetic domain structure tended to have a simple structure with one or two 180° domain walls. In this case, reduced domain size is mainly attributed to decrease in domain wall energy as a result of the reduction of  $K_1$ . In the Fe-rich region of the Fe-Al binary alloy,  $K_1$  decreases as Al contents increases [21]. The magnetic domain wall energy is proportional to  $\sqrt{K_1}$ , so domain wall energy decreases as Al contents increases. To reduce magnetostatic energy, domains become subdivided, so domain wall area increases. In Fe-Si non-electrical steels,  $K_1$  and magnetic domain wall energy may be the factors that predominantly control domain size [22]. In Fe with Al 6.54 wt%, the complexity of the closure domain structure increased, and 90° domains formed. At Al > 4.68 wt%,  $K_1$  and the domain wall energy decrease

further. With this reduced wall energy, the number of created domain walls increases and the closure domain structure forms. Therefore, magnetostatic energy decreases dramatically. Once the closure domain structure forms, 90° domains subdivide due to the high magnetostriuctive self energy of Fe with Al 6.54 wt% [23]. Magnetostrictive self energy is proportional to the domain volume of 90° domains [24], so a certain domain size is determined by energy balance between magnetostrictive self energy and domain wall energy. The abrupt decrease in domain size at Al 6.54 wt% (Fig. 2) demonstrates a further decrease in domain size due to high magnetostriction  $\lambda_{100}$ .

#### 3.2. {100} magnetic domain wall motion and magnetic properties in Fe-Al NO steels

Al contents affects domain wall motion and magnetic properties of Fe-Al NO steels when average grain size and crystallographic texture are controlled. Magnetic domain wall motion in {100} grains (Figs. 3–5) was observed by Kerr microscopy under sinusoidal AC field at frequency of 0.1 Hz. The average grain size of the final sheets with 0.53 ≤ Al ≤ 9.65 wt% was measured (Fig. 6) [16]. The final sheets with 25 μm and 50 μm average grain size were chosen for texture analysis (Fig. 7) [16] and magnetic property measurement (Figs. 8 and 10) [16]. The texture was quantified by A-deviation angle parameter (Fig. 7 (b)), proposed by L. Kestens [20]. The difference of A-deviation angle parameters ranging from 36.3 to 38.2 will not change magnetic properties significantly.

##### 3.2.1. Hysteresis loss

Hysteresis loss increases with increasing Al contents at the same grain size (Fig. 8) [16]. Hysteresis loss results from energy dissipation during discontinuous magnetization [25]. This loss is closely related to the rearrangement of magnetic domains. Therefore, to understand hysteresis loss behavior, domain wall motion from demagnetized state to the magnetically saturated state (Figs. 3–5) is significant because the magnetization process strongly depends on the complexity of magnetic domain structure.

The tendency of hysteresis loss in Fe-Al is mainly due to pinning effect by 90° walls. Considering the non-uniform 180° domain wall motion under external fields, the velocity  $v_i$  of the  $i$ th domain wall can be represented as [26]:

$$v_i(t) = \bar{v}(t) + \Delta\bar{v}_i(t) + \Delta v_i(t), \quad (1)$$

where  $\bar{v}(t)$  is the average domain wall velocity;  $\Delta\bar{v}_i(t)$  is the difference between  $\bar{v}(t)$  and  $v_i(t)$  due to a difference in the peak displacement of domain walls; and  $\Delta v_i(t)$  is the remaining difference between  $\bar{v}(t)$  and  $v_i(t)$  due to brief pinning effects. Based on this assumption, the average power loss  $P_V$  per unit volume was proposed when 180° walls moves from time 0 to time  $T$  [26]:

$$P_V = 1.6P_{cl} \left( \frac{2L}{d} \right) \left[ 1 + \sum_{i=1}^N \left( \frac{\Delta\bar{v}_i(t)}{\bar{v}(t)} \right)^2 \right] + \frac{\beta_{180}}{wT} \sum_{i=1}^N \int_0^T \Delta v_i^2(t) dt \quad (2)$$

where  $N$  is the number of domain walls;  $P_{cl}$  is classical eddy current loss;  $2L$  is the domain width;  $w$  is the width of the specimen;  $d$  is the thickness of the specimen; and  $\beta_{180}$  is the viscous damping constant of 180° walls. Eq. (2) does not consider wall bowing or wall nucleation. The first term implies eddy current loss including anomalous loss which is discussed in the next section. The second term implies hysteresis loss due to pinning effects by 90° walls or precipitates. Domain wall velocity is closely related to Barkhausen noise [27], which is in turn closely related to hysteresis loss. In steel with Al 6.54 wt%, the magnetization process was retarded by pinning of 90° walls (Fig. 5); motion of domain walls was pinned by 90° walls from 2/16 s to 8/16 s after the external field was applied. This increases the difference in the velocities of domain walls  $\Delta v_i(t)$ . However, the domain wall motion of steel with Al 1.85 wt% (Fig. 3) and 4.68 wt% (Fig. 4) were free of pinning by the 90°

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