Effect of Phosphorus on the Magnetic Losses of Non-oriented 2% Si Steel

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(Received on November 4, 2011; accepted on January 10, 2012)

The effect of P, ranging from 0.0 to 0.10 mass%, on the core losses of electrical steels containing 2.0 mass% silicon has been investigated. The core losses were separated into hysteresis losses, classical eddy current losses and anomalous losses. The hysteresis losses were found to decrease with increasing P content due to a larger grain size brought about by P additions. The anomalous losses were proportional to the square root of the grain size, and decreased with increasing volume fraction of grains with a ND || <111> orientation. The lowest anomalous losses were obtained at 0.05 mass% P content.

KEY WORDS: non-oriented electrical steel; magnetic losses; magnetic domains.

1. Introduction

Non-oriented electrical (NO) steel is used to lower the energy loss during the operation of rotational machines such as AC motors.

The most important magnetic properties of NO steel are the core losses. The core losses, P_{Total} (in W/Kg) can be separated into three components, the classical eddy current losses, P_E , the anomalous loss, P_A , and the hysteresis losses P_H :

$$P_{Total} = P_E + P_A + P_H$$

P_H is equal to the area of the quasi-static B-H loop. Hysteresis losses are strongly influenced by the steel microstructure in particular the grain size, distribution of precipitates and the crystallographic texture.

Assuming a sample with a sheet geometry, a sinusoidal induction and full flux penetration conditions, the eddy losses can be written as¹⁾

$$P_E = 0.1645 \frac{t^2 \cdot f^2 \cdot B_m^2}{\rho \cdot \delta}, \text{ W / Kg}$$

Where B_m is the maximum induction, t is the thickness of the sheet, f is the frequency, ρ is the resistivity and δ is the density of the material.

P_E is reduced by an increase of the electrical resistivity. This is usually achieved by the addition of Si. The addition of P also results in an increase of the electrical resistivity but this option has received less attention.

The anomalous losses, P_A are related to the domain wall motion and they are affected by microstructural features such as the grain size and the crystallographic texture.^{2,3)} Quantitative relations for the influence of the microstructure on P_A are not yet available.

The dominant loss component depends on the working

frequency. Hysteresis losses are proportional to the frequency, f . The classical eddy current losses and the anomalous losses are proportional to f^2 and $f^{1.5}$, respectively.²⁾ Whereas both Si and P additions are known to reduce the eddy current losses by increasing the resistivity, the precise effect of P on the microstructure of NO-steel and its influence on the magnetic properties, are not well documented. In the present work, the effect of P additions on the magnetic properties of NO electrical steel was therefore evaluated in depth.

2. Experiments

Steels containing 2 mass% Si and two different P contents (0.05 and 0.10 mass%) were prepared by vacuum induction melting (**Table 1**). The C and N contents were controlled to less than 20 ppm. The vacuum melted ingots were hot rolled to a thickness of 2.5 mm and cold rolled to a thickness of 0.35 mm, prior to annealing at 900°C in Ar for 1, 5, 10, 30, 150 and 750 minutes. The materials preparation procedure is illustrated schematically in **Fig. 1**.

The magnetic properties measurements were carried out on recrystallization annealed sheets 50 × 50 mm² in size in a single sheet tester in both the rolling direction (RD) and in the transverse direction (TD). The AC magnetic properties were measured at a frequency of 50 Hz and 400 Hz on a Brockhaus MPG100. The electrical resistivity of the samples was measured on a Quantum Design-Physical Property

Table 1. Chemical composition of the samples used in the present study. The concentrations are in mass-%.

Steel	P	Si	C	Mn	Al
0% P	0.000	2.09	<0.002	–	<0.002
0.05% P	0.050	2.01	<0.002	0.0050	<0.002
0.1% P	0.100	2.00	<0.002	0.0120	<0.002

Fig. 1. Schematic of the thermo-mechanical cycle used in the present study. The final Non-Oriented steel strip thickness was 0.35 mm.

Measurement System (PPMS).

The microstructure and crystallographic texture were measured by means of an Electron Backscattered Diffraction (EBSD) system mounted on a ZEISS Ultra 55 Scanning Electron Microscope (SEM). The measured area, $5 \times 5 \text{ mm}^2$, was chosen large enough to have statistically meaningful data for approximately 2 000 grains. The textures of samples were represented by means of the orientation distribution function (ODF) in Euler space.

Transmission Electron Microscopy (TEM) specimens were prepared by twin-jet electro-polishing at 40 V in a solution of 5% HClO_4 +95% CH_3COOH at 10 °C. Final specimen perforation by Ar-ion milling was done to avoid the dissolution of the precipitates by the etchant during twin-jet electro-polishing. The Lorentz microscopy method was used in a JEOL 2010 TEM to observe the interaction between magnetic domain boundaries and precipitates. A Bruker D8 X-ray diffraction (XRD) equipped with a Co-target was used to identify the precipitates contained in the microstructure.

Magnetic domain observation was done by Kerr microscopy. Orientation Imaging Microscopy (OIM) was used to identify grains having an exact $\langle 100 \rangle \parallel \text{ND}$ orientation and the magnetic domain structure of the selected grains was subsequently observed on a Zeiss Kerr microscope in the demagnetized state and in the presence of an external field.

3. Results

3.1. Grain Growth and Crystallographic Texture Evolution during Annealing

The magnetic properties of NO steel are dependent on grain size⁴⁾ because the grain boundaries immobilize moving magnetic domain walls. As a result, both the size of the magnetic domains and the energy dissipation by domain wall motion are determined by the grain size. The average grain size, d , of the NO-steels used in the present study was measured by EBSD and the results are shown in Fig. 2.

Burke and Turnbull⁵⁾ derived the kinetics of grain growth on the assumption that the driving pressure on a boundary is solely due to its curvature. According to their analysis, the grain growth should have a $t^{1/2}$ dependence where t , is the annealing time. Hu⁶⁾ reported that the grain growth kinetics for pure iron had a $t^{1/2.5}$ dependence. In the Fig. 2, the time-dependence of the grain size is shown in a $\ln(d)$ - $\ln(t)$ graph.

Fig. 2. Annealing time dependence of the average grain size, d , at 900 °C. The grain size measurements is based on EBSD data.

The recrystallization of the P-free steel at 900 °C started earlier than for the P-added steels. The recrystallization of the 0.05% P steel required an annealing time in excess of 5 min. The 0.10% P steel did not recrystallize until the annealing time exceeded 10 min. However once a recrystallized microstructure was obtained, the grain sizes grew at a larger rate in the 0.10% P and 0.05% P steels. The average grain size of the 0.10% P steel was about 180 μm after annealing for 30 min. In contrast, the average grain size of the P-free steel was approximately 90 μm . It is worth mentioning that the grain growth exponents of the P-free and 0.10% P steels was 5 to 2.5, respectively. The grain size of the 0.05% P steel annealed at 900 °C for 750 min was about 320 μm while the average grain size of P-free steel was only about 140 μm . These results imply a pronounced influence of P on the mobility of grain boundaries.

Solute may influence recovery by pinning dislocations.⁷⁾ When a solute strongly impedes the dislocation movement, recovery can be delayed by their interaction with dislocations. This might be the main reason for the delay of the recrystallization in steel with 0.05% P and 0.10% P additions. Also, it is known that grain boundary migration becomes faster when the boundary mobility on the driving force increases.⁷⁾ The grain boundary mobility decreases by solute segregation, which causes the solute drag effect, and the precipitates, which cause a boundary pinning effect. A decrease of grain growth rate was therefore expected in steels with P additions rather than the observed increase in grain growth rate. The effectiveness of the solute drag effect is affected by an orientation dependence of the solute segregation to the boundary rather than an intrinsic structure dependence of grain boundary mobility. The grain boundary curvature is a main source of the driving force of grain boundary migration. The P content may play a role sustaining the grain boundary curvature by preventing grain boundaries from becoming wavy during the process of grain growth. The effect of P content on recrystallization and grain growth can be summarized as follows: the addition of P content results in a longer recrystallization start time but once the recrystallization is initiated, the grain growth rate is accelerated by the presence of P.

There have been efforts to evaluate the effect of the crystallographic texture on the core losses of NO steels⁸⁻¹⁰⁾ by

Fig. 3. Crystallographic texture components in an ODF $\phi_2=45^\circ$ section with their crystal directions along the external field H when the sample is aligned along (a) RD and (b) TD.

using a single orientation-related parameter. The ‘Texture factor’ is such a parameter. It is the ratio of the volume fraction of ND $\parallel$ $\langle 111 \rangle$ oriented grains and the volume fraction of ND $\parallel$ $\langle 100 \rangle$ oriented grains. **Figure 3** show the location of the main crystallographic texture components for ferritic steels in a $\phi_2=45^\circ$ ODF section. The hard magnetization axis for Fe and ferritic Fe alloys is the $\langle 111 \rangle$ axis. This axis cannot be parallel to an H field applied parallel to the RD when the major crystallographic texture components belong to the α -fiber and γ -fiber as shown in Fig. 3. The $\langle 111 \rangle$ axis can be however parallel to an applied H field along the TD direction for grains with a $\{112\}\langle 110 \rangle$ orientation. As a consequence, the effect of H $\parallel$ $\langle 111 \rangle$ orientated grains will never be high because ND $\parallel$ $\langle 111 \rangle$ oriented grains will never have a $\langle 111 \rangle$ axis parallel to the external field when it is applied parallel to the sheet plane. This orientation can however be regarded as a measure of number of grains with an orientation away from the easy axis of magnetization. The volume fraction of the ND $\parallel$ $\langle 100 \rangle$ oriented grains was used to quantify the volume fraction of grains with their axis of easy magnetization in the sheet plane. A low value for the texture factor implies that the volume fraction of ND $\parallel$ $\langle 100 \rangle$ oriented grains is larger than the volume fraction of ND $\parallel$ $\langle 111 \rangle$ oriented grains. A higher texture factor has been shown to be related to higher hysteresis losses.⁸⁾ Moses and Pluta¹⁰⁾ observed a decrease of the anomalous losses when the intensity of the GOSS orientation increased. The GOSS orientation has a $\langle 100 \rangle$ axis parallel to the RD. When the external field is parallel to the RD, the magnetization is expected to be similar to that of grains with a $\{100\}\langle 100 \rangle$ orientation. The texture factor values obtained for the three steels after various annealing times are shown in **Fig. 4**. The grains with a $\langle 100 \rangle \parallel$ ND orientation and a $\langle 111 \rangle \parallel$ ND orientation were defined as grains with an orientation within 10° of the exact crystallographic texture component orientation. Only fully recrystallized samples, *i.e.* samples which were annealed for more than 10 min, were selected for the texture factor measurements. The value of the texture factor decreased with increasing annealing time except for the 0.05% P steel. The texture factor of the 0.05% P steel increased from 4.8 to 8.6 when the annealing time was increased from 10 min to 750 min. A low texture factor 3.2 was measured for the 0.05% P steel annealed for 30 min. The values of the texture factor of the 0.10% P steel were always low, regardless of the annealing time. This is due to

Fig. 4. Annealing time dependence of the Texture factor (ND $\parallel$ $\langle 111 \rangle$ / ND $\parallel$ $\langle 100 \rangle$) as a function of P content.

the fact that the intensity of the ND $\parallel$ $\langle 111 \rangle$ orientation did not change during annealing.

3.2. Magnetic Property Measurements

The important application properties for non-oriented electrical steels are the core losses at 50 Hz and 1.5 T (W15/50) and, the core losses at 400 Hz and 1.0 T (W10/400). W10/400 core losses have not yet been extensively studied. Recently, high frequency core losses, W10/400, have become a major research subject as they are relevant for electric vehicle applications. The core loss properties are influenced by parameters such as crystallographic texture, grain size, electrical resistivity, sheet thickness, steel cleanliness, domain size and stress state. The relations between the main control parameters and the magnetic loss components, P_H , P_E and P_A , are listed in **Table 2**. The crystallographic texture improvement and the optimization of the grain size have been the main microstructural parameters studied in relation to the reduction of the core losses of NO steels in the present work.

These two parameters, however, change simultaneously during recrystallization annealing and grain growth, it is not possible to keep the texture or the grain size fixed in a controlled manner when analyzing the effect of the P content on the core losses. The effect of the P content on the core losses was therefore studied as a function of the annealing time at 900°C . The P content dependence of the core losses, W15/50 and W10/400, is shown in **Fig. 5**. The value of W15/50 increased with increasing P content in the samples annealed

for 10 min. The value of W15/50, however, decreased with increasing P content in the samples annealed for 30 min. W10/400 was independent of the annealing time. The minimum core losses, 23.4 W/Kg, were obtained from the 0.05% P sample annealed for 30 min.

The electrical resistivity and the calculated classical eddy current loss values are shown in Fig. 6. The electrical resistivity increases with increasing P content. The addition of 1 wt% P increases the electrical resistivity by $49 \mu\Omega\cdot\text{cm}$. The classical eddy current loss, P_E , was calculated to be 0.44 W/kg. The parameter values used to calculate P_E were $d=0.35 \text{ mm}$, $f=50 \text{ Hz}$, $B_m=1.5 \text{ T}$, $r=34 \mu\Omega\cdot\text{cm}$ and

Table 2. Characteristics properties of the magnetic losses in NO electrical steels.

	Origin	formula	Control parameter
P_H	Localized eddy currents at Barkhausen jumps. Rearrangement of domain patterns.	$P_H / f = \oint H \cdot dB$	Crystallographic texture Grain size Purification
P_E	Electromotive forces (EMFs) are generated in accordance with Faraday's law when a steel sheet is placed in an alternating magnetic field.	$P_E / f = 0.1645 \frac{t^2 \cdot f \cdot B_m^2}{\rho \cdot \delta}$	Sheet thickness Electrical resistivity
P_A	Irregular magnetization process. Micro-eddy currents are concentrated around moving domain walls.	$P_A = P_{\text{Total}} - P_H - P_E$	Crystallographic texture Grain size Purification Domain control

Fig. 5. W15/50 and W10/400 core losses as a function of the P content.

$D=7.7 \text{ g/cm}^3$ for the P-free steel. As the electrical resistivity increases with increasing P content, the classical eddy current loss value, W15/50 was 0.38 W/kg for the 0.1 mass% P steel.

The core losses were separated into three parts: classical eddy current losses (P_E), anomalous losses (P_A) and hysteresis losses (P_H). The sum of P_E and P_A is usually referred to as the apparent eddy current loss,¹¹⁾ and it can be separated from P_H by the analysis of the total loss at different frequencies. P_H increases linearly with the frequency f , while the apparent eddy current loss has a f^2 -dependence. P_H and P_A , calculated by subtracting P_E from the apparent eddy current losses, are shown in Fig. 7.

Figure 7(a) shows that the hysteresis losses increase with an increasing P content after a 10 min annealing. The lowest hysteresis loss value was 1.786 W/Kg, for the 0.10% P steel annealed for 30 min (Fig. 7(b)).

The anomalous losses increased with increasing P content and with longer annealing times. In the case of a low B field (W10/50 and W10/400), this tendency was weaker than in the case of a high B field (W15/50 and W15/400), as shown in Figs. 7(f), 7(g) and 7(h). The 0.05% P steel had the lowest anomalous losses at W15/400 as shown in Figs. 7(e)–7(g). This was due to a decrease of the anomalous losses in the 0.05% P steel.

The DC hysteresis curves and B-H magnetization curves

Fig. 6. Electrical resistivity and classical eddy current losses as a function of the P content.

Fig. 7. Hysteresis and anomalous losses as a function of the P content: (a) 10 min, (b) 30 min, (c) 150 min and (d) 750 min.

at W15/400 are shown in Fig. 8. Samples having a similar grain size were selected, to minimize grain size effects in the measurements. The increase of the area in B-H curves in the W15/400 condition is caused by additional classical eddy current losses and anomalous losses. The amount of anomalous losses can therefore be regarded as the area difference

Fig. 8. B-H magnetization curves of 2% Si steel with 0%, 0.05% and 0.1% P for a flux density B of 1.500 mT (a) DC curves and (b) 400 Hz AC.

of the two B-H loops in Figs. 8(a) and 8(b).

Figure 8(a) shows clearly that the addition of P results in an increase of the coercive field, H_C , when the external H field is increased. The 0.05% P steel had a smaller initial permeability for magnetizations less than 1.0 T. In a high external field, the magnetic permeability of the P-added steel was higher than for the P-free steel. As can be seen in Fig. 8(b), the 0.05% P steel had the lowest H_C value and the highest magnetic permeability regardless of the magnetic induction. The B-H loop of the 0.10% P steel was similar to the B-H loop of the P-free steel. The coercive field, H_C , however was similar to that of the P-free steel.

4. Discussion

4.1. Effect of Grain Size and Crystallographic Texture on the Magnetic Losses

The hysteresis losses have been reported to have an inverse square root dependence on the grain size.^{12–15} The relation between the anomalous losses, P_A , and the grain size, d , has been reported to be $P_A \propto \sqrt{d}$.^{16,17} de Compos *et al.*¹⁸ explained that it was due to the fact that the equilibrium domain wall distance is proportional to the square root of the grain size. The values of P_H and P_A in the W10/400 and W15/400 conditions are shown in Fig. 9. In Fig. 9(a), the hysteresis losses show a clear inverse grain size dependence, and the anomalous losses fit the expected $\sqrt{d}$ dependence. In Fig. 9(b), both hysteresis losses and anomalous losses also fit the expected grain size dependence. The results imply that at a low magnetic induction, *i.e.* away from the magnetic saturation, the influence of grain size on the anomalous losses is not dominant. The influence of the grain size starts to dominate as the magnetic induction increases. It is worth mentioning that the anomalous loss of the 0.05% P steel did not follow a clear grain size dependence and that this steel had smaller losses than expected.

The relation between the texture factor and the magnetic

Fig. 9. Grain size dependence of the hysteresis and anomalous losses: (a) W10/50, (b) W15/50, (c) W10/400 and (d) W15/400.

loss components is shown in **Fig. 10**. The hysteresis losses increase with increasing texture factor (Fig. 10(a)). The results obtained for P_A are more complex. P_A is dependent on the grain size and a lower grain size results in lower losses. The P_A of the samples having average grain sizes in the range of $105\ \mu\text{m}$ to $320\ \mu\text{m}$ decreased with increasing texture factor value, but the rate of the decrease increased with increasing grain sizes. The value of P_A of samples with smaller grain sizes is more sensitive to the texture factor parameter. This inverse correlation has been reported previously by Moses and Pluta.¹⁰⁾ They argued that the magnetization mechanisms of grains close to the GOSS orientation and the non-GOSS oriented grains were different. The domain wall creation and motion is the main source of core losses in the low flux density W10 condition, while domain wall motion, which is the source of the anomalous losses P_A , is more important in the high flux density W15 condition. In the work of Moses and Pluta, the low and high flux densities were used to divide the magnetization process into the early stage of domain wall creation, the stage of domain wall motion and the stage of domain rotation. At low flux density, domain wall creation and domain wall motion take place. Domain wall motion and domain rotation occur at high flux density. In the ND $\parallel$ $\langle 100 \rangle$ oriented grains, the domain wall motion can start at a much smaller external field strength than in the grains with other orientations. This is very likely the reason of the observed increase of P_A with a decreasing texture factor, *i.e.* with an increasing volume fraction of ND $\parallel$ $\langle 100 \rangle$ oriented grains.

Fig. 10. (a) Hysteresis and (b) anomalous losses as a function of the texture factor for $B=1\ 500\ \text{mT}$ and $f=400\ \text{Hz}$.

On the other hand, the low texture factor means the larger volume fraction of ND $\parallel$ $\langle 100 \rangle$ oriented grains. In an entirely $\{100\}\langle 001 \rangle$ oriented material, a significantly larger value of P_A was reported than in the case of a GO-steel.¹⁹⁾ The domain rearrangement in a ND $\parallel$ $\langle 100 \rangle$ oriented material leads to excessive anomalous losses. Thus, the increase of the volume fraction of ND $\parallel$ $\langle 100 \rangle$ oriented grains results in an increase of P_A .

The magnetic anisotropy of Fe when an external field is applied in the main low index crystal directions is represented in **Fig. 11**. A low texture factor implies that a relatively large volume fraction of ND $\parallel$ $\langle 100 \rangle$ oriented grains is present in the microstructure. There is a higher likelihood for grains to have an axis of easy magnetization parallel to an external field direction. The magnetization process of the grains having a ND $\parallel$ $\langle 100 \rangle$ orientation results initially from small reversible domain wall motions. This is followed by irreversible domain wall motion. The domains eventually rotate into the magnetic field direction. Domain rotation was not achieved at $B=1.5\ \text{T}$ when the external field direction was parallel to the $\langle 100 \rangle$ crystal direction in the present work. In other words, the main source of the energy loss in the case of a low texture factor steels was domain wall motion. As P_A is a measure of the energy loss due to domain wall motion, the large P_A value at $B=1.5\ \text{T}$ is essentially a consequence of the low texture factor. This also means that the increase of P_A is less important in the W10 condition. (See Figs. 7(g) and 7(h)). In the case of samples with small grain sizes, the domain wall motion is limited by the grain boundaries and P_A depends mainly on the texture factor.

4.2. Effect of Precipitates

In the previous section the characteristics of the hysteresis losses and the anomalous losses could be explained on the basis of sample grain size and crystallographic texture. For the 0.05% P steel unexpected properties were however obtained. The possible influence of precipitates on the magnetic properties must therefore be considered in this case. The effect of precipitates on the properties of Si steels has been investigated by Shimanaka *et al.*²⁰⁾ who analyzed the effect of non-metallic precipitates on the magnetic properties of NO steels. Small amounts of precipitates containing of C, N, S and O were reported to be essential to guarantee low core losses in NO steel. Shimanaka *et al.* explained their observation by arguing that precipitates induce severe perturbations in the magnetic structure of electrical steel and constitute obstacles to the free movement of domain walls.

Fig. 11. (a) schematic of the magnetic anisotropy of Fe. (b) Effect of the orientation on the domain wall motion and domain rotation during magnetization.

A possible influence of phosphide precipitates has not yet been reported.

In the XRD results of the 0.05% P steel (Fig. 12(a)) and 0.10% P steel (Fig. 12(b)) small diffraction peaks corresponding to three phosphorus-containing precipitates can be observed. The Fe_3P precipitate is a soft magnetic compound (Bohr magneton, $\mu_B=1.38^{21}$). The two other phosphides, FeP_4 and SiP , are non-magnetic. The amount of Fe_3P in the 0.05% P steel was clearly very small, and the size of the precipitates was also expected to be small. Song *et al.*²²⁾ reported that the typical size of Fe_3P in an IF steel is about only 10–20 nm. The diffraction peak height for Fe_3P in the 0.10% P steel is smaller than for the 0.05% P steel.

The direct observation of the effect of the P-bearing precipitates on the domain walls was carried out by Kerr microscopy and the results are shown in Fig. 13. $\langle 100 \rangle \parallel \text{ND}$ -oriented grains were identified by EBSD in the microstructure of the 0.05% P and the 0.10% P steels. The grains were observed in the demagnetized state by Kerr microscopy (Fig. 13(a) and 13(b)). Although both grains were oriented with $\text{ND} \parallel \langle 100 \rangle$, their domain structures were complex. Curved domain walls with strong curvature were observed in the 0.05%P steel. A $\{100\}\langle 001 \rangle$ oriented grain (at 'A') and a slightly misoriented grain from the exact $\{100\}\langle 001 \rangle$ orientation (at 'B') in the 0.05% P steel were studied. The latter grain had a fir-tree domain pattern which is typically observed for a small misorientation from the perfect $\text{ND} \parallel \langle 100 \rangle$ orientation.¹⁹⁾ The grain indicated by 'A' had a complex domain pattern. The domain wall was not aligned to the $[100]$ crystal direction of the grain. The grain consisted of three different types of domains, each one with a specific contrast. The domain walls were aligned at 55° to the $[100]$ and the $[010]$ directions. These domain walls were 90° domain walls. The 90° domain walls have a higher boundary energy than the 180° domain walls which are oriented parallel to a $[100]$ directions. A $\{100\}\langle 013 \rangle$ oriented grain (at 'C') was observed in the 0.10% P steel. The domain walls were parallel to one of the $[100]$ crystal directions of the grain and these domain walls were therefore 180° domain walls.

Fig. 12. X-ray diffraction (XRD) spectrum of (a) 0.05% P steel sample annealed for 30 min and (b) 0.10% P steel sample annealed for 750 min.

The domain wall motion was also analyzed in the 0.05% P steel by Kerr microscopy at increasing external field (Fig. 14). As the external field increased, the size of the domains with a dark contrast increased, and the size of the domains with bright contrast gradually decreased. Domains with an intermediate grey contrast were located in between domains with a dark and a bright contrast. The domain walls between domains with a dark and a grey contrast remained visible when the field was increased. The observed complex domain pattern was due to the presence of 90° domain walls. The formation of 90° domains does not reduce the stray field energy effectively. The formation of 90° domain walls, rather than the lower energy 180° domain walls, may be due to internal stresses generated by the precipitates. Domain walls are strongly influenced by local lattice distortions and very fine and evenly dispersed precipitates can create local strain fields, which will give raised to the formation of 90° domain walls.

Kerr microscopy produces an image contrast related to the magnetization direction of a magnetic domain but there is no contrast of domain boundary itself. Lorentz contrast microscopy however allows the direct observation of domain boundaries. Domain boundaries in the 0.05% P steel were therefore observed in Lorentz contrast TEM microscopy and an example is shown in Fig. 15. The characteristic black and white lines are due to the domain boundaries in Lorentz microscopy. Additional domains were clearly formed around coarse precipitates (<100 nm), and the pinning of domain walls by smaller precipitates was also observed.

Fig. 13. Image combining the magnetic domain structure observed in the de-magnetized state by Kerr microscopy and ND IPF maps obtained by EBSD for (a) the 0.05% P steel annealed at 900°C for 30 min and of (b) the 0.10% P steel annealed at 900°C for 750 min. The arrows indicate precipitates close to the domain walls.

Fig. 14. Kerr microscopy images of the 0.05% P steel at increasing external field strength. The grain orientation and the external field orientation are shown on top.

Fig. 15. TEM Lorentz microscopy of pinned domain walls and the generation of additional domains by precipitates.

In the case of precipitates-free steel, the domain walls are free to move when the material is magnetized as the domain wall motion is also not influenced by the presence of very fine precipitates. Small Cu precipitates (<30 nm) have recently been shown to have no impact on the magnetic properties of electrical steel.²³⁾ A similar effect may occur for fine Fe₃P precipitates which could decrease the de-magnetized domain size and result in a decrease of the anomalous losses. In the present work, the 0.05% P steel had smaller anomalous losses in the W15/400 condition than for the P-free steel. The addition of 0.05% P resulted in a more complex domain structure with a larger number of domain walls. Because of this complex domain structure, the velocity of the domain walls remains small, and the micro-eddy currents which are the source of the anomalous losses are reduced compared to the case of fewer but mobile walls.

In the case of coarser precipitates which were observed in the 0.10% P steel, their effect is not only to pin domain boundaries but also to decrease the magnetic permeability at high magnetic induction. The additional domains surrounding the coarse precipitates are more difficult to magnetize parallel to the external field. The magnetic permeability value of the three steels, *i.e.* the B/H ratio, is shown in **Fig. 16**. The magnetic permeability should in principle be measured in DC conditions from a magnetization curve of a sample in the de-magnetized state. The values represented in Fig. 16 are directly related to external field strength required to achieve a certain magnetic flux densities, such as 1.0 T and 1.5 T and the B/H ratio is not the precise definition of the magnetic permeability. Although the values increase with increasing P content at a low external field, B=1.0 T in DC conditions, the values are shown to decrease with increasing

Fig. 16. Relative magnetic permeability values of the P-free, 0.05% and 0.10% P steels obtained in DC conditions and at 400 Hz.

P content at a high external field, $B=1.5$ T, in both AC and DC conditions. The increase of the magnetic permeability values by an increase of the P content at a low field is simply due to the crystallographic texture effect, *i.e.* it is due to a low texture factor. The decrease of the magnetic permeability values due to increase of the P content at a high field might be due to the presence of the precipitates. The precipitates cause an increase of the core losses due to an increase of the coercive field.²⁴⁾ As coarse precipitates generate additional domains which are difficult to magnetize, the magnetic permeability is reduced. The observed effect of the small P precipitates on the core losses in the 0.05% P steel described in the present work is different from the reported effect of coarse precipitate. The 0.05% P steel had the highest magnetic permeability value at $B=1.0$ T and 400 Hz. In these conditions, the lowest core losses were also measured.

5. Conclusion

The effects of P additions on the magnetic properties of a 2% Si NO steel are listed in Table 3. The grain growth, the recrystallization and the crystallographic texture evolution during annealing is influenced by P additions. In the present work, the effect of crystallographic texture and grain size brought about by P additions, on the core losses of NO steel was investigated. The recrystallization of 0.05 mass% and 0.10 mass% P added steels was significantly delayed. The grain growth rate in these steels was however much higher and the final grain size was larger compared to the grain size in a P-free steel. The crystallographic texture was also improved by P addition and this resulted in reduced hysteresis losses at $B=1.0$ T.

P-containing precipitates were observed to have both of positive and negative effects on the magnetic losses. A clearly negative effect was that large precipitates pinned the domain walls and generated extra domains around the precipitates in the 0.10% P steel. A positive effect was the decrease of the anomalous losses in the 0.05% P steel. Although the presence of the precipitates is generally avoided in NO steels, the results of the present work suggest that it is possible to reduce the anomalous losses by the introduction of specific precipitates.

Table 3. Effect of P additions on the magnetic properties of a 2% Si NO steel.

Effect of P	Control parameter	Increase of P content	Remark
Indirect effect on magnetic properties	Crystallographic texture	Reduction of texture factor	P_H reduce
	Recrystallization	Delay of recrystallization	
	Grain growth	Acceleration of grain growth Grain growth exponent: 5 to 2.5	P_H reduced P_A increased
Direct effect on magnetic properties	Electrical resistivity	Increase of electrical resistivity: $49 \mu\Omega\text{-cm/1 wt\% P}$	P_E reduced
	Domain structure	0.05% P: complex domain structure 0.10%P: domain wall pinning and generation of additional domain around precipitates	P_H increased, P_A reduced P_H, P_A increased at 1.5T

For energy-efficient high-speed rotating machinery, the high frequency anomalous and eddy current losses are considered to be the main magnetic losses. Eddy current losses are reduced by an increase of the electrical resistivity and a reduction of the sheet thickness. Methods to reduce the anomalous losses of NO steel have not yet been unambiguously determined. Although a small grain size and a pronounced ND $\parallel$ $\langle 111 \rangle$ oriented texture are definitely not favorable for the core losses at 50 Hz, both parameters were found to reduce the anomalous losses clearly in the W15/400 conditions. The addition of 0.05% P also proved to lead an alternative method to reduce anomalous losses at the cost of an increase of the hysteresis losses due to a more complex domain structure.

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