Quantitative Analysis of Factors Increasing Coercive Force of Iron Powder Cores-Influence of Porosity*1

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The effect of porosity on coercive force in iron powder cores with different porosities was analyzed quantitatively. The coercive force of the iron powder cores decreased 11.0 A m⁻¹ with a decrease in porosity of 0.01. From in-situ observation by Kerr effect microscopy, nucleation of the reverse domain was observed in local areas along narrow gaps such as the contact interface between particles and fine pores among particles, and nucleation of the reverse domain did not occur at coarse pores. This indicates that the local decrease in the diamagnetic field with a decrease in porosity may be reduced in these areas, resulting in a decrease in coercive force. This result suggests that densification of iron powder cores can be an effective method for reducing coercive force. [doi:10.2320/matertrans.Y-M2021810]

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1. Introduction

Powder cores produced by compaction of soft magnetic metal powders such as iron powder with an insulation coating have higher saturation magnetic flux density than oxide magnetic materials such as ferrite and lower eddy current loss than metal magnetic materials such as electrical steel sheets. Because these properties of powder cores make it possible to reduce iron loss in applications with high operation magnetic flux densities and high operation frequencies, such as magnetic cores of high-speed motors and magnetic cores of reactors for automobiles, in comparison with conventional magnetic materials, powder cores are already used practically in some parts.1) While eddy current loss is low in iron powder cores, the ratio of hysteresis loss to iron loss is high compared with electrical steel sheets. Therefore, further improvement of properties and use in a wider range of applications can be expected by reduction of hysteresis loss.

Since hysteresis loss is proportional to coercive force,2) it is necessary to reduce coercive force in order to decrease hysteresis loss. In soft magnetic materials, coercive force increases as a result of pinning of domain wall movement during magnetization.3) Although precipitated particles, grain boundaries and dislocations are known domain wall pinning sites, the porosity of iron powder cores is also thought to affect the coercive force. Thus, quantification of the individual contributions of the factors which increase coercive force is important for reducing coercive force.

Various studies have examined the relationship between pinning sites and the coercive force of iron powder cores. Nishi et al.4) focused on inclusions as pinning sites and reported an increase in coercive force with an increase in the number of oxide particles per unit volume of a pure iron powder core. Tajima et al.5) also confirmed a decrease in hysteresis loss with a decrease in porosity in an iron powder core, suggesting that porosity has some effect on coercive force. However, previous studies did not fully clarify the mechanism of the increase in coercive force caused by these structural defects.

The model proposed by Pfeifer et al.3) which assumes that coercive force is the sum of the contributions of each pinning site, is a suitable approach for studying the coercive force increase mechanism of each structural defect. In previous work, the authors applied the model of Pfeifer et al. to an iron powder core6,7) and proposed analyzing the factors which increase the coercive force of a pure iron powder core by dividing those factors into the grain boundary, dislocations introduced by plastic deformation of particles, and other remanence factors (pores, inclusions, etc.). That work demonstrated that quantitative separation of the contributions of individual factors is possible.

Among the remanence factors, porosity is an intrinsic coercive force increasing factor of iron powder cores. Since it is impossible to obtain an iron powder core with a perfect dense structure because powder cores are produced by the powder compaction process, clarification of the effect of porosity on coercive force is important when using iron powder cores as magnetic cores. Based on the concept proposed by Pfeifer et al., in the present study, the contribution of porosity to the coercive force of iron powder cores was quantitatively separated by comparison with low carbon steel sheet samples having porosity of 0, and the mechanism by which porosity is related to reverse domain generation and domain wall transfer was clarified.

2. Experiment

2.1 Preparation of iron powder cores

The manufacturing process of iron powder cores is shown in Fig. 1. Here, water atomized powder was annealed at 1223 K for 3.6 ks in a hydrogen atmosphere, after which the particle size distribution of the annealed powder was optimized to 106 to 150 µm by sieving. The apparent density of the sieved powder was 3.47 Mg m⁻³, and the volume based median diameter D₅₀ measured by laser diffraction was 147.9 µm. The chemical composition of the sieved powder

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is shown in Table 1. An insulation coating of silicone resin (SR2400: Dow Corning Toray) was applied to the sieved powder. A silicone resin solution of 1 mass% diluted with xylene was mixed so that the resin solid content was 0.05 mass% or 0.10 mass% with respect to the sieved powder. After the drying process, the mixed powder was cured at 473 K in air for 7.2 ks to obtain an insulation-coated iron powder having a silicone resin layer adhering to the surface of the iron powder particles. The insulation-coated iron powders were compacted into ring-shaped cores (outer diameter: 38 mm, inner diameter: 25 mm, height: 6 mm) at room temperature and compaction pressures of 980, 1470 or 1960 MPa. To remove the strain induced during compaction, these as-compacted cores were annealed at 873 K for 2.7 ks.

In order from the lowest compaction pressure, the cores made from the 0.05 mass% insulation-coated iron powder were referred to as L1, L2 and L3, and those made from the 0.10 mass% insulation-coated iron powder were referred to as H1, H2 and H3.

2.2 Preparation of steel sheet ring

The chemical composition of the ultra-low carbon hot-rolled steel sheet is also shown in Table 1. The hot-rolled steel sheet was cold rolled to a thickness of 0.5 mm, and the cold-rolled steel sheet was annealed at 873 K in a nitrogen atmosphere. The annealing time was varied to 0, 30, 180 or 600 s to obtain steel sheets with four different crystal grain sizes. These annealed sheets were electrical discharge machined into a ring shape with the same outer and inner diameters as the iron powder cores, and are denoted as S1, S2, S3 and S4 in order from the lowest annealing time.

2.3 Evaluation

The densities and magnetic path lengths of the cores were calculated from their dimensions and weights. The magnetic path length is defined as the circumference of a circle whose diameter is the average of the outer and inner diameters of the ring. The porosity $P$ of the iron powder cores was calculated from eq. (1).

$$P = \frac{D_t - D_p}{D_t}$$  \hspace{1cm} (1)

Where $D_t$ is the true density of pure iron (7.87 Mg m$^{-3}$), and $D_p$ is the density of the iron powder core.

The direct current (DC) hysteresis loop was measured with a DC magnetometer (Metron, Inc. Type: SK-110). Primary and secondary coils (100 and 40 turns, respectively) were wound with $0.6 \text{ mm}$ insulated copper wire. The maximum magnetic induction was 1.0 T, and the coercive force was evaluated from the DC hysteresis loop.

For optical micrographs, each specimen was molded in a thermoplastic resin, polished and etched with nital. The cores were cut before molding to observe the cross section perpendicular to the circumference. The crystal grain size was measured from the optical micrograph by the intercept method. The average crystal grain size of more than 40 particles of each specimen was defined as the crystal grain size for each of the base powders and annealed cores.

The magnetic domain of iron powder core L1 was observed with a Kerr effect microscope (NeoArk, BH-786 V-JS). First, the core was embedded so that the radial cross section of the ring was the observation plane, and one side was mirror-polished. After the thickness of the molded core was reduced to 0.5 mm by polishing, a mirror finish was also applied to the opposite surface. After chemical polishing with colloidal silica, domain observation was carried out. The applied magnetic field was directed in the observation plane, and observation was started when the magnetic domain disappeared as a result of application of the magnetic field. The behavior of the magnetic domain generated in the process of gradually lowering the magnetic field was observed. The effect of plastic strain on the magnetic domain structure in the domain observation region was investigated.

Table 1 Chemical composition.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>O</th>
<th>S</th>
<th>N</th>
<th>P</th>
<th>Mn</th>
<th>Si</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base iron powder</td>
<td>0.001</td>
<td>0.024</td>
<td>&lt;0.001</td>
<td>&lt;0.0005</td>
<td>&lt;0.002</td>
<td>&lt;0.010</td>
<td>&lt;0.010</td>
<td>0.002</td>
</tr>
<tr>
<td>Base Steel sheet</td>
<td>0.001</td>
<td>0.008</td>
<td>&lt;0.001</td>
<td>0.0018</td>
<td>&lt;0.002</td>
<td>&lt;0.010</td>
<td>&lt;0.010</td>
<td>0.004</td>
</tr>
</tbody>
</table>
by conducting a SEM/EBSD (Scanning Electron Microscopy/Electron Backscattering Diffraction) analysis. A JSM-7100F (JEOL Ltd.) was used for the analysis, and the KAM (Kernel Average Misorientation) was calculated and mapped from the EBSD data. The analysis conditions were the same as in the previous report.7)

3. Experimental Results

The relationship between compaction pressure and porosity of the iron powder cores is shown in Fig. 2. Porosity decreased with an increase in the compaction pressure, and porosity was also lower with the smaller amount of insulation coating. The relationship between compaction pressure and coercive force is shown in Fig. 3. Coercive force decreased with an increase in compaction pressure from 980 to 1470 MPa with both amounts of insulation coating powder. However, no difference in coercive force between the 1470 MPa and 1960 MPa compaction pressures was observed, and the amount of insulation coating had no effect on coercive force. The relationship between the porosity and coercive force of the iron powder cores is shown in Fig. 4. Coercive force decreased with a decrease in porosity, but saturated under the $P$ of 0.016. This suggests that factors other than porosity contribute to the coercive force of the iron powder cores.

Optical micrographs of the microstructure of the iron powder cores are shown in Fig. 5. The crystal grain boundaries in the iron powder particles were confirmed by nital etching. The relationship between the average crystal
grain size $d_i$ measured by the intercept method from the optical micrographs and porosity is shown in Fig. 6. $d_i$ decreased with a decrease in porosity.

The relationship between the annealing soaking time and coercive force of the steel sheet rings is shown in Fig. 7. Coercive force decreased with an increase in soaking time. Optical micrographs of the microstructure of the steel sheet rings are shown in Fig. 8, and the relationship between the soaking time and average crystal grain size of the steel sheet ring is shown in Fig. 9. The average crystal grain size of the steel sheet rings increased with an increase in soaking time. The relationship between the average crystal grain size and coercive force of the steel sheet rings is shown in Fig. 10. Coercive force decreased with an increase in crystal grain size.

From the above results, it was suggested that the effect of the crystal grain size should be separated in order to consider the effect of $P$ on the coercive force of iron powder cores.

4. Discussion

4.1 Quantification of influence of porosity on coercive force of iron powder cores

The coercive force and crystal grain size $d_i$ of the iron powder cores changed as porosity changed. Pfeifer et al.\cite{3}
analyzed coercive force by assuming that coercive force is the sum of the contributions of individual pinning sites. In the previous report, the coercive force of the iron powder core was expressed as the sum of the contributions of grain size, dislocations and other pinning sites, and the mechanism of domain wall pinning by grain boundaries and dislocations was analyzed. In this paper, this mechanism is discussed in greater detail by expressing the coercive force by eq. (2).

Here, we assume that Pfeifer’s model can also be applied to the contribution of porosity.

\[ H_c = H_{ck} + H_{c-dis} + H_{c-p} + H_{c-ex} \quad [A \text{ m}^{-1}] \]  

(2)

Where \( H_{ck} \) is contribution of crystal grain boundary, \( H_{c-dis} \) is contribution of dislocation, \( H_{c-p} \) is contribution of porosity and \( H_{c-ex} \) is other contributions (inclusions etc.). \( H_{ck} \) in eq. (2) can also be expressed as eq. (3) by using the average crystal grain size \( d \).

\[ H_{ck} = A/d \quad [A \text{ m}^{-1}] \]  

(3)

Where \( A \) is a coefficient determined by domain wall energy and saturation magnetization, and also experimentally evaluated from the relationship between the inverse of crystal grain size and coercive force. The relationship between the inverse of crystal grain size and coercive force of the steel sheet rings is shown in Fig. 11. The plots show a highly correlated linear relationship with a slope of \( 1.3 \times 10^3 \). As described in the following, all steel sheet rings were fully recrystallized, and the influence of the strain induced by electro discharge machining is considered to be very small. The terms other than \( H_{ck} \) can be regarded as constant for the change of \( d \). Therefore, eq. (4) is obtained by substituting the slope into eq. (3), and the contribution of the crystal grain size to coercive force can be calculated from the average crystal grain size.

\[ H_{ck} = 1.3 \times 10^3/d \quad [A \text{ m}^{-1}] \]  

(4)

It has also been reported that \( H_{c-dis} \) in eq. (2) is proportional to the square root of the dislocation density \( \rho \), and is expressed as eq. (5).

\[ H_{c-dis} = \gamma \cdot \rho^{1/2} \quad [A \text{ m}^{-1}] \]  

(5)

Where \( \gamma \) is a coefficient determined by saturation magnetization, domain wall area per volume, average distance between domain walls and distribution of dislocations, and was reported to be 923. The dislocation density in the iron powder cores increases during compaction, but is reduced by recrystallization by annealing, and the dislocation density at 973 K, where recrystallization was completed, was approximately \( 1.2 \times 10^{13} \text{ m}^{-2} \). The annealing temperatures of the iron powder cores and the steel sheet rings prepared in this study were both 873 K, which is as much as 100 K lower than in the previous report. In spite of this significantly lower annealing temperature, recrystallization is considered to be complete because the optical micrographs did not reveal etching marks in the crystal grains caused by plastic strain, i.e., unrecrystallized regions with a high dislocation density.

The strain caused by electro discharge machining (EDM) is also smaller than that in shearing processes such as punching. In a study by Tono et al. using Fe–3Si, the strain-affected area was very small, even at a distance of about 0.03 mm from the cut surface. Therefore, in this study, the effect of the strain caused by EDM on coercive force is assumed to be very small, and \( H_{c-dis} \) of all iron powder cores and steel sheet rings is estimated to be 28.0 A m\(^{-1}\) from both the coefficient \( \gamma = 923 \) and the dislocation density \( \rho = 1.2 \times 10^{13} \text{ m}^{-2} \).

Equation (6) is obtained by substituting \( H_{c-dis} \) (28.0 A m\(^{-1}\)) and eq. (4) into eq. (2).

\[ H_c = 1.3 \times 10^3/d + 28 + H_{c-p} + H_{c-ex} \quad [A/m] \]  

(6)

Rearranging this formula, eq. (7) is obtained.

\[ H_{c-p} + H_{c-ex} = H_c - 1.3 \times 10^3/d - 28 \quad [A/m] \]  

(7)

In addition, \( H_c = 1.3 \times 10^3/d - 28.0 \) is replaced by \( H_{ck-dis} \) and eq. (8) is obtained.

\[ H_{ck-dis} = H_{c-p} + H_{c-ex} \quad [A/m] \]  

(8)

Where \( H_{c-ex} \) includes the contribution of inclusions. However, since all iron powder cores in this research were made from the same base iron powder, the contribution of inclusions can be regarded as a constant. Thus, the contribution of \( H_{c-p} \) can be estimated from the relationship between \( H_{ck-dis} \) and porosity \( P \). The relationship between porosity \( P \) and \( H_{ck-dis} \) calculated from eq. (8) is shown in Fig. 12. The plots of steel sheet rings S1 to S4 are also shown in Fig. 12 as \( P = 0 \).
core increases 11.0 A m$^{-1}$ for the iron powder cores, while the value of eq. (9) is 10.2 A m$^{-1}$ for the steel sheet rings and the iron powder cores are almost identical, the coercive force should be investigated by microstructural observation by transmission electron microscopy (TEM).

Fig. 12 Relationship between porosity and $H_{c-k-dis}$ of iron powder cores. The coercive force should be investigated by microstructural observation by transmission electron microscopy (TEM).

From the above discussion, the contribution of porosity to the coercive force of the iron powder cores was quantitatively extracted by using the Pfeifer model, in which the coercive force is the sum of the contributions of the individual pinning factors.

### 4.2 Mechanism of contribution of porosity to coercive force

From the discussion in the previous section, the contribution of porosity $H_{c-p}$ was found to be proportional to $P$. Since a porosity (pore) is a nonmagnetic region contained in the microstructure of an iron powder core, its effect on coercive force is expected to be close to that of a nonmagnetic inclusion. Neel et al.\(^\text{15}\) attempted to quantify the contribution of inclusions to coercive force from the viewpoint of the magnetostatic energy due to the disappearance of domain walls and the formation of magnetic poles, and in the case of pure iron, that contribution is expressed as eq. (10).

\[
H_c = 2.9 \times 10^4 \cdot \alpha [\text{A m}^{-1}] \quad (10)
\]

Where $\alpha$ is the volume fraction of nonmagnetic inclusions. Considering that a nonmagnetic inclusion is regarded as a pore and $\alpha$ is equivalent to $P$ in eq. (9), the coefficient of $2.9 \times 10^4$ in eq. (10) is considered to correspond to $C_p$ in eq. (9). As mentioned above, $C_p$ is estimated to be $1.1 \times 10^3$ in this study. This is smaller than the coefficient of $2.9 \times 10^4$ in eq. (10), suggesting that the contribution of porosity to the coercive force of iron powder cores is due to a mechanism different from the magnetostatic energy change caused by the disappearance of domain walls and the formation of magnetic poles near inclusions, as pointed out by Neel et al. In order to clarify the mechanism of the contribution of pores to coercive force, the magnetic domain structure in the iron powder cores was observed by Kerr effect microscopy. The KAM map of the iron powder core L1 obtained by the SEM/EBSD method and the magnetic domain observation results observed by changing the external magnetic field are shown in Fig. 13 and Fig. 14, respectively. Focusing on the KAM map, it can be seen that almost all regions are KAM $< 1^\circ$ and the dislocation density is sufficiently low, even though damage by partial polishing was confirmed. Therefore, the effect of dislocations on the magnetic domain structure in the observation area can be regarded as negligible. The magnetization direction of the magnetic domain observation results is the right side in the horizontal direction of the page, and the red line in the figure shows the iron powder particle...
boundary. At 1062 Oe, where the external magnetic field is the highest, the magnetic domain structure consists of magnetic domains aligned in almost the same direction. When the magnetic field is gradually lowered, the generation of reverse magnetic domains is observed, as indicated by the white contrast in the observation area. In the external magnetic field of 224 Oe, the local filling rate of the particles was high, and generation of preferential reverse magnetic domains from the narrow gaps between particles and regions with pore circumferences was confirmed. However, generation of a clear reversed domain was not confirmed in the vicinity of the coarse pore at the right end of the observation area. Therefore, the region where reversed domains are generated is a region where magnetization reversal by an external magnetic field easily occurs and coercive force is low, whereas the vicinity of the pore where a clear reversed magnetic domain was not confirmed seems to be a region where magnetization reversal by an external magnetic field is difficult.

Since a magnetic powder core is an aggregate of soft magnetic particles with insulating coatings, a demagnetizing field which originates from a gap between the particle surface and the particle might be generated in the structure. Takajo et al. formulated the influence of the demagnetizing field in the structure on the permeability of an iron powder core by using the effective demagnetizing field constant $N$, and furthermore showed that the effective demagnetizing field coefficient decreases with an increase in density, i.e., a decrease in porosity. Conversely, from the results of magnetic domain observation (Fig. 13) of the iron powder cores, it is estimated that the effective demagnetizing field coefficient increases locally in the vicinity of pores with a large gap between particles, and this makes magnetization reversal by an external magnetic field difficult. In addition, increasing such regions makes magnetization reversal due to the generation of reverse magnetic domains difficult, and as a result, coercive force is increased. This conjecture is consistent with the relationship between porosity and coercive force discussed in 4.1.

A decrease in coercive force due to an interaction with neighboring particles like that described above has also been reported in hard magnetic materials consisting of single domain particles. This implies that coercive force might be reduced by enhanced magnetic interaction with neighboring particles even if the powder particle has the multidomain structure used in this research. However, the possibility that the observed reverse domains are reflux domains is also suggested. The effect of refluxing domains on coercive force is known to be due to the lancet domain in grain oriented electrical steel sheets and an increase in coercive force with an increase in refluxing domains has been reported. Thus, it is difficult to determine the cause of the increase in coercive force from this study alone. Additional domain wall observation and quantification of the magnetizing direction will be required in order to resolve this issue.
5. Conclusions

In this study, pure iron powder cores with the same dislocation density and inclusions but different porosities were fabricated, and the influence of porosity on coercive force was quantitatively evaluated. In addition, the mechanism of inhibition of domain wall movement, i.e., domain wall pinning, by the pores was revealed by domain observation in the demagnetization process. The conclusions were as follows.

(1) As the porosity of the iron powder cores decreased, the crystal grain size became finer due to recovery of plastic strain and recrystallization, but coercive force decreased.

(2) Based on the result in (1), the contributions of crystal grain size and porosity to coercive force were quantitatively separated by using the relationship between the inverse of the crystal grain size and coercive force of steel sheet rings with zero porosity, and the model proposed by Pfeifer et al., in which coercive force is assumed to be the sum of the contributions of each pinning site. As a result, the contribution of porosity to coercive force decreased with a decrease in porosity. The decrease in coercive force per 0.01 decrease in porosity is estimated to be 11.0 A m$^{-1}$.

(3) Magnetic domain observation of the iron powder cores by Kerr effect microscopy revealed that preferential reverse magnetic domains are generated from narrow gaps between particles and areas with minute pore circumferences. In regions of these types, the demagnetizing field coefficient is locally low, and as a result, the effective magnetic field rises and generation of reverse magnetic domains with magnetization reversal is thought to become easy.

(4) From these results, it was concluded that the coercive force of pure iron powder cores is reduced by decreasing porosity by densification and by increasing narrow pores, such as the contact interface between particles, where reverse domains are easily generated.

REFERENCES