

# Newly Developed Iron Powder for Highly Efficient Dust Cores

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*Dust cores are made by compacting insulated magnetic powder. One of the advantages of dust cores is magnetic isotropy, which enables three-dimensional magnetic circuit designs. Such designs are expected to reduce the size and weight of magnetic parts. There is a strong demand to reduce core loss so that the cores can be used more for magnetic parts, such as motor cores. This paper reports on methods for reducing iron loss in dust cores and on the magnetic properties of the dust cores consisting of improved powder made by a new method. A dust core with an iron loss of 28.6W/kg was produced. This core is comparable with ones made of laminated steel sheets.*

## Introduction

To decrease the environmental burden and to save energy, there are growing needs for electromagnetic components with reduced loss. Components such as photovoltaic power conditioners and the reactors that are used for boost converters of hybrid electric vehicles (HEVs) and electric vehicles (EVs) require magnetic materials that have low magnetic loss and can be used at higher frequencies. In addition, the electric motors used for HEVs and EVs call not only for reduced magnetic loss, but also for smaller size and lighter weight.

Dust cores, made by compacting insulated soft-magnetic powder, have the advantage that they can suppress eddy current loss because the constituent particles are electrically insulated. The eddy current loss is proportional to the second power of frequency. Thus, components used at higher frequencies can better exploit this advantage. Because of this, dust cores are progressively being used for applications such as the reactors for the boost converters of HEVs.

In addition, dust cores have isotropic magnetic characteristics. This enables the designing of new magnetic circuits with three-dimensional structures, which have been difficult to design using conventional magnetic cores made of laminated steel sheets having magnetic anisotropy. Because of this, various efforts are being made to exploit dust cores for the downsizing and weight reduction of components, including electric motors<sup>1), 2)</sup>. Such components, however, are typically driven by the commercial frequency, which is so low that the iron loss is governed predominantly by hysteresis loss, rather

than by eddy current loss. This makes it difficult to take advantage of the reduced eddy current loss of dust cores, hence the desire for a dust core with decreased hysteresis loss.

The present paper describes technologies, including the determination of optimum temperature for magnetic annealing to relieve strain for reducing the iron loss of dust cores. This paper also includes the development of a highly heat-resistant insulation coating for the optimum magnetic annealing, as well as the effect of powder particles and crystal grain sizes on iron loss. Also introduced are the characteristics of a powder compact with reduced iron loss, the compact having been developed on the basis of the knowledge that was obtained.

## 1. Factors determining iron loss of dust core

In general, iron loss consists of eddy current loss and hysteresis loss. The eddy current loss is further classified into intra-particle eddy current loss, caused by the eddy current flowing inside each individual particle, and inter-particle eddy current loss, caused by the eddy current flowing across the particles throughout the component.

The hysteresis loss of a material, on the other hand, corresponds to the looped area in the BH (magnetic flux density vs. magnetic field) curve, in which the size of the loop correlates strongly with the material's coercive force (i.e., coercivity). Thus, decreasing the coercivity decreases the hysteresis loss. The factors determining coercivity are particle composition (solid solution elements), precipitates, inclusions, crystal grain boundaries, powder surfaces and dislocations (strains), i.e., the factors that inhibit the movement of domain walls.

The factors determining the intra-particle eddy current loss include particle composition (solid solution elements), which affects the electrical resistivity of each particle, and particle size, which defines the area within which the eddy current flows. The factors determining the inter-particle eddy current loss include the electrical resistivity of the insulation coating, which affects the inter-particle insulation (**Fig. 1**).

Among these factors, the coercivity is decreased more significantly as the temperature for magnetic annealing, performed to relieve strain, rises.

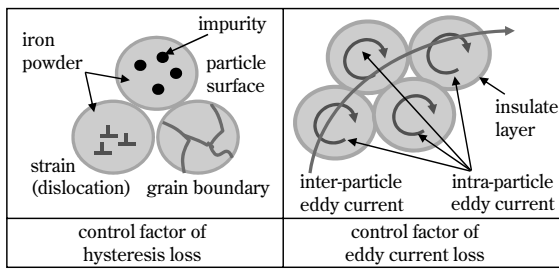


Fig. 1 Control factor of iron loss

Conventional dust cores, however, suffer from the problem of the lack of heat resistance of the insulation coating on the particle surfaces, which causes the fracture of inter-particle insulation during annealing at elevated temperatures, resulting in increased inter-particle eddy current loss. Another known technique for decreasing coercivity is to increase particle size. Intra-particle eddy current loss, however, is proportional to the second power of particle size; hence particle size is a factor controlling trade-off characteristics.

From the above, it can be seen that an important issue in reducing the iron loss of a dust core is to suppress its eddy current loss and at the same time to decrease its hysteresis loss.

## 2. Optimum annealing temperature for relieving stress from compacts

Dust cores are compacted in dies. Therefore, the powder particles are plastically deformed and have a large amount of strain introduced. Strain (dislocation) is a factor which increases coercivity and can be relieved by heat treatment. A study was conducted to determine the optimum annealing temperature for strain relief.

### 2.1 Experimental method

Pure iron powder (300NH, manufactured by Kobe Steel; average particle size, approximately  $100\mu\text{m}$ ) is mixed with 4.0mass% of highly-pure alumina fine powder (manufactured by Showa Denko K.K.; average particle size, approximately  $1.3\mu\text{m}$ ), along with an organic binder. The particles of the highly-pure alumina powder are adhered to the surface of iron powder particles, forming insulation layers. The powder mixture was compacted into ring-shaped samples, each having an outer diameter of 45mm, inner diameter of 33mm and height of 5mm, by die-wall lubricated compaction with a compacting pressure of 980MPa. Zinc stearate was used as the die wall lubricant. Each compact was annealed in a nitrogen atmosphere at a temperature in the range of 673 to 1,073K for 7.2ks. The direct-current magnetic

properties were measured on these samples, using a BH curve tracer (BHS-40S by Riken Denshi Co., Ltd). Furthermore, the sample cross-section was etched with NITAL (an etching solution consisting of ethyl alcohol and nitric acid) so as to be subject to metallographic observation under an optical microscope.

## 2.2 Results and discussions

Fig. 2 shows the relationship between the temperatures for magnetic annealing and the resulting coercivities of the samples. The coercivity decreases as the annealing temperature approaches 973K. This is considered to be caused by the relief of strain in the compacts by the magnetic annealing. The sample annealed at 1,073K exhibits a coercivity higher than that of the sample annealed at 973K. There is only a slight difference in coercivity between the compact annealed at 873K and the one annealed at 973K. This result indicates that the coercivity-decreasing effect of annealing becomes nearly saturated at about 873K.

Fig. 3 are optical micrographs of the cross-sections of two compacts, i.e., one before magnetic annealing, and the other after magnetic annealing at 1,073K. The crystal grains of the compact annealed at 1,073K are finer than they were before magnetic annealing. This shows recrystallization. In general, the smaller the grain size, the higher the coercivity. Thus, the increase in coercivity of the compact annealed at 1,073K is attributable to the grain refining caused by recrystallization. If we examine this in more detail, in the temperature range no lower than 873K, the

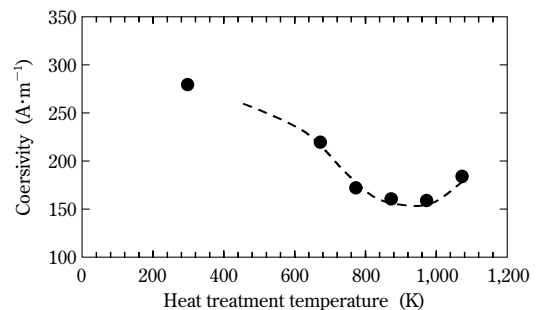


Fig. 2 Relationship between coersivity and annealing temperature

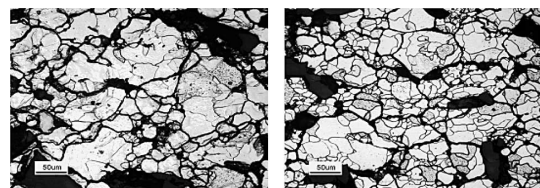


Fig. 3 Cross sectional microstructure of before annealed (left) and annealed at 1,073K(right) compact

coercivity-decreasing effect of magnetic annealing lessens, and the upper limit annealing temperature, effective in decreasing coercivity, is regarded as to be approximately 973K. In addition, magnetic annealing at 1,073K has turned out to adversely affect the coercivity reduction.

From the above, the optimum temperature for magnetic annealing of dust core made from pure iron powder is concluded to exist in the temperature range from 873K to 973K.

### 3. Heat resistance of insulation coating

The magnetic annealing at the optimum temperature determined in section 2 is effective in decreasing coercivity and thus reducing hysteresis loss; however, conventional insulation coatings suffer from the problem of their heat resistance not being high enough. The following outlines a heat resistant coating developed to solve this problem.

#### 3.1 Experimental method

Three powder samples mainly consisting of pure iron powder, 300NH, were prepared. Each powder sample was coated with two layers, i.e., one inorganic layer and one organic layer. Each inorganic layer was formed from one of three phosphoric acid-based solutions (A, B or C) for insulation. The organic layer consists of silicone resin having heat resistance. The coated powder samples were compacted by die-wall lubricated compaction with a pressure of 980MPa into plate-shaped specimens, each having dimensions of 12.7mm × 31.8mm × 5mm. The compact specimens were annealed in a nitrogen atmosphere at a temperature in the range of 773 to 873K for 1.8ks. Their electrical resistivities were measured by a four-terminal method.

#### 3.2 Results and discussion

The electrical resistivity of an insulation coating varies depending on the coating material, coating thickness and magnetic annealing temperature. These factors, determining the electrical resistivity of insulation coating, were changed to study the eddy current losses of samples with various electrical resistivities (Fig. 4). The result indicates that an electrical resistivity higher than about 100 μΩ·m effectively suppresses the inter-particle eddy current loss. It is thus concluded that the coating used for a dust core must have an electrical resistivity that causes the overall resistivity of the compact to be higher than about 100 μΩ·m.

Fig. 5 compares the dependence of electrical

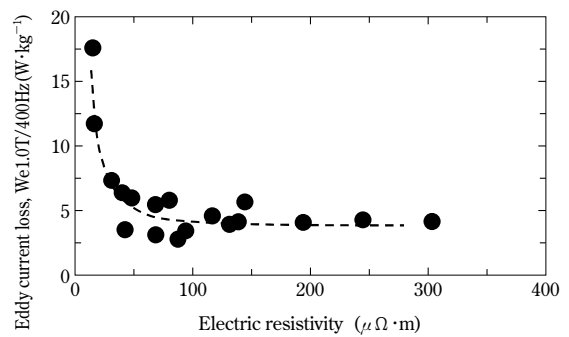


Fig. 4 Relationship of eddy current loss and electric resistivity

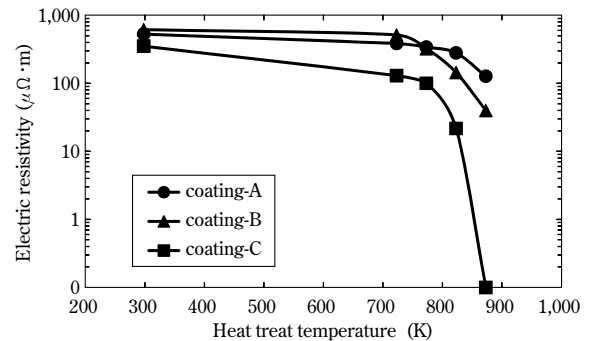


Fig. 5 Relationship between electric resistivity and annealing temperature

resistivities on the annealing temperature of powder compacts, each coated with two layers, one layer consisting of one of the three inorganic materials made from phosphoric acid solutions and the other layer consisting of silicone resin. All the coatings exhibit a decrease in electrical resistivity with rising annealing temperature. The decrease is considered to be due to the deterioration of the coatings. The two-layered coating, consisting of a layer of inorganic material (A) and a layer of silicone resin, exhibits a smaller decrease in electrical resistivity and has an electrical resistivity higher than 100 μΩ·m at an annealing temperature of 873K, the resistivity required at the optimum magnetic annealing temperature. There are reports on the improvement in heat resistance of inorganic coating made from a phosphoric acid-based solution. Such reports include one focusing on the vitrification temperature of coating<sup>3)</sup> and another focusing on the diffusion of elements<sup>4)</sup>. However, the cause of the high heat resistance of the two-layer coating, consisting of the inorganic layer (A) and silicone resin, as reported in this paper, requires further investigation.

Conventional insulation coatings have low heat resistance, limiting the magnetic annealing temperature to around 773K. The newly developed two-layered coating allows magnetic annealing at 873K, a temperature high enough for the strain relief of dust cores.

#### 4. Effect of powder particle size and crystal grain size on iron loss

Studies were conducted on the effects of powder particle size and crystal grain size on iron loss. The powder particle size, in particular, affects both the hysteresis loss and eddy current loss. Thus, a study was conducted from the two aspects of hysteresis loss and eddy current loss. Also outlined is the relation between the powder particle size and crystal grain size, which are inseparably connected.

##### 4.1 Experimental method

Pure iron powder, 300NH, was heat-treated in powder form. Each heat-treatment cycle was conducted in a nitrogen atmosphere at 1,243K for 5.4ks and the cycle was repeated three times to grow crystal grains. The resulting powder was sifted using sieves with mesh sizes of 180, 150, 100, 75, 63 and 45 $\mu$ m. Powder samples were prepared with the finer particles removed sequentially, as described in 4.2. Each powder sample thus obtained was compacted into a ring-shaped specimen having a compact density of 7.60Mg/m<sup>3</sup> by die-wall lubricated compaction. Each compact specimen was annealed in a nitrogen atmosphere under one of two conditions, i.e., 823K for 1.8ks, or 873K for 1.8ks. The annealed specimens were measured for their direct current magnetic properties. Iron loss was measured by an automatic magnetic measurement apparatus (manufactured by Yokogawa Electric Corporation). The conditions for iron loss measurement were the excitation magnetic flux density, 1.5T, and excitation frequency, 200Hz.

##### 4.2 Results and discussion

In a magnetic material, interfaces such as grain boundaries and powder surfaces provide sites for preventing domain wall migration. To study this behavior, pure iron powder was heat treated to grow its crystal grains. Fig. 6 shows the cross-sectional micrographs of the powders before and after the heat treatment. The heat treatment has been shown to grow the crystal grains.

Fig. 7 compares the coercivities of the compacts of the powders before and after the heat treatment applied for crystal grain growth. The figure indicates that coercivity is decreased as a result of the grain growth.

After the grain growth heat treatment, the powder was sifted using sieves with mesh sizes of 180, 150, 100, 75, 63, 45 $\mu$ m. Powder samples were prepared with finer particles removed sequentially. The size

distribution width of the powder samples were A) 0~250 $\mu$ m; B) 45~250 $\mu$ m; C) 63~250 $\mu$ m; D) 75~250 $\mu$ m; E) 106~250 $\mu$ m; F) 100~250 $\mu$ m; and G) 180~250 $\mu$ m. Fig. 8 shows the coercivities of the compacts made from these powders. It has turned out that coercivity is effectively decreased by growing crystal grain and removing finer particles from the powder to increase the average particle size.

If all the powder particles become single crystals as a result of the powder heat treatment for crystal grain growth, no further increase in the average grain size is expected. Thus, finer particles serve to decrease coercivity. When removing finer particles by sieving, it is effective to remove particles smaller than the crystal grain size; however, fine crystals in coarse particles still inhibit the decrease in coercivity.

Thus, in order to decrease coercivity, it is important to combine the removal of fine particles (coarsening) and the growth of crystal grains.

It should be noted that increasing the particle size increases the intra-particle eddy current. Therefore, particle size should be selected by incorporating the

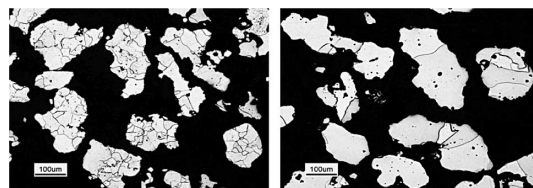


Fig. 6 Cross sectional microstructure of powder with(right) and without(left) grain growth treatment

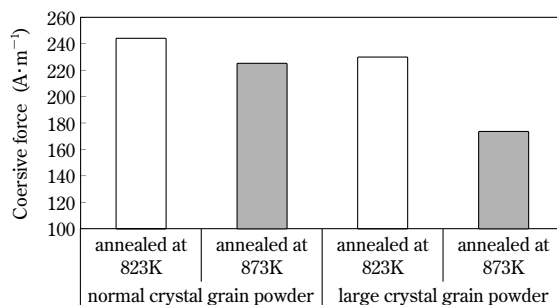


Fig. 7 Coercive force comparison of crystal grain size

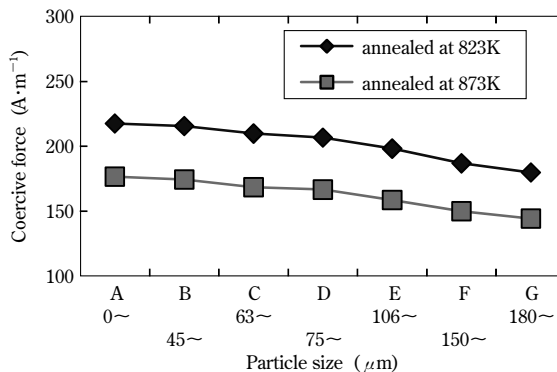


Fig. 8 Relationship of particle size and coercive force

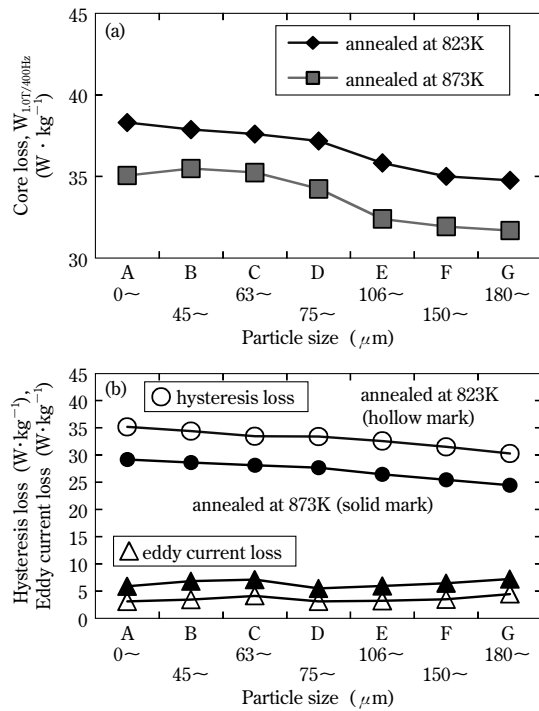


Fig. 9 Relationship between particle size and core loss(a), and between particle size and hysteresis, and eddy current loss(b)

decreasing effect of hysteresis loss and increasing effect of eddy current loss.

Fig. 9 (a) represents the iron losses of compacts made from iron powders with or without fine particles, and Fig. 9 (b) represents the respective iron loss, separated into hysteresis loss and eddy current loss. As shown in Fig. 9 (b), removing fine particles is effective in decreasing both coercivity and hysteresis loss and has a minimal effect in increasing eddy current loss within the particle size distribution investigated. Therefore, the iron loss shown in Fig. 9 (a) monotonically decreases as a result of removing fine particles.

### 5. Characteristics of newly developed iron powder with reduced iron loss

The following introduces the iron powder with reduced iron loss developed on the basis of the knowledge obtained.

To investigate the characteristics of compacts made from the newly developed powder, pure iron powder (i.e., starting material, 300NH), was classified using a sieve with  $150\mu m$  mesh openings to remove fine particles. The remaining coarse powder was treated to grow its crystal grains. The treated powder was coated by two-layer insulation, consisting of a phosphoric inorganic layer (A) and a silicone resin layer. This powder was heated to 403K, compacted by die-wall lubricated compaction at a pressure of 1,176MPa, and was annealed in a nitrogen atmosphere at 873K for 1.8ks to prepare the compact specimen.

The magnetic properties and iron losses of the specimens are shown in Table 1 and Fig.10, respectively. Also shown for comparison are the characteristics of a conventional material and electrical steel sheets. The ring-shaped specimens for the electrical steels were prepared by wire cut. The conventional dust core in Fig.10 was made from 300NH with conventional insulation coating applied, after which it was annealed at 773K for 1.8ks.

The newly developed powder exhibits significantly reduced iron loss compared with the conventional materials. It shows reduced iron loss in the frequency range above 200 to 300Hz, compared with a conventional electrical steel sheet, JIS 50A400. It also shows reduced iron loss in the frequency range above 700~1kHz, compared with JIS 35A360. Thus the newly developed powder is expected to be used for applications such as motors that rotate at relatively high speed.

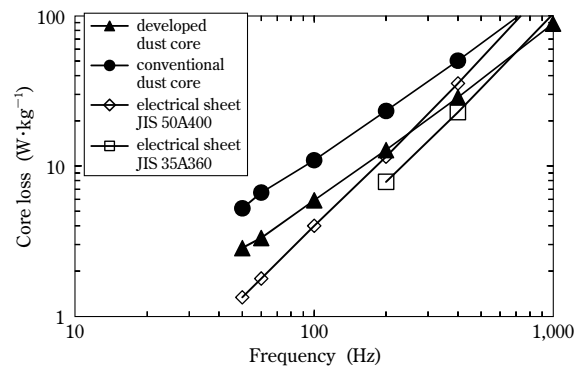


Fig.10 Comparison of core loss

Table 1 Properties of developed material

|                  |              | size distribution (sieve size) ( $\mu m$ ) | density ( $Mg/m^3$ ) | heat treatment temperature (K) | flux density (@8,000A/m) (T) | maximum permeability | coersivity (A/m) | core loss (1.0T/400Hz) ( $W/kg$ ) | transverse rupture strength (MPa) |
|------------------|--------------|--|----------------------|--------------------------------|------------------------------|----------------------|------------------|-----------------------------------|-----------------------------------|
| dust core        | developed    | 150~250                                    | 7.69                 | 873                            | 1.66                         | 669                  | 134              | 28.6                              | 76                                |
|                  | conventional | 0~250                                      | 7.61                 | 823                            | 1.58                         | 446                  | 243              | 50.4                              | 82                                |
| electrical sheet | JIS 50A400   | —  | —                    | as cutted                      | 1.8                          | 6,213                | 60               | 35.5                              | —                                 |
|                  | JIS 35A360   | —  | —                    | as cutted                      | 1.79                         | 6,532                | 59               | 22.9                              | —                                 |

## Conclusion

As a means for decreasing coercivity and hysteresis loss, a new iron powder was developed focusing on magnetic annealing, powder particle size and crystal grain size. The following was found:

- The optimum magnetic annealing temperature for dust cores made from pure iron powder lies in the range from 873 to 973K. Annealing at temperatures above this range adversely increases coercivity. This is partially due to the refinement of crystal grains caused by recrystallization.
- An electrical resistivity higher than about  $100\mu\Omega\cdot\text{m}$  is required to suppress the magnetic loss caused by inter-particle eddy current. An insulation coating was developed which maintains the required resistivity even after magnetic annealing at 873K. This coating has made it possible to decrease coercivity and, therefore, hysteresis loss.
- The effects of crystal grain size and powder particle size on the coercivities of compacts have been clarified. A significant reduction of coercivity is made possible by combining the size effects of

crystal grains and powder particles, rather than controlling them independently.

- Combining the newly-developed heat-resistant insulation coating with crystal grain coarsening and particle coarsening (removal of fine particles) enable the production of a dust core having iron loss comparable with that obtained by conventional electrical steel sheets. Dust cores have the feature of being able to suppress eddy current loss at high frequencies. Thus, the newly developed powder can be used as a material with iron loss lower than that achieved by electrical steel sheet in the high-frequency range (several hundred Hz to 1kHz or above).

## References

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