Microstructural Evaluation of Nd-Fe-B Jet-Milled Powders

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Nd-Fe-B sintered magnets have high energy products and are used for various applications. Motors of hybrid vehicles (HEVs) are one of the major applications. For usage of HEVs, Dy is added to Nd-Fe-B magnets to maintain coercivity at high temperature environment. However, due to low natural resources of Dy, Dy-free or Dy-lean Nd-Fe-B sintered magnets are strongly required. To achieve high coercivity, it is necessary that microstructure of sintered magnets is consisted of both fine main phase particles and homogeneously distributed Nd-rich phases around the main phase. In this study, the microstructure of Nd-Fe-B jet-milled powders and the distribution of the Nd-rich phase were investigated. The distribution of the Nd-rich phase was evaluated by the ratio of grains which contain the Nd-rich phase. With decreasing size of the jet-milled Nd-Fe-B powder, the Nd-rich phase tends to aggregate and its distribution becomes inhomogeneous. The powder size of Nd-Fe-B jet-milled powder is much smaller than the average lamella interval of strip cast alloys.

Keywords: neodymium-iron-boron alloy, coercivity, jet-milled powder, microstructure, neodymium-rich phase distribution

(Received April 27, 2009; Accepted July 16, 2009; Published September 2, 2009)
additive rare earth elements, such as Pr and Tb. The powder size of the Nd-Fe-B powders was evaluated by the laser diffraction method. The lamella interval of SC alloys and the distribution of Nd-rich phase of jet-milled powders were observed and evaluated by Scanning Electron Microscope (SEM)—Energy Dispersive X-ray Spectroscopy (EDX). The lamella interval of SC alloy was evaluated by the point counting method. The average interval of Nd-rich phase was measured at various distances from the wheel surface. Details of evaluation methods were described in our previous paper. The distribution of Nd-rich phase was evaluated by the ratio of grains that contained the Nd-rich phase.

3. Results and Discussion

Figure 1(left) shows SEM backscattered electron images of taken from the cross section of SC alloy and Fig. 1(right) shows the relation between average lamella interval and distance from the wheel surface of SC alloys. In Fig. 1(left), bright and dark regions correspond to Nd-rich phase and main phase, respectively. This microstructure shows similar structure as previous reports. Figure 1(right) shows average interval of Nd-rich phase measured from SEM observation. The interval of lamella near the wheel surface is larger than that in the region of near the free surface. This feature has been also reported in our previous report. Actual microstructure contains submicron Nd$_2$Fe$_{14}$B grains as shown in our previous report. The average interval of lamella is evaluated to be 3.8 µm from SEM images.

Figure 2 shows the cumulative distribution ($Q_3$) of jet-milled Nd-Fe-B powders. The three kinds of jet-milled powders with different average diameter ($d_{50}$) were prepared. The average diameter ($d_{50}$) of powder 1, powder 2, and powder 3 were 1.6, 2.6, and 4.6 µm, respectively. The average diameter of powder 3 is larger than the average lamella interval of SC alloys. The average diameter of powder 1 is smaller than half of the average lamella interval.

Figure 3 shows the SEM-EDS mapping of Fe and Nd for jet-milled Nd-Fe-B powders. The $d_{50}$ is (a) 1.6, (b) 2.6, and (c) 4.6 µm.

The particle size of the main phase and the Nd-rich phase is discussed in the following. The SEM-EDS mapping of the powder 1, 2, and 3 are shown in Fig. 3(a), (b), and (c), respectively. The bright and gray regions show the region where Nd and Fe, respectively, are rich. Black regions correspond to the sample stage. The model of an ideal triple
junction is shown in Fig. 4(a). Assuming that the shape of Nd$_2$Fe$_{14}$B particles is circle, the unit shape of the ideal triple junction is shown as a triangle which consists of a part of three Nd$_2$Fe$_{14}$B particles and Nd-rich phase. The three gray triangles and black region at the center of the triple junction show Nd$_2$Fe$_{14}$B and the Nd-rich phase, respectively. In this ideal triple junction, the center region surrounding three Nd$_2$Fe$_{14}$B powder particles are assumed to be filled with the Nd-rich phase. The ratio of the area covered with three Nd$_2$Fe$_{14}$B (A$_1$) to the area covered with the Nd-rich phase (A$_2$) is calculated to be 9.8 to 1. The average area ratio evaluated from Fig. 3(a), (b), and (c) is plotted as a function of d$_{50}$. The result is shown in Fig. 4(b). Here, we define parameters A$_2'$ and A$_2'/A_1$ in order to evaluate amount of the Nd-rich phase in the triple junctions. The value of A$_2'$ equals 9.8 times of A$_2$. When the value of A$_2'/A_1$ equals to be 1, the area ratio becomes ideal state. Figure 4(b) shows that the value of A$_2'/A_1$ increases with decreasing d$_{50}$. The average lamella interval is shown as dashed line in Fig. 4(b). The A$_2'/A_1$ reaches about 1.4 when d$_{50}$ is 1.6 μm. This shows that the Nd-rich phase become larger than the ideal state. The fine powder contains relatively large Nd-rich particles.

The distribution of Nd-rich phase is also important. The ratio of grains which contain the Nd-rich phase (P) was evaluated as shown in Fig. 5. When the powder has good distribution, which means the Nd-rich phase is well-distributed, the ratio becomes close to 100%. On the other hands, when the powder has a bad distribution, the ratio decreases. The value of P decreases with decreasing the powder diameter. The Nd-rich phase is aggregated and its distribution becomes worse with decreasing d$_{50}$.

The simple model of jet-milling pulverization is also discussed. The schematic images of the model are shown in Fig. 6. It is assumed that its lamella interval of SC alloy (l) and the thickness of Nd-rich phase of the SC alloy (t) are constant. Moreover, the shape of the powder particles, which is pulverized by the jet-milling method, is assumed to be cubic. The length of the cube is set to be d. The values of t and l are set to be 0.5 μm and 3.8 μm, respectively. These values are chosen from the results of the SEM observation shown in Fig. 1. As the value of d, the average diameter of jet-milled powders is adopted. The probability (P) that the Nd-rich phase is observed in a particle is calculated by an equation as follows.

$$P = \frac{5}{6} \times \left(1 - \frac{((l-t)-d)^2}{(l-t)^2}\right) \times 100$$

Fig. 5 The average powder diameter dependence of the ratio of grains which contains the Nd-rich phase. Solid circles show the value which is evaluated from SEM-EDS mapping (shown in Fig. 3). The solid curve is guide for eyes. The dashed curve shows calculated value. The dashed line shows the average lamella interval of SC alloy.

Fig. 6 The schematic images of pulverization model of calculation which is shown in Fig. 5. The value of the lamella interval of SC alloy (l) and the thickness of Nd-rich phase of the SC alloy (t) is decided from SEM observation. The length of the cubic (d) is adopted the average diameter of jet-milled powders.
The relationships between the lamella interval of SC alloy and jet-milled Nd-Fe-B powder size were evaluated. The Nd-rich phase is aggregated and the distribution of Nd-rich phases becomes worse when the average diameter of Nd-Fe-B jet-milled powders decreases. It is caused by the fact that the powder size of Nd-Fe-B jet-milled powder is much smaller than the average lamella interval of strip cast alloys.

Acknowledgement

A part of this study was financially supported by a Rare Metal Substitute Materials Development Project (Development of technology for reducing dysprosium usage in a rare-earth magnet) from the METI in Japan. The authors also thank to Santoku Corporation for providing SC alloys.

REFERENCES