

## Fabrication of Deep-Sub-Millimeter-Thick Compacts Using Spark Plasma Sintering

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### Abstract

*Nd-Fe-B type powder was sintered using spark plasma sintering method. Fabricated compact sintered at the temperature of 700 °C, is found to be a composite magnet with Nd-Fe-Co-B and  $\alpha$ -Fe. The compact sintered at 700 °C shows slightly low coercivity and large remanent magnetization comparing to the compact sintered at 600 °C due to the formation of  $\alpha$ -Fe phase, resulting in the large maximum energy product. Maximum energy product tends to decrease with decreasing thickness of sintered compacts below 0.5 mm in thickness.*

**Keywords :** spark plasma sintering, maximum energy product, neodymium iron boron

### 1. Introduction

Since Nd-Fe-B was invented at 1983[1,2], magnetic properties for Nd-Fe-B were improved, and Nd-Fe-B type permanent magnets replaced Sm-Co magnets and are widely used for actuators, motor, MRI, sensors and so on. Larger maximum energy product is still required for permanent magnets to meet demand of the advanced modern technology. In order to increase maximum energy product, rare earth dopants are utilized for increase in coercivity although remanent magnetization decreases[3,4,5]. Thus 59.5 MGOe of maximum energy product was already achieved for sintered compacts[6] so far.

Spark plasma sintering (SPS) method which is able to sinter compact at relatively low temperature in a short sintering time, has been employed to fabricate compacts. Saito *et al.* showed that composite magnets with Nd-Fe-B of main phase were obtained[7,8] using SPS and melt-spun ribbon.

In this study, thin Nd-Fe-B type sintered compacts were fabricated by SPS with Nd-Fe-Co-B powder, and its crystallographic and magnetic properties were evaluated.

### 2. Experimental and Results

Coercivity and saturation magnetization of Nd-Fe-Co-B particle are 8700 Oe and 115 emu/g, respectively. Mean particle diameter is nominally 250  $\mu$ m. The powder was sintered at the temperature of 600, 700, 800 °C with 60 MPa of pressure for 5 minutes in carbon paper using SPS. Sintered compact was cut and polished to be  $5 \times 2 \times 1.5$  mm<sup>3</sup> in x, y, and z direction. Magnetic property was measured in y direction

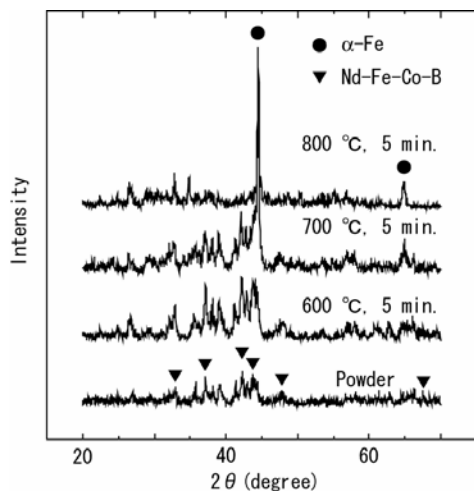
using vibrating sample magnetometer. Demagnetizing fields were not compensated for the estimation of magnetic properties.

Figure 1 shows XRD patterns for the sintered compacts and Nd-Fe-Co-B powder. XRD pattern for the compact sintered at 600 °C shows that main phase of Nd-Fe-B exists without the other markable phases. Soft magnetic  $\alpha$ -Fe phase, however, appears for the compact sintered at the temperature of 700 °C and above. This means that fabricated compact with the sintering temperature at 700°C is composite magnet.

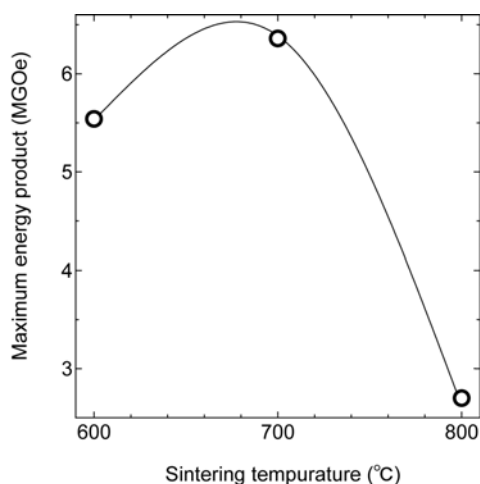
Maximum energy products for the sintered compact are shown as a function of sintering temperature in Fig. 2. Obtained maximum energy products are comparable to that fabricated by SPS with amorphous melt-spun ribbon[9]. As shown in Fig. 2, maximum energy product is maximal at around 700°C. The reason is considered that increase in saturation magnetization due to the formation of  $\alpha$ -Fe results in large remanent magnetic flux density, although coercivity slightly decreased comparing to the compact sintered at 600 °C. This may imply an obtained compact is composite magnet with strong exchange coupling between Nd-Fe-B phase and  $\alpha$ -Fe phase, called spring magnet.

Compacts sintered at 700 °C were ground to be thin, and maximum energy products were estimated as a function of the thickness as shown in Fig. 3. In the figure, maximum energy product is normalized by 12 MGOe which is maximum energy product for the 1-mm-thick sintered compact. Normalized maximum energy product decreases with decreasing the compacts thickness as shown in the figure. Mori *et al.* reported similar tendency in terms of sintering methods[10], in which grain size much smaller

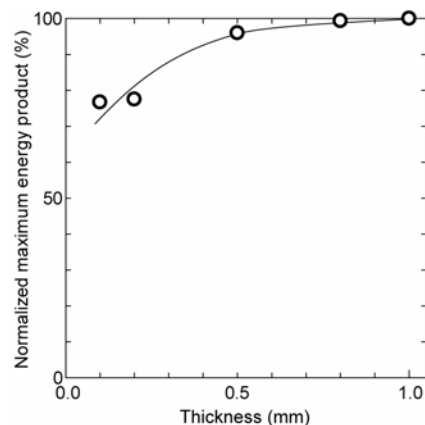
than the surface roughness for the compact is considered to be preferable to retain large maximum energy product for the compact ground to be sub-millimeter in thickness. Particle size of starting powder used in this study is nominally 250  $\mu\text{m}$ , which is much larger than surface roughness and compact thickness. Such large particle size might cause reduction in maximum energy products for thin compact. This implies that the reduction in normalized maximum product is possible to be suppressed using SPS method and Nd-Fe-Co-B particles smaller than that used in this research.



**Fig. 1.** XRD patterns for compacts sintered at 600, 700, 800 °C, and starting powder.



**Fig. 2.** Maximum energy product as a function of sintering temperature.



**Fig. 3** Thickness dependence of normalized maximum energy product for compact sintered at 700 °C.

### 3. Summary

Nd-Fe-B type sintered compacts were fabricated and its magnetic properties were estimated. Composite magnet with Nd-Fe-Co-B and  $\alpha\text{-Fe}$  was obtained for sintering at 700 °C using spark plasma sintering method. Fabrication of deep-submillimeter-thick sintered compacts with large maximum energy product is considered to be possible in the case that starting particle with small size and spark plasma sintering method are employed.

### 4. References

- [1] M. Sagawa, S. Fujimura, H. Yamamoto and Y. Matsuura: *J. Appl. Phys.*, Vol. 55 (1984) p. 2083.
- [2] J. J. Croat, J. F. Herbst, R. W. Lee and F. E. Pinkerton: *J. Appl. Phys.*, Vol. 55 (1984) p. 2079.
- [3] M. Sagawa, S. Fujimura, H. Yamamoto and K. Hiraga: *IEEE Trans. Magn.*, Vol. MAG-20 (1984), pp. 1584.
- [4] R. D. Brown and J. R. Cost: *IEEE Trans. Magn.*, Vol. 20 (1989), p. 3117.
- [5] D. N. Brown, M. Smith, B. M. Ma and P. Campbell: *IEEE Trans. Magn.*, Vol. 40 (2004), pp. 2895.
- [6] NEOMAX environmental sustainability report, (2005) p. 26.
- [7] H. Ono, N. Waki, M. Shimada, T. Sugiyama, A. Fujiki, H. Yamamoto and M. Tani: *IEEE Trans. Magn.*, Vol. 37 (2001) p. 2552.
- [8] T. Saito, T. Takeuchi and H. Kageyama: *J. Appl. Phys.*, Vol. 97(2005) p. 10H103.
- [9] H. Ono, T. Tayu, N. Waki, T. Sugiyama, M. Shimada, M. Kanou, A. Fijiki, H. Yamamoto, K. Takasugi and M. Tani: *J. Appl. Phys.*, Vol. 93(2003) p. 4060.
- [10] K. Mori, R. Nakayama and K. Morimoto: *J. Jpn. Soc. Powder and Powder Metallurgy*, Vol. 52, (2005) p.700.