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# Proven technologies on high-performance Nd-Fe-B sintered magnets

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#### Abstract

Recently, we have newly developed the  $460 \text{ kJ/m}^3$  magnet.  $B_r$  value of this magnet shows 1.533 T that corresponds to almost 96% of the theoretical value of Nd<sub>2</sub>Fe<sub>14</sub>B compound. And the  $H_{cJ}$  value of this magnet is kept at 784 kA/m. One of the advanced technologies leading to this success is an improvement of the strip-casting alloy that has finer dendrites with scattered R-rich phase. Even though the rare-earth content of the alloy is only 12.3 at.% that is close to the stoichiometric 2–14–1 phase, the new alloy has finer dendrites with keeping an excellent orientation without the primary Fe phase. Furthermore, we have succeeded in stabilizing powder after milling with using some lubricants and to control the microstructure to be fine and uniform with keeping higher alignment of the 2–14–1 grains through sintering. Also, we have been investigating how to improve thermal stability and the corrosion resistance not only by treating surface coating but also by improving microstructure of magnets. As a result, we have been mass-producing high-performance magnets having 2.0 MA/m of  $H_{cJ}$  for motor applications. In this paper, market trend of NdFeB sintered magnets is introduced and the latest processing technologies to produce higher energy Nd–Fe–B sintered magnets are described.

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#### 1. Introduction

NdFeB sintered magnets [1] have been remarkably expanding in the global markets. The trend of the production weight of Nd-Fe-B magnets in Japan is shown in Fig. 1, in which the share of applications of Nd-Fe-B sintered magnets is estimated. Total weight of the 2003 production of Nd-Fe-B sintered magnets probably reaches 5250 metric tonnes. Although the 2001 production shrunk because of economic recession in the field of information technology, the 2003 production has recovered to the expected usual level. Although the principal application has been VCM, the ratio of VCM applications divided by the total NdFeB production has been declining year by year with improving magnetic properties of NdFeB sintered magnet because of downsizing of computer. However, further extents are shown not only in computer devices but also in the mobile phone, audio and video systems, and navigation systems of driving supports

\* Corresponding author. *E-mail address:* KANEKO.Y@neomax.co.jp (Y. Kaneko). and so on. In these applications, high-performance magnets having  $400 \text{ kJ/m}^3$  of (BH)<sub>max</sub> with keeping thermal resistance over 85 °C are strongly required.

Recently, motor applications by using NdFeB sintered magnets are rapidly increasing as shown in Fig. 2, because improvements in motor efficiency are urgent to reduce  $CO_2$  exhaust gas. Market demands for these magnets are focused on (1) higher energy products, (2) higher thermal stability and reliability and (3) sales price reduction. In order to solve these requirements, it is very important to investigate how to realize an excellent alignment of 2–14–1 grains and how to increase coercive force with a minimum content of Dy through the powder metallurgical process.

# 2. Processing technologies of NdFeB sintered magnets

NdFeB sintered magnets are mass-produced through a powder metallurgical process as shown in Fig. 3. Rare-earth elements such as Nd, Pr, Dy and Fe, Fe–B are melted in a

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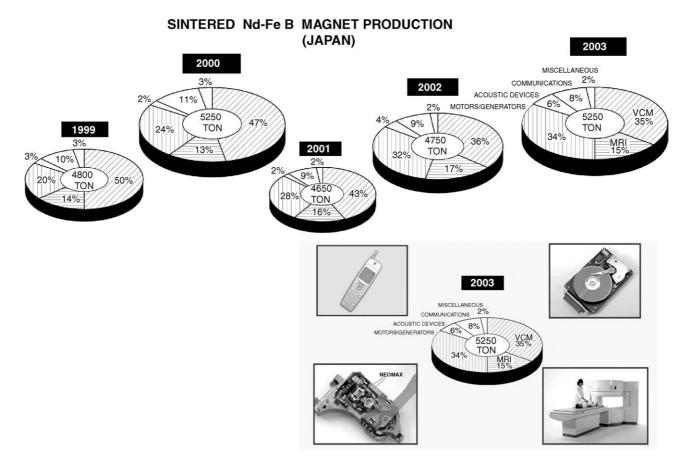


Fig. 1. Trend of production amounts and application fields on NdFeB sintered magnets in Japan.

vacuum furnace, then, coarse milling is done by the hydrogen treatment. After that, fine milling is done by using a Jet-mill to approximately  $3 \mu m$  of fine powders, then fine powders are compacted in a magnetic field. The compacted body is sintered and heat-treated in a vacuum condition with Ar gas. Finally, a sintered body is ground and surface-treated.

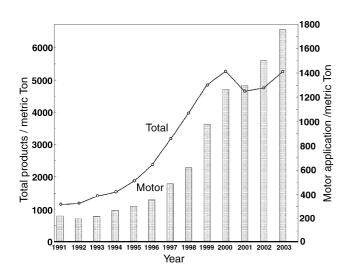


Fig. 2. Expanding of production amounts of NdFeB sintered magnets for motor use.

Recently, the strip-casting, which is one of the major turning points to improve the microstructure of NdFeB sintered magnet, has been developed [2]. Fig. 4 shows a comparison of the microstructure between a strip-casting alloy and a conventional book-molded alloy. A strip-casting alloy has finer dendrites with scattered rare-earth rich phase, in which the rare-earth rich phase easily generates a liquid phase because of the uniformly surrounding 2–14–1 dendrites. Furthermore, as the Jet-milling process improved almost twice efficiency in comparison with the conventional method, the resultant particle size distribution is also easily controlled to be fine with an increase of the productivity [3].

#### 3. Toward the highest performance magnet

The theoretical value of the maximum energy product of NdFeB magnets is calculated to be  $512 \text{ kJ/m}^3$  (64 MGOe) [4]. Many efforts to achieve higher energy product have been done as shown in Fig. 5. The key technologies to improve magnetic properties are based on these issues: (1) compositional control, (2) densification through sintering, (3) controlling of grain size, (4) alignment of the 2–14–1 grains [5,6].

In order to increase the volume fraction of 2-14-1 phase, it is very important to control the composition close to the stoichiometric 2-14-1 phase with keeping a necessary volume

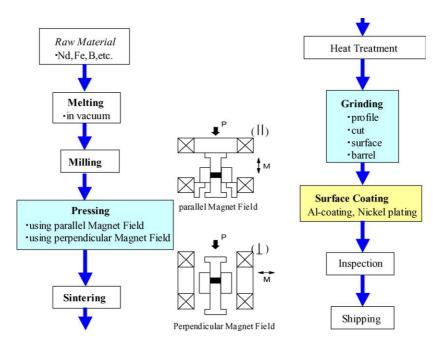


Fig. 3. Typical manufacturing process of NdFeB sintered magnets.

of the Nd-rich phase. However, as the rare-earth elements are easily oxidized on contact with air, essential content of rare-earth element to generate the rare-earth rich phase is consumed, then magnetic properties are much damaged due to the change in the densification behavior.

So, we have been investigating to improve the microstructure of the strip-casting alloy as shown in Fig. 6. Even though the rare-earth content of the alloy is only 12.3 at.% that is close to the stoichiometric 2-14-1 phase, the newly developed alloy has finer dendrites with keeping an excellent orientation without the primary Fe phase [7]. As a result, we can improve not only in compositional and microstructural uniformity of magnets, but also in size and morphology of 2-14-1 grains after sintering.

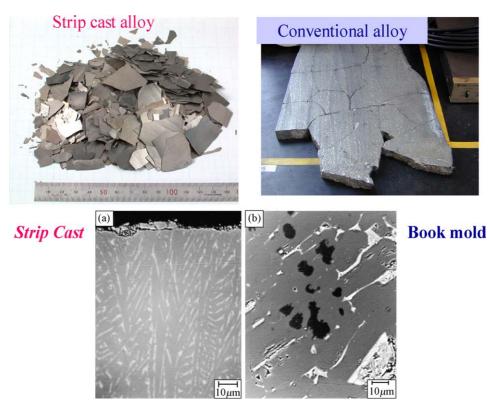


Fig. 4. Comparison with the appearance and the microstructure of NdFeB alloys between the strip-casting and the book mold.

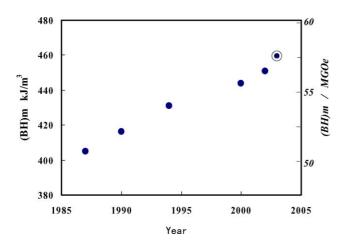
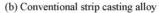


Fig. 5. Progress of developing high energy NdFeB sintered magnets in laboratory level.

(a) Newly developed alloy

Recently, we have succeeded in developing the  $460 \text{ kJ/m}^3$  magnets [8]. The demagnetization curve of this magnet is shown in Fig. 7. The  $B_r$  value reaches 1.533 T that corresponds to almost 96% of the theoretical value of the Nd<sub>2</sub>Fe<sub>14</sub>B compound. The  $H_{cJ}$  value is kept at 784 kA/m and the (BH)<sub>max</sub> value of this magnet shows 460 kJ/m<sup>3</sup>.

The technological advancement of this magnet is analyzed in Table 1 in comparison with the mass-produced 400 kJ/m<sup>3</sup> magnet and target property of 480 kJ/m<sup>3</sup>. In case of this record, the volume fraction of 2–14–1 phase is improved to be 97.8%, then the degree of alignment of 2–14–1 grains is enhanced to be 98.5%. So,  $B_r$  value calculates 1.53 T that corresponds with the trial data. Consequently, the estimated limitation of  $B_r$  seems to be 1.55 T, which can be achieved by further improvements of powder making and aligning method.



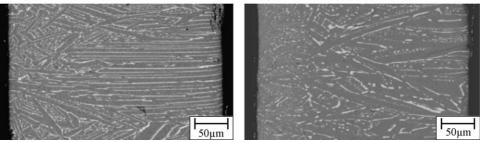


Fig. 6. Microstructural comparison of strip-casting alloys prepared by (a) the newly developed method and (b) the conventional method.

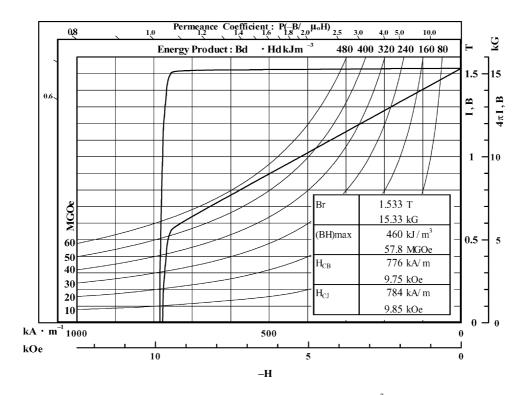


Fig. 7. Demagnetization curve of the newly developed 460 kJ/m<sup>3</sup> magnet.

Table 1

	Composition (at.%)				Volume fraction of 2–14–1 phase (%)	Density (%)	Alignment (%)	<i>B</i> <sub>r</sub> (T)
480 kJ/m <sup>3</sup> (target)	12.3	5.7	Bal.	0.4	98.6	99.0	99.3	1.55
480 kJ/m <sup>3</sup> (this record)	12.5	5.7	Bal.	0.6	97.8	99.0	98.5	1.53
400 kJ/m <sup>3</sup> (mass-production)	13.7	5.7	Bal.	1.8	93.8	99.0	96.0	1.43

Analytical results of technological advancements on processing to improve residual flux density  $(B_r)$  of NdFeB sintered magnets

#### 4. High grades of NdFeB sintered magnets

In order to avoid increase in the material cost of NdFeB sintered magnets, we have investigated technologies to improve coercive force with the minimal content of Dy. In particular, the resource of Dy becomes a significant problem as the motor applications of Nd–Fe–B magnets grow, because the amount of Dy deposits is estimated to be about one-tenth of Nd deposits. However, as the mechanism of the coercive force of NdFeB sintered magnet and the role of grain boundaries is not understood completely,  $H_{cJ}$  value of NdFeB magnets is still less than the theoretical estimation. Therefore, it is very important to control not only the grain size and morphology, but also grain boundary phase [9].

As one of the solutions is to reduce impurities, such as oxygen, we have developed a new process that decreases the oxygen content below 2000 ppm in the final product by the way to stabilize powders after milling by using some lubricants. As a result, we can improve the coercive force with minimum using of Dy. The material distribution of NdFeB sintered magnets is shown in Fig. 8. Already, high-performance magnets having more than  $400 \text{ kJ/m}^3$  are put into practical applications, and then AH series are applied for motor applications widely.

On the other hand, in order to apply NdFeB sintered magnets for motors and precious devices, improvements in both thermal stability and corrosion resistance with keeping the high flux density become essential. The features of the proven surface coating technologies are summarized in Table 2.

The metallic coatings, such as Al and Ni, show excellent ability to prevent the corrosion of this magnet from occurring under severe conditions of high temperature and humidity even for a long-term operation. Moreover, the Al deposition has a uniform thickness in all portion of magnet and also a superior adhesive strength with any adhesion due to the wet ability and the roughness of the Al deposited surface.

Recently, in order to meet customer's requirements on cutting cost of the surface coating, we have built some variations of Al-coating in accordance with the performance of devices. For example, the new Al-coating named EAC does not use chromic acid as a final treatment for Al film to meet environmental issue, then, the thin type Al which has less than

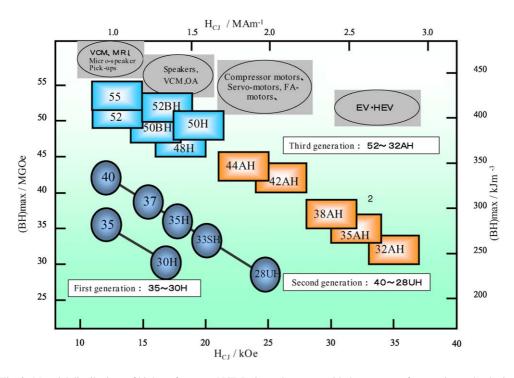


Fig. 8. Material distributions of high-performance NdFeB sintered magnets with the progress of processing technologies.

Table 2 Features of typical surface coating technologies for NdFeB sintered magnets

Coating	Contents	Thickness (µm)	Features	Applications
Aluminum	Aluminum-chromate	7–19	Excellent adhesive strength	Motors, generators, sensors pick-ups for
			Uniform thickness of Al film	CD, DVD, automobile applications
	Eco-aluminum	7–19	Non-Chromic acid	
	Thin type aluminum	$\sim 5$	Thin type	Speakers, etc.
	Pure type aluminum	$\sim 5$	Compressors usage	
Nickel	Direct electro Ni plating	10-20	Ni only electro plating	VCM
	High corrosion resistance	15–25	System electro plating with some special elements	Motors, sensors
	Electroless Ni plating	$\sim 5$	Electroless plating	Two-pole cylindrical motors
Epoxy	Electro deposition	20-30	High electrical insulating	Pick-ups for CD
	Spray coating	10-20	Uniform thickness of film	Rodless cylinder
Simple treatment	V coating	~3	Simple chemical treatment	Motors
	M treatment	$\sim 2$	Simple stabilized treatment	
Special treatment	TiN coating	5-10	Available in high vac. $(10^{-10} \text{ Pa})$	Undulator
-	Polyimide coating	5-10	High electrical insulating	Pick-ups for CD

 $5 \,\mu\text{m}$  of thickness is widely applied for IT devices, and compressor motors of the air-conditioner. On the other hand, a simple chemical treatment for NdFeB sintered magnets has attracted a great deal of attention for IPM motors, also inorganic coating shows excellent protection from the salt splay condition.

## 5. Summary

As Nd–Fe–B sintered magnets have been improved not only in magnetic properties but also in cost-performance, motor applications of Nd–Fe–B sintered magnets have been expanding in Japan year by year. The aim of using highperformance magnets is to achieve electric power saving, low emission, high over all performance and so on. Therefore, fundamental investigations and improvements of the processing technologies which control microstructure and sintering behavior are urgent in order to contribute to further development of new applications within the minimal cost condition. Toward the further expanding of NdFeB sintered magnets, we are challenging to achieve the highest performance magnets.

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