Coercivity Mechanism of Ga-Doped Nd-Fe-B Sintered Magnets

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The alignment dependence and angular dependence of coercivity (ALDC and ANDC, respectively) of aligned and isotropically aligned Ga-doped Nd14.79Ga0.57B5.24Co1.11Fe74sintered magnets were analyzed, and the results were compared with those of ferrite magnets, which were reported in our previous paper. The coercivity of Ga-doped Nd-Fe-B magnets decreases as alignment improves. The ALDC of this magnet is similar to that of ferrite magnets. The angle of the magnetization-reversal area (AMRA) of the aligned Ga-doped magnets has at coercivity an angular tilt of 41.4°, which is wider than that of the ferrite magnets. The ANDC of Ga-doped Nd-Fe-B sintered magnets displays similar behavior with that of ferrite magnets but is slightly lower than that of ferrite magnets, which is expected from AMRA of Ga-doped Nd-Fe-B sintered magnets. These results support our proposed coercivity mechanism; specifically, the magnetic domain walls are pinned at grains with tilted easy magnetization directions or c-axis tilted from the easy magnetization of the magnet. When the magnetic domain walls are de-pinned from the pinning sites, they jump through several grains, thereby determining the coercivity.

Index Terms—Coercivity, coercivity mechanism, magnet, Nd-Fe-B magnet.

I. INTRODUCTION

D ISCOVERED in 1983 [1], Nd-Fe-B sintered magnets have excellent remanence and coercivity properties. They are mainly found in brush-less motors used in industrial robots, compressor motors for air conditioners, traction motors in electric and hybrid-electric vehicles, and electric power steering motors. Applications using Nd-Fe-B sintered magnets are expanding rapidly to satisfy demands for energy savings and gas emission reductions.

With this expanding market for motors using Nd-Fe-B magnets, the need for improvements in magnetic properties, especially coercivity, is strongly increasing because the applications involve high-temperature environments. To improve coercivity, heavy rare-earth elements such as Dysprosium (Dy) and Terbium (Tb) are conventionally used. Recently, there has been an increased awareness that these heavy rare-earth metals are a limited resource and it is very important to discover new methods to replace these elements while achieving higher coercivities. To solve these problems, two main approaches were proposed and have already been introduced in manufacturing. One involves grain boundary diffusion; the other exploits the Ga-doped Nd-Fe-B sintered magnets [2], [3]. Both achieve high coercivities and thus provide a solution to reduce heavy rare-earth metal used in motor applications. Of importance, though, is the need to elucidate the coercivity mechanism and to improve the coercivity of materials.

To date, several different mechanisms have been proposed and are still in discussion [4]–[7]. They are mainly nucleation mechanisms and focus on reversing single Nd2Fe14B grains. The discussions mainly cover two themes. One is the coherent rotation of magnetization and the other is the penetration of the magnetic domain wall in a grain after nucleation of the reverse magnetization by thermal fluctuation. They originate from the magnetic properties of the grain boundary phase surrounded by Nd2Fe14B grains [8]. From the Nd-Fe-B ternary phase diagram, this grain boundary is considered to be a nonmagnetic or less-magnetic phase in the stable state, and every Nd2Fe14B grain is magnetically isolated or has negligibly small interaction with other grains [9]. In this condition, a natural idea is that the coercivity of these materials is determined by the nucleation of a reverse magnetization. However, this picture of the boundary phase was changed recently [10]. The iron content in the boundary phase obtained with an atom probe is higher than the expected content given by the ternary phase diagram. When the boundary phase is a ferromagnetic material, the nucleation model, which is believed to be behind the magnetization reversal in a grain, needs to be changed. From this aspect, we need to carefully consider again what happens in an entire magnet comprising 1010 grains per cubic centimeter when the average grain size in Nd-Fe-B sintered magnets is 5 μm.

The results of magnetization measurements, such as the alignment dependence of coercivity (ALDC), the angular dependence of coercivity (ANDC), and the recoil properties of magnetization of Nd-Fe-B sintered magnets and ferrite magnets, show that the pinning mechanism of the magnetic domain wall is related to magnetic reversals [11]–[18]. The reversals not only occur in one grain but also occur in several grains simultaneously in aligned magnets. From the ALDC, coercivity decreases as the alignment (α) improves, that is, α = Br/Jr with Br and Jr denoting the remanence and saturation magnetization [19]–[22]. From the coherent...
rotation model, coercivity is expected to increase as alignment improves [11]. The modified coherent model, including a secondary term associated with the anisotropy energy ($K_2$), [4], also predicts an increase in coercivity. The experimental data of the ALDC show that a coherent rotation does not happen in actual magnets. From the recoil properties of Nd-Fe-B sintered magnets, a small demagnetization, after demagnetization caused by surface degradation in a low demagnetization field, occurs linearly for any demagnetization field until coercivity [12]. This phenomenon shows that a number of reversed grains or magnetic domains already exist in the magnets before the magnetic field reaches its coercivity value. These results show that some skepticism is required concerning the idea that thermal fluctuation causes the nucleation of magnetization reversals and determines coercivity [6]. It is reasonable to consider that the accumulated reversed magnetic domain walls or reversed grains generated in a lower magnetic field than that for coercivity prompt reversals in other grains when the demagnetization field is close to coercivity and determine their coercivity when the magnetic field reaches the coercive field.

The ALDC results show that, for Nd-Fe-B and Nd-Dy-Fe-B sintered magnets, the coercivities decrease for isotropically aligned magnets as alignment improves [21], [22]. The phenomenon occurs not only in Nd-(Dy)-Fe-B sintered magnets; ferrite magnets also show a similar trend in coercivity. In particular, the coercivity decreases steeply in high alignment areas ($\alpha > 0.95$), the decrease creating trouble in many applications. To reduce the size, every application requires high $B_r$, but the substitution of Nd by Dy or the addition of Ga to Nd-Fe-B magnets causes a reduction in $B_r$. To avoid this reduction in coercivity within a highly aligned area ($\alpha > 0.95$), an excess of Dy is required but that reduces $B_r$. The ALDC results show that this phenomenon derives from the coercivity mechanism of Nd-(Dy)-Fe-B sintered magnets and ferrite magnets.

In these magnets, we defined the coercivity decrease ratio as [11], [23]

$$\beta = \frac{(H_{c\text{J}} - H_{\text{c Isotropy}})}{H_{\text{c Isotropy}}} \times 100$$ (1)

where $H_{c\text{J}}$ is the coercivity of the aligned magnet and $H_{\text{c Isotropy}}$ is the coercivity of the isotropically aligned magnet. The extrapolated line of $\beta$ over $\alpha > 0.95$ was found to reach $-30\%$ that of isotropically aligned magnets at perfect alignment [21], [22]. We also found that the ALDC results of Nd-Fe-B, Nd-Dy-Fe-B, and ferrite magnets have different ALDC. Another question generated from ALDC is whether its entire curve for these magnets has been explained by the Kondorskii law or the magnetic domain wall motion (MDWM) using their alignment distribution functions ($P(\theta)$). Here, $\theta$ is the angle of the tilted grains from the easy magnetization direction of the magnet. The results obtained from electron backscattered diffraction (EBSD) data show that $P(\theta)$ is close to a Gaussian distribution

$$P(\theta) \propto \frac{1}{\sqrt{2\pi}\sigma} \exp\left(-\frac{\theta^2}{2\sigma^2}\right)$$ (2)

where $\sigma$ is its standard deviation. It was found that although Nd-Fe-B, Nd-Dy-Fe-B, and ferrite magnets had different ALDC, we were unable to identify any differences between alignment distributions in these magnets with similar alignment [12], [23].

For poly-crystalline magnets, the sum of the components of the magnetization of every grain determines $B_r$ and is calculated using $P(\theta)$. From this result, $\alpha$ is obtained

$$\alpha = \frac{B_r}{J_s} = \frac{1}{2} \int_{0}^{\pi/2} P(\theta) \sin 2\theta d\theta.$$ (3)

In magnetization reversals, a reverse magnetic field ($-H$) is applied in the opposite direction to the magnetization direction. Every grain experiences a magnetic field $-H \cos \theta$ to their crystalline easy magnetization direction (c-axis direction if every grain is a single crystal). We then assume that all grains isolate any one grain from the interaction and each individually reverses its magnetization when the reverse magnetic field reaches its coercivity value, $H \cos \theta = H_{c\text{J Grain}}$, where $H_{c\text{J Grain}}$ denotes the coercivity of the grain or the average coercivity of grains. Using (3), the coercivity $H_{c\text{J}}$ of a magnet is obtained from

$$J = \frac{1}{2} J_s \left(\int_{\theta_1}^{\pi/2} P(\theta) \sin 2\theta d\theta - \int_{0}^{\theta_1} P(\theta) \sin 2\theta d\theta\right) = 0.$$ (4)

The second term represents the reversed magnetization area, and $\theta_1$ denotes the angle of the magnetization reversal area (AMRA). With $\theta_1$, $\beta$ follows:

$$\beta = \left(-\frac{1}{\sqrt{2\cos \theta_1}} - 1\right) \times 100.$$ (5)

In [12] and [24], the calculated $\alpha$ and $\beta$ values obtained using (3) and (5) were compared with the experimental data. On the other hand, the $\alpha$ values agreed well, the $\beta$ values deviated considerably. This discrepancy along with the ALDC suggest that the coercivity of these magnets is determined by the MDWM, but that grains in these magnets do not reverse independently as assumed under the MDWM or the $1/\cos \theta$ law. The magnetic domain walls are expected to be pinned at tilted grains, and when these domains de-pin from pinning grains, a cluster of grains reverse simultaneously.

It is also expected that the tilt angle of the grains, that is, the included angle between the c-axis of a grain and the easy magnetization direction of the magnet, at which the magnetic domain walls are pinned, are distinct for each material and hence may give rise to differences in the ALDC for Nd-Fe-B, Nd-Dy-Fe-B, and ferrite magnets. In [13] and [14], the temperature dependence of the ALDC and the ANDC for Nd-Fe-B, Nd-Dy-Fe-B, and ferrite magnets were investigated. The experimental results showed that both ALDC and ANDC results exhibited a temperature dependence that was co-related. With increasing temperature, both the ALDC and ANDC followed closely the trend expected from the MDWM, strongly suggesting that they are different aspects originating from the coercivity mechanism of these magnets. The belief is that, if the ALDC and ANDC have a common
connection, a more concrete picture of the coercivity mechanism would follow.

Substituting the experimental values of $\beta$ into (5) yields a $\theta_1$ trend obtained from the experiment that can be compared with those from calculations. The alignment dependence of $\theta_1$ derived from $\beta s$ for Nd-Fe-B, Nd-Dy-Fe-B, and ferrite magnets and the calculated $\theta_1$ values show that the two sets of $\theta_1$ values diverge given the same alignments and produce the same angle as that for low alignment magnets [17], [18]. These results indicate that the magnetization reversal of the aligned magnet occurs as for a magnet with low alignment. Moreover, they also support our previous hypothesis that the magnetic domain walls are pinned at grains tilted from the easy aligned magnet occurs as for a magnet with low alignment. These results indicate that the magnetization reversal of the ferrite magnets and the calculated $P(\theta)$ need for this assumption because $P(\theta)$ decreases from 0 to 45° − $\Delta h$, where $\Delta h$ goes to zero [18], [25]. When $\Delta h$ is zero, the coercivity is zero at 90°. The ANDC of the isotropically aligned magnet of Nd-Fe-B and that of the ferrite magnets agree well with calculations up to 60° [18], [25].

From this analysis, another question arises, specifically, what is the relationship between the ALDC and ANDC for Nd-(Dy)-Fe-B sintered magnets and ferrite magnets? Nd-(Dy)-Fe-B sintered magnets and ferrite magnets exhibit different behaviors in magnetization, anisotropic energy, and exchange energy. Nd-(Dy)-Fe-B magnets are ferromagnetic materials but ferrite magnets are ferrimagnetic materials. One wonders whether this method of analysis used in [17], [18], and [25] is valid for these different materials. In answer, it is better to find similar properties of the ALDC and ANDC for both Nd-Fe-B sintered magnets and the ferrite magnets and apply the same method of analysis in their assessment. In addition, one would need to accumulate the validated data to apply this method to different materials. The candidate material we chose and examined was Ga-doped Nd-Fe-B sintered magnets and compared their ALDC and ANDC with those of ferrite magnets given in [18].

II. EXPERIMENT

The Ga-doped Nd-Fe-B sintered magnets of composition Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{57}$ with $\alpha = 0.5$, 0.942, 0.945 were used for this experiment. These magnets were made using the powder metallurgical method, which has been described elsewhere [11], [12]. Magnets of different alignments were made by applying different alignment fields during pressing; the isotropically aligned magnet was made in the absence of an alignment field. To measure their magnetic properties, $J_s$, $B_r$, and $H_{cl}$, the magnets were cut into 7 mm cube ($7 \times 7 \times 7$ mm) specimens and measured using a pulse field magnetometer (TPM-2-08S25VT-C, Toei Industry Co., Ltd., Tokyo, Japan). An 8-T magnetic field was applied to obtain a saturation magnetization. For measurements of the ANDC, cylindrical magnets of diameter 4 mm and length 4 mm were used having an axially transverse magnetization direction. $H_{cl}$ of ANDC of $\alpha = 0.945$ and 0.5 specimens were measured using a vibration sample magnetometer equipped with a high-temperature super-conducting magnet (VSM-5HSC, Toei Industry Co., Ltd., Tokyo, Japan) with a 5-T maximum magnetic field. Their values were compared with those of ferrite magnets. For scanning electron microscopy (SEM), backscattered electron imaging (BEI), and energy-dispersive X-ray (EDX) analysis, we used a field-emission scanning electron microscope (JSM-6330FS, JOEL Ltd., Tokyo, Japan).

III. RESULTS AND DISCUSSION

From BEI and EDX images of Nd, Fe, and Ga of Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{57}$ with $\alpha = 0.945$ (Fig. 1), several different grain boundary phases are identifiable such as the Nd$_6$(Fe,Ga)$_{14}$ and the Nd-rich phases surrounding the Nd$_2$Fe$_{14}$B grains. The phases of this magnet have already been described elsewhere [26]. Before performing measurements of the ALDC and ANDC, a check of $B_r$ of the specimens is important. In our previous paper on ferrite magnets [18], we reported that the ANDC is significantly changed by the alignment, especially in low alignment areas. From (3), $B_r$ of an isotropically alignment magnet is expected to be half the saturation magnetization ($\alpha = 1$). Fig. 2 shows $B_r$ and $J_s$ of the specimens with various
alignment. Note that, for the isotropically aligned magnet, $B_r$ is 0.719 T and is almost half the saturation magnetization (1.439 T), which is expected from (3). The coercivities of $\alpha = 0.5$, 0.942, and 0.945 specimens were 1693, 1604, and 1595 kA/m, respectively, indicating a decrease in coercivity as the alignment improves. In high alignment magnets, the coercivity is observed to change clearly even with slight adjustments in alignment. $\beta$ for Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{bal}$ (Fig. 3) indicates that the coercivity changes in Ga-doped Nd-Fe-B sintered magnets are slower than those of Nd-Fe-B and Nd-Dy-Fe-B sintered magnets [17] and is similar with that of ferrite magnets [18]. From the ALDC results of Ga-doped Nd-Fe-B magnets, the ANDC of Ga-doped Nd-Fe-B is expected to have a similar behavior to that of ferrite magnets.

Fig. 4 shows the AMRA of Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{bal}$ obtained from (5) and compare with the calculated AMRA from (4). It was found that the AMRA of Ga-doped Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{bal}$ is 41.4°, which is slightly larger than 40.7° of SrO$_6$Fe$_2$O$_3$ [18]. In [17] and [18], we pointed out that the AMRA of these magnets is the same as that for a low alignment magnet calculated from (4). Moreover, using a larger $\sigma$ or a broader $P(\theta)$, obtained from (2) and (4), than those of actual aligned magnets, ANDCs of these magnets could be qualitatively explained. If a similar analysis is applied to Ga-doped Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{bal}$, the ANDC of Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{bal}$ is expected to be lower than that of SrO$_6$Fe$_2$O$_3$.

Fig. 5 shows the coercivity of both Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{bal}$ and SrO$_6$Fe$_2$O$_3$ [18] measured at various angles. The coercivity of the former is nearly 1600 kA/m, which is almost four times larger than that of the latter. Nevertheless, when normalized by the coercivity measured at 0°, the ANDC of the two look similar (Fig. 6). ANDC of Ga-doped Nd-Fe-B sintered magnet is slightly lower than that of ferrite magnets; this is to be expected from the AMRA of these magnets (Fig. 4). This result strongly suggests that the same analysis applied to SrO$_6$Fe$_2$O$_3$ [18] is also valid for Nd$_{14.79}$Ga$_{0.57}$B$_{5.24}$Co$_{1.11}$Fe$_{bal}$.

These results also support the following coercivity mechanism of Nd-Fe-B sintered magnets and ferrite magnets mentioned in [12]–[18]. Grains with reversed magnetization or magnetic domain walls are generated in lower magnetic fields than the coercivity. The magnetic domain walls can be pinned at grains with a tilted c-axis (i.e., the easy magnetization direction of the grain) from the easy magnetization...
were found to decrease as alignment improves. Even in high alignment areas, $\beta$ is smaller than that of Nd$_{14.2}$B$_{6.2}$Co$_{1.0}$Fe$_{bal}$ and Nd$_{14.2}$Dy$_{0.3}$B$_{6.2}$Co$_{1.0}$Fe$_{bal}$ [17] and is similar to that of SrO$_6$Fe$_2$O$_3$ [18]. The AMRA of Nd$_{14.79}$Ga$_{0.7}$B$_{2.24}$Co$_{1.1}$Fe$_{bal}$ with $\alpha = 0.945$ is $41.4^\circ$, which is larger than $30^\circ$ of Nd$_{14.2}$B$_{6.2}$Co$_{1.0}$Fe$_{bal}$ [17] and $36^\circ$ of Nd$_{14.2}$Dy$_{0.3}$B$_{6.2}$Co$_{1.0}$Fe$_{bal}$ [17], and is similar but slightly larger than $40.7^\circ$ of SrO$_6$Fe$_2$O$_3$ [18]. As expected from these ALDC results, the ANDC of Nd$_{14.79}$Ga$_{0.7}$B$_{2.24}$Co$_{1.1}$Fe$_{bal}$ was found to show similar behavior with that of SrO$_6$Fe$_2$O$_3$ reported in [18]. The ANDC of Nd$_{14.79}$Ga$_{0.7}$B$_{2.24}$Co$_{1.1}$Fe$_{bal}$ decreases as the angle of the demagnetization field increases from $0^\circ$ to $40^\circ$; thereafter, coercivity begins to increase, which is supposed to be the behavior expected from the Stoner–Wohlfarth model or coherent rotation of the magnetization. However, the ALDC shows that this behavior is explained by the pinning and de-pinning of magnetic domain walls. Even in Ga-doped Nd-Fe-B sintered magnets, which were reported to better isolate grains by thick and non-magnetic grain boundaries [26], the results obtained from the ALDC show that the coercivity of these magnets is still determined by the MDWM. The ANDCs of Ga-doped Nd-Fe-B sintered magnets behave similarly albeit at a slightly lower level than that of SrO$_6$Fe$_2$O$_3$ [18]. The ANDC results of isotropically aligned specimens display a similar behavior to that of Nd$_{14.79}$Ga$_{0.7}$B$_{2.24}$Co$_{1.1}$Fe$_{bal}$, Nd$_{15}$B$_6$Co$_{1}$Fe$_{bal}$, and SrO$_6$Fe$_2$O$_3$ [18], [25] and agree well with the calculation up to an angle of $60^\circ$.

From our previous and present studies, we found that the ALDC of the aligned Nd-Fe-B sintered magnets including Ga-doped Nd-Fe-B sintered magnets and ferrite magnets varies with composition and temperature. In addition, the ANDCs vary with composition, alignment, and temperature. The difference in AMRA obtained from the ALDC relates to the number of reverse grains, which gives a secondary effect in the jump of magnetic domain walls after de-pinning. This means that a wider or larger AMRA has more grains related to the simultaneous magnetization reversal during jumps in magnetic domain walls. The ALDC and ANDC were shown to be correlated and that they were another aspect originating from the coercivity mechanism itself. However, the isotopically aligned magnets behave similarly and agree well with the calculation results. These results support our proposed coercivity mechanism; specifically, in aligned magnets, magnetic domain walls are pinned at grains with tilted c-axis or easy magnetization direction of the grain from the easy magnetization direction of the magnet, and when the magnetic domain walls are de-pinned from these pinning sites, a number of grains reverse simultaneously causing the magnetic domain wall to jump. Brought about by this magnetization reversal mechanism, the aligned magnets behave like low alignment magnets. However, for isotopically aligned magnets, either all grains or a small number of grains reverse independently in accordance with the $1/cos\theta$ law.

IV. CONCLUSION

From ALDC experimental data of Ga-doped Nd$_{14.79}$Ga$_{0.57}$B$_{2.24}$Co$_{1.1}$Fe$_{bal}$ sintered magnets, their coercivities
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