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<tr>
<td>Citation</td>
<td>IEEE Transactions on Magnetics, 44(11), pp.4199-4201; 2008</td>
</tr>
<tr>
<td>Issue Date</td>
<td>2008-11</td>
</tr>
<tr>
<td>URL</td>
<td><a href="http://hdl.handle.net/10069/23905">http://hdl.handle.net/10069/23905</a></td>
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Improvement in Magnetic Properties of PLD-Made Nd-Fe-B Thick Film Magnets

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Coercivity of pulsed laser deposition (PLD)-fabricated Nd-Fe-B thick film magnets were enhanced by synergistic use of an additive (Zr, Nb or Ga) and an adoption of pulse annealing as a high-speed crystallization method. The role of the additives and the pulse annealing mainly work on the increase of coercivity without the deterioration of remanence and \((BH)_{\text{max}}\). For example, after pulse annealing of an as-deposited film prepared from a \(\text{Nd}_{2.5}\text{Fe}_{14}\text{B} + \text{Ga}_{0.5}\) target, the obtained coercivity reached approximately 1700 kA/m. We also found that \((BH)_{\text{max}}\) value reaches approximately 70 kJ/m\(^3\) by reducing Nd content and adding Zr or Nb.

Index Terms—Coercivity, pulsed laser deposition, remanence, thick film magnets.

I. INTRODUCTION

We have already reported the fabrication of isotropic Nd-Fe-B thick film magnets, which have coercivity, remanence, and \((BH)_{\text{max}}\) values of approximately 1000 kA/m, 0.6 T, and 60 kJ/m\(^3\), respectively, prepared by the PLD method with a high deposition rate of approximately 90 \(\mu\)m/h \cite{1}. In addition, an application of a 200 \(\mu\)m-thick Nd-Fe-B film on a Fe substrate to a milli-size motor was carried out as a practical usage, and it was confirmed that the rotation speed and torque constant under no-load test are 15160 rpm and 0.0236 mNm/A, respectively, at the gap of 0.1 mm between a rotor and a stator. As the advancement in micro-machines such as small motors depends on magnetic properties of small size magnets, a further improvement in magnetic properties of film magnets is strongly required.

According to the observation on microstructure for the previously reported films \cite{2}, the size of \(\text{Nd}_{2.5}\text{Fe}_{14}\text{B}\) grains ranged widely from 5 to 440 nm, and the average grain size was estimated at 150 nm, which suggests that refinement in grain size is a key technology to improve magnetic properties of the thick film magnets. It has also been reported that a use of additives such as Zr, Nb, Ga is effective to obtain homogeneous microstructure, and resultantly to increase remanence and coercivity for Nd-Fe-B based ribbons and HDDR powder \cite{3,4,5}, respectively.

This contribution reports magnetic properties of the PLD-fabricated thick films prepared from Nd-Fe-B targets including Zr, Nb, Ga additives. Furthermore, the pulse annealing as a high-speed crystallization method was applied to the films including the additives.

II. EXPERIMENTAL

The targets with the composition of \(\text{Nd}_{2.5}\text{Fe}_{14}\text{B}\), \(\text{Nd}_{2.5}\text{Fe}_{14}\text{B} + \text{Mo}_{0.5}\) at.\% (\(M = \text{Zr}, \text{Nb}, \text{Ga}\)) and \(\text{Nd}_{2.5}\text{Fe}_{14}\text{B} + \text{Mo}_{0.5}\) at.\% (\(M = \text{Zr}, \text{Nb}, \text{Ga}\)) were prepared. They were ablated with a Nd-YAG pulse laser (\(\lambda = 355 \text{ nm}\)) at the repetition rate of 30 Hz, and the films were deposited on Ta substrates. The distance between a target and a substrate and deposition time were fixed at 10 mm and 1 h, respectively. In this experiment, the deposition rate was varied from 20 to 40 \(\mu\)m/h, and the film thickness ranging was 20 to 40 \(\mu\)m. Before the ablation, the chamber was evacuated down to approximately \(10^{-5}\) Pa with a molecular turbo pump. In addition, a Ti sublimation pump was used as an auxiliary pump during the deposition. As-deposited films were amorphous, and, therefore, they were crystallized by annealing with an infrared furnace whose maximum output power was 8 kW. We set the annealing temperature, heating rate and holding time at 923 K, 400 K/min and 0 min, respectively, as the conditions of a conventional annealing (CA) method.

In addition, we adopted a pulse annealing (PA) method, in which samples were instantaneously crystallized for 1.7–2.0 s as a high-speed crystallization method (see Fig. 1). As shown in Fig. 2, the size of \(\text{Nd}_{2.5}\text{Fe}_{14}\text{B}\) grains for a sample annealed by PA method was smaller than that of one annealed by CA method \cite{6}. A peeling phenomenon was not observed in all samples which indicated that the mechanical properties of the films prepared by PA method were comparable with those of ones prepared by CA method \cite{7}.

After a sample was magnetized up to 7 T with a pulse magnetizer, magnetic properties were measured with a vibrating sample magnetometer which could apply a magnetic field up to approximately 1800 kA/m reversibly. All the post-annealed films were magnetically isotropic and, therefore, in-plane magnetic properties were shown in this article. The analysis of crystal structure was carried out with an X-ray diffractometer.
(XRD) and the average thickness was estimated from hysteresis loops of as-deposited films as described elsewhere [8].

III. RESULTS AND DISCUSSION

As mentioned in Introduction, it has been reported that use of additives is effective to obtain homogeneous structure, and resultantly they improved the magnetic properties of Nd-Fe-B based ribbons and HDDR powder [3]–[5]. In this experiment, we also tried to improve the magnetic properties of the films by using Zr, Nb and Ga. Fig. 3 shows demagnetization curves of PLD-fabricated Nd-Fe-B thick film magnets prepared from Nd$_2$Fe$_{14}$B + M$_{\theta \text{at.}\%}$ (M = Zr, Nb, Ga) targets. Use of additive of Zr, Nb and Ga enabled us to increase coercivity of the samples by 100–300 kA/m compared with that of additive free one.

For a further increase in coercivity, the PA method together with one of the above-mentioned additives was adopted. Fig. 5 shows the demagnetization curves of PLD-fabricated Nd-Fe-B thick film magnets prepared from Nd$_2$Fe$_{14}$B + M$_{\theta \text{at.}\%}$ (M = Zr, Nb, Ga) targets followed by the PA method. The role of the additives and the PA method mainly work on the increase of coercivity. For example, after a pulse annealing of an as-deposited film prepared from a Nd$_2$Fe$_{14}$B + Ga$_{\theta \text{at.}\%}$ target, the obtained coercivity reached approximately 1700 kA/m. In the previous report for the grain refinement in isotropic Nd-Fe-B ribbons [4], remanence enhancement was reported together with increase in coercivity. Our experiment, however, did not indicate the remanence enhancement, suggesting that the intergran exchange interaction among Nd-Fe-B grains was ultimately little. On the other hand, it is considered that the enhancement in coercivity is attributed to the increases in numbers of grains with a single domain structure. In Fig. 6, the above-mentioned results were summarized. It was clarified that an adoption of PA method as a high-speed crystallization together with additives such as Ga showed the synergistic results for the enhancement in coercivity.

As the cooperated effect of the PA method and additives causes a remarkable increase in coercivity, we can reduce the Nd content of a target. Therefore, we prepared the film magnets from Nd$_2$Fe$_{14}$B + M$_{\theta \text{at.}\%}$ (M = Zr, Nb, Ga) targets.
The coercivity values of the samples prepared from Ga added targets reached approximately 1700 kA/m. We also found that $(BH)_{\text{max}}$ value reaches approximately 70 kJ/m$^3$ by reducing Nd content and adding Zr or Nb.

ACKNOWLEDGMENT

This work was supported in part by the Ministry of Education, Science, Sports and Culture of Japan under a Grand-in-Aids (19360147 and 10136533) and the energy use rationalization technology and strategy developments program in 2005 from New Energy and Industrial Technology Development Organization (NEDO, Project P03033) of Japan.

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Manuscript received March 02, 2008. Current version published December 17, 2008. Corresponding author: M. M. Nakano (e-mail: mnakano@na-gasaki-u.ac.jp).