Review of Fabrication and Characterization of Nd–Fe–B Thick Films for Magnetic Micromachines

M. Nakano¹, S. Sato¹, F. Yamashita², T. Honda³, J. Yamaski³, K. Ishiyama⁴, M. Itakura⁵, J. Fidler⁶, T. Yanai¹, and H. Fukunaga¹

¹Department of Electrical Engineering and Electronics, Nagasaki University, Nagasaki 852-8521, Japan
²Motor Technology Center, Matsushita Electric Industrial Co., Ltd., Osaka 574-0044, Japan
³Department of Applied Science for Integrated System Engineering, Kyushu Institute of Technology, Kitakyushu 804-8550, Japan
⁴Research Institute of Electrical Communication, Tohoku University, Sendai 980-8577, Japan
⁵Department of Applied Science for Electronics and Materials, Kyushu University, Fukuoka 816-8580, Japan
⁶Vienna University of Technology, A-1040 Vienna, Austria

Isotropic Nd–Fe–B thick film magnets were prepared by a high-speed pulsed laser deposition method followed by a post annealing. The deposition rate of 90 μm/h could be successfully achieved, and a pulse annealing was adopted as the post annealing process in order to enhance coercivity. Use of a substrate heating system under the high deposition rate enabled us to obtain anisotropic thick films with \((BH)_{\text{max}}\) of approximately 120 kJ/m³, which show the potential for an improvement in the properties of the micromachines. Novel micromachines comprising the isotropic films were introduced.

Index Terms—Laser ablation, micromachines, micromotors, thick film devices.

I. INTRODUCTION

NUMEROUS studies [1], [2] on microactuators and micro-machines comprising small permanent magnets indicate that reduction in thickness for bulk magnets without deterioration of magnetic properties [3]–[5] and preparation of thick film magnets [6]–[9] thicker than 10 μm are key points to developing magnetic microactuators and systems (MAGMAS). For example, Nienhaus et al. have prepared disk-rotors made from Nd–Fe–B powders for planar micromotors [2]. Nakamura et al. [4] prepared a 100-μm-thick sintered bulk magnet with \((BH)_{\text{max}}\) of 245 kJ/m³ by using refinement of grain size and surface treatment after a mechanical etching process. Since Cadieu [6] has reported a 40-μm-thick Sm–Co film magnet in 1989, many researchers have reported sputtering-made anisotropic thick film magnets prepared under the high deposition rate of 20–40 μm. Recently, Uehara [9] also reported high \((BH)_{\text{max}}\) value of 279 kJ/m³. In addition, other fabrication methods for thick film magnets such as the aerosol deposition method [10], plasma spraying [11], and tape casting [12] have been reported.

In our study, the pulsed laser deposition (PLD) method was used for preparing thick film magnets applied to various applications. Some researchers have reported PLD-prepared Nd–Fe–B [13], [14], Sm–Co [15], and Sm–Fe–N films [16], and the thickness of almost all the samples was less than 1 μm because the aims in fabricating the films were based on application for recording media and physical interest such as the epitaxial method. We prepared Nd–Fe–B film magnets thicker than 20 μm under the deposition rate of 20–40 μm/h by the PLD method [17]. In addition, the thick films were applied to a dc brush-less motor [18], a micromachine swimming in liquid [19], and an electromagnetic friction-drive motor.

This contribution reports the magnetic properties and structure for the PLD-fabricated isotropic Nd–Fe–B films under the high deposition rate up to 90 μm/h. Improvement in magnetic properties by preparing anisotropic films using a substrate heating system [18] was introduced. We also reviewed three applications comprising the isotropic films [19], [20].

II. PREPARATION OF THICK FILM MAGNETS

We used a high-speed PLD method to fabricate Nd–Fe–B thick film magnets. In order to compensate loss of metallic Nd due to oxidation, the nominal composition of targets was set to Nd₂₄Fe₃₄B, which include a larger amount of Nd than the stoichiometric composition. These targets were ablated with a Nd–YAG pulse laser (λ = 355 nm) at the repetition rate of 30 Hz, and the distance between a target and various substrates such as Ta and Fe was fixed at 10 mm, as shown in Fig. 1. The deposition rate was varied from 20 to 90 μm/h by controlling the laser power from 3 to 10 W at the irradiation exit of a Nd–YAG laser system. Before the ablation, the chamber was evacuated down to about 10⁻⁴ Pa with a molecular turbo pump.

Fig. 1. Diagram of PLD method. The distance between a target and a substrate was fixed at 10 mm. The laser power was controlled from 3 to 10 W at an irradiation exit of a laser system.
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The structure of all the as-deposited films prepared without using a substrate heating system was amorphous. In order to form the Nd$_2$Fe$_{14}$B crystallized structure, the as-deposited films were postannealed under the vacuum of $10^{-3}$ Pa by using two methods, Methods A and B. In Method A, the samples were heated up to the designated temperature of 923 K under the heating rate of 400 K/min with an infrared furnace, and then they were cooled down to room temperature. In Method B, a pulse annealing with a high heating rate beyond 850 K/min was done.

In-plane and perpendicular $M-H$ loops were measured with a vibrating sample magnetometer under an applied field up to 2.5 T. Before the measurement, a pulsed field exceeding 8 T was applied to all the samples in order to magnetize. The analysis of crystal structure was carried out with an X-ray diffractometer. Thickness was measured with a digital micrometer. Nd–Fe–B films with the thickness range up to 1200 μm could be successfully obtained without deterioration of mechanical properties although the coercivity slightly decreased as the thickness increased (see Fig. 3).

Although the surface roughness increased with increasing laser power and deposition rate, the peeling phenomenon was not observed in all the as-deposited films. We also confirmed that the postannealed films did not peel from its substrate. It is generally said that a peeling phenomenon occurs as the...
thickness of films increases because of the increase in an inner stress; however, the above-mentioned PLD-made samples did not show the peeling phenomenon. From these results, the increase in the laser power is effective in the increase in deposition rate without deterioration of magnetic and mechanical properties.

B. Microstructure of Isotropic PLD-Made Nd–Fe–B Films and Adoption in a Pulse Annealing

Investigation on microstructure of PLD-prepared Nd–Fe–B films is effective for the improvement in magnetic properties. Fig. 8 shows a transmission electron microscopy (TEM) image of a PLD-prepared isotropic film with the coercivity and remanence of 1080 kA/m and 0.59 T, respectively. The sample shown in Fig. 8(b) was prepared by etching from the surface by an FIB. TEM observations for the inside [Fig. 8(a)] of the film and boundary on a Ta substrate [Fig. 8(c)] were executed.

The microstructure indicated that the film was mainly composed of Nd₅Fe₁₄B grains whose size varied widely from 5 to 440 nm, and the average grain size was estimated as 130 nm. In addition, the grain growth seemed to occur directly on the substrate, because the boundary phase between the sample and the substrate did not affect the grain growth behavior in PLD-prepared thick films.

High-speed crystallization has been reported as one of hopeful methods for reducing the grain size and its distribution, and resultantlly it enables us to improve the coercivity values of sputtered Nd–Fe–B films and rapid-quenched Pr–Fe–B ribbons.

IV. Anisotropic Nd–Fe–B Film Magnets Prepared by the High-Speed PLD Method with a Substrate Heating System

We further attempted to develop the above-mentioned PLD-made Nd–Fe–B thick films, and preparation of anisotropic thick film magnets was carried out by adoption of a substrate heating system between a target and a substrate. Use of the system enabled us to obtain anisotropic Nd–Fe–B thick film magnets under a high deposition rate.

We adopted a continuous deposition method, which means that a film is continuously deposited during heating substrate. Although the obtained remanence and \((B H)_{\text{max}}\) values of the anisotropic films were higher than those of decreased by 40% compared to that of the isotropic ones [17], [19]. The deterioration of coercivity can be attributed to a heterogeneous grain growth due to heating during the long deposition time.

In order to overcome this difficulty, we adopted a new method, in which the deposition process was intercepted repeatedly during the substrate heating. We designated this method as the interceptive deposition method (IDM). The new preparation method, IDM, was applied to the preparation of Nd–Fe–B film magnets, and it was clarified that IDM is effective in fabricating anisotropic Nd–Fe–B thick film magnets under the deposition rate of 20–50 μm/h. Although the mechanism for the improvement in magnetic properties is under investigation, a magnetic micromotor comprising the IDM-made films is a hopeful candidate as a developed micromachine.

V. Micromachines Comprising Isotropic PLD-Made Nd–Fe–B Films

Fig. 10 shows the structure of a small dc brush-less motor comprising a 200-μm-thick isotropic PLD-prepared Nd–Fe–B film magnet, which has 971 kA/m in coercivity and 0.57 T in remanence, respectively. The film was deposited on a Fe substrate and the thickness and diameter of the motor were 0.8 and 5 mm, respectively. We confirmed that it rotates at 15160 rpm under no-load test, and has torque constant of 0.0236 mNm/A at the gap of 0.1 mm between a rotor and a stator. The motor is applicable to a small hard disk and a vibration motor inside a mobile phone.

We have further reported a spiral type micromachine with 0.14 mm in outer diameter and 1.0 mm in length prepared by a tungsten wire deposited on Nd–Fe–B film magnet. As the film magnet was magnetized in the circumferential direction, the machine rotated in synch with the rotating external magnetic field and the spiral structure generated propellant force. In our experiment, three types of liquids with kinematic viscosity of 1, 10, and 100 mm²/s, respectively, were used, and the external magnetic field of 8 kA/m was applied under the frequency range between 2 and 10 Hz. It was found that the wireless micromachine.
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Isotropic Nd-Fe-B thick film magnets prepared by the high-speed PLD method could be applied to a small dc brush-less motor, a machine swimming in liquid, and an electromagnetic friction-drive micromotor. Anisotropic PLD-prepared Nd-Fe-B thick film magnets are promising materials to advance various small magnetic micromachines.

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