Journal of Alloys and Compounds 408–412 (2006) 1422–1425

Journal of ALLOYS AND COMPOUNDS

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Microstructure and magnetic properties of PLD-made Nd–Fe–B thick films

M. Nakano [a,*], R. Kato [a], S. Hoefinger [b], J. Fidler [b], F. Yamashita [c], H. Fukunaga [a]

[a] *Faculty of Engineering, Nagasaki University, 1-14 Bunkyou-Cho, Nagasaki 852-8521, Japan*
[b] *Vienna University of Technology, A-1040 Vienna, Austria*
[c] *Matsushita Electric Industrial Co. Ltd., 7-1-1 Morofuku, Daito, Osaka 574-0044, Japan*

Available online 20 July 2005

Abstract

Hard magnetic properties and microstructure of PLD-made Nd–Fe–B thick film magnets were studied. The films were mainly composed of $Nd_2Fe_{14}B$ grains, and the average grain size was approximately 150 nm. In addition, we succeeded to fabricate a milli-size DC brush-less motor with a PLD-made Nd–Fe–B film.

Keywords: Permanent magnets; Magnetic films and multi-layers; Laser processing

1. Introduction

Some researchers reported thick film magnets by sputtering method [1,2]. The deposition rate is not sufficient for applying them to mass production. We, therefore, have proposed a high-speed PLD method as an attractive approach for obtaining Nd–Fe–B film magnets under a high-deposition rate of 20–40 μm/h [3]. The magnetic properties of the PLD-made films were comparable to those of Nd–Fe–B films prepared by sputtering method [1,2]. In order to apply the PLD-made film magnets to small electronic devices such as a milli-size motors, further investigations on magnetic properties and microstructure of the films are required.

In this report, the relationship between target compositions and magnetic properties was investigated. In addition, TEM and SEM observations were carried out for the films. We confirmed that a milli-size DC brush-less motor with a PLD-made Nd–Fe–B film magnet 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.

* Corresponding author.
E-mail address: mnakano@net.nagasaki-u.ac.jp (M. Nakano).

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doi:10.1016/j.jallcom.2005.04.188

2. Experimental procedure

The targets with the compositions of $Nd_XFe_{14}B$ ($X = 2.2$, 2.4 and 2.6) were prepared. They were ablated with a Nd-YAG pulse laser at the repetition rate of 30 Hz, and the films were deposited on Ta and Fe substrates. In this study, the area of the both substrates was 100 mm^2. The distance between a target and a substrate was controlled in the range of 3–30 mm. Fig. 1 shows a photograph of plume at the target–substrate distance of 10 mm.

Before the ablation, the chamber was evacuated down to approximately 10^{-4} Pa with a molecular turbo pump. In addition, a Ti sublimation pump was used as an auxiliary pump during the deposition. Almost as-deposited films were amorphous, and therefore, they were crystallized under the vacuum of 10^{-3} Pa as described below. The samples were heated up to the designated temperature (923 K) under the heating rates of 150, 400 and 800 K/min with an infrared furnace, and then they were cooled down to room temperature.

Magnetic properties were evaluated with a vibrating sample magnetometer (VSM). The analyses of structure and composition were carried out with an X-ray diffractometer (XRD) and an energy dispersive X-ray spectrometer (EDX). The morphology of the surface and the cross-section was observed with a scanning electron microscope (SEM). The

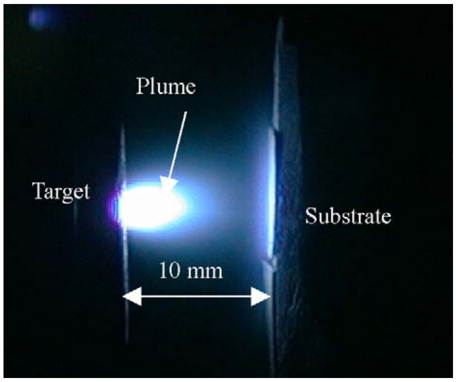

Fig. 1. Photograph of ablation phenomenon. The distance between a target and a substrate was 10 mm.

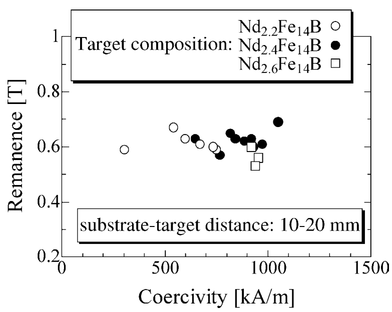

Fig. 2. In-plane properties of Nd–Fe–B films prepared using $Nd_XFe_{14}B$ (X = 2.2, 2.4 and 2.6) targets. The distance between a target and a substrate was 10–20 mm.

microstructure of the samples was observed with a transferring electron microscope (TEM). Although the evaluation of volume of Nd–Fe–B films is very important in order to determine M_r and $(BH)_{max}$ correctly, we used two methods in this experiment (Methods A and B). In the samples deposited on Ta substrates, the average thickness was estimated using hysteresis loops of as-deposited films as described elsewhere [3] (Method A). In Method A, it was difficult to evaluate the thickness of Nd–Fe–B films deposited on Fe substrates. The average thickness of the samples, therefore, was directly measured with a micrometer (Method B) although Method B was considered to have approximately ±1 μm error.

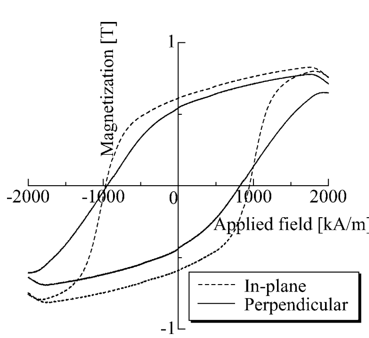

Fig. 3. In-plane and perpendicular M–H loops of an Nd–Fe–B film magnet fabricated on a Ta substrate. The sample was prepared using an $Nd_{2.2}Fe_{14}B$ target, and the distance of 10 mm between a target and a substrate. The thickness of the film was approximately 40 μm.

3. Results and discussion

3.1. Relationship between magnetic properties of Nd–Fe–B thick film magnets and target compositions

The in-plane properties of Nd–Fe–B films fabricated by using each target are shown in Fig. 2. Decrease in Nd ratio of the targets reduced the value of coercivity, while increase in Nd ratio reduced the value of remanence. We, therefore, confirmed that an $Nd_{2.4}Fe_{14}B$ target has an optimum composition for obtaining comparable magnetic properties to those of Nd–Fe–B films prepared by sputtering method [1,2].

Fig. 3 shows in-plane and perpendicular M–H loops of a 40 μm-thick Nd–Fe–B film fabricated using an $Nd_{2.4}Fe_{14}B$

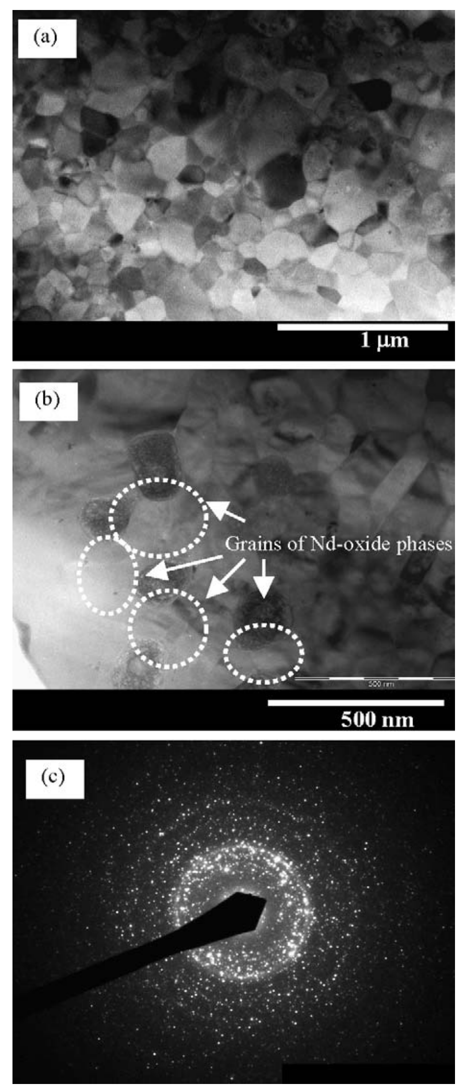

Fig. 4. TEM observation of PLD-made Nd–Fe–B thick film magnet. The M–H loop of the sample is shown in Fig. 3. (a) TEM image, (b) TEM image and (c) Diffraction pattern at selected area (a).

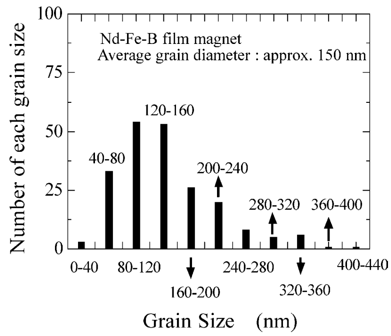

Fig. 5. Distribution of grain size in an Nd–Fe–B film magnet. The graph is partly corresponding to the TEM image of Fig. 4a.

target. The sample had magnetically isotropic magnetic property for in-plane and perpendicular directions.

3.2. Microstructure and homogeneity of Nd–Fe–B thick film magnets

Fig. 4 shows TEM micrographs and a diffraction pattern of the sample shown in Fig. 3. In almost all parts of the sample, Fig. 4a-type microstructure of typical Nd–Fe–B grains was observed. In addition, XRD patterns indicated $Nd_2Fe_{14}B$ phase in the sample. We, however, found that there would be the grains with Nd oxide phases, such as Nd_2O_3 (see Fig. 4b). The existence of the non-magnetic phases is consistent with the lower remanence value compared with theoretical one of 0.8 T in isotropic $Nd_2Fe_{14}B$ phase. In addition, the result of Fig. 4c agrees with the isotropic magnetic property as displayed in Fig. 3. Fig. 4a and other similar pictures showed the distribution of grain size in a PLD-made Nd–Fe–B film magnet as shown in Fig. 5. The size of grains varied widely from 5 to 440 nm, and the average grain size was estimated as 150 nm. Livingstone reported that 300 nm is a critical grain size for single-domain [4], and his result suggests that PLD-made Nd–Fe–B films are probably composed of both grains with single-domain and multi-domains, respectively.

In order to apply the above-mentioned films to small motors, SEM observation was carried out. As shown in Fig. 6,

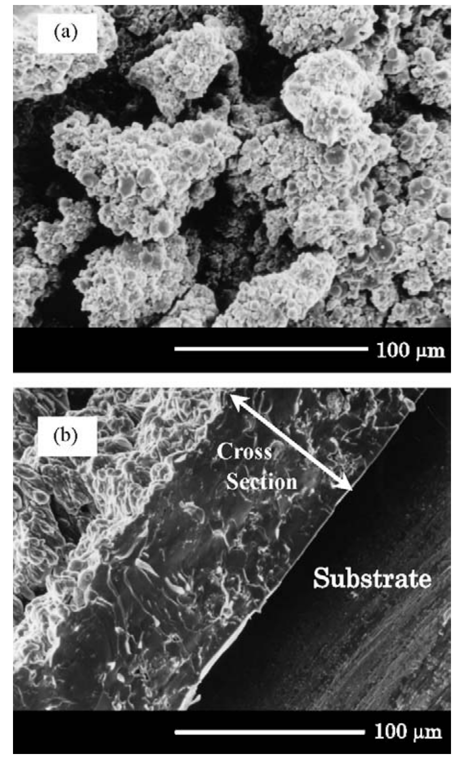

Fig. 6. SEM observation of PLD-made Nd–Fe–B thick film magnet. The thickness of the film was approximately 80 μm. (a) Surface and (b) cross-section.

although the surface roughness of PLD-made films was larger than that of the one prepared by sputtering method, we confirmed that the film can be applied for a milli-size motor at a gap of 0.1 mm. In addition, SEM observation of cross-section in Nd–Fe–B film magnets indicated the high-packing density of the film.

3.3. Fabrication of a milli-size motor using a 200 μm-thick Nd–Fe–B thick film magnet

Fig. 7 shows the structure of a milli-size DC brush-less motor with a 200 μm-thick PLD-made Nd–Fe–B film

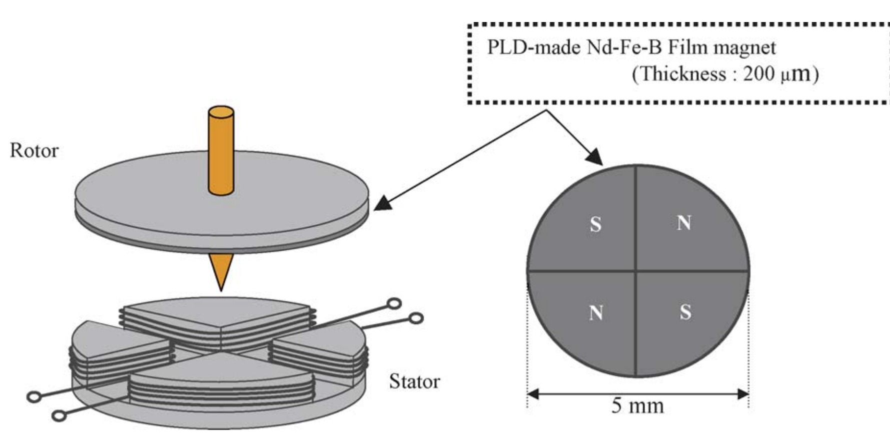

Fig. 7. Diagram of a milli-size DC brush-less motor with a PLD-made Nd–Fe–B film magnet.

magnet. Here, the film was deposited on a Fe substrate. The thickness and diameter of the motor were 0.8 and 5 mm, respectively. We confirmed that the motor 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. Therefore, the developed PLD-made Nd–Fe–B film magnet prepared by the proposed method is expected to be applied for high-performance milli-size brush-less motor of the axial gap type.

4. Conclusion

In this study, investigation on the magnetic properties, microstructure, and homogeneity was carried out. It was clarified that the high-speed PLD method is promising for obtaining Nd–Fe–B film magnets that can be applied for a milli-size motor. In order to apply this material for the practical application, we have to increase the deposition area.

Acknowledgements

This work was supported in part by the Ministry of Education, Science, Sports and Culture of Japan under a Grant-in-Aid (Nos. 15656084 and 16686022).

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