Nd–Fe–B thick film magnets with Nb additive prepared by vacuum arc deposition method

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Isotropic Nd–Fe–B thick film magnets were prepared by a vacuum arc deposition method with the deposition rate of approximately 10 μm/h followed by pulse-annealing process. It was found that an optimum amount of Nb additive is effective to enhance the coercivity without the deterioration of remanence and $(BH)_{max}$ values of the isotropic thick films. © *2011 American Institute of Physics*. [doi:10.1063/1.3566061]

I. INTRODUCTION

A lot of studies on anisotropic Nd–Fe–B thick film magnets have been carried out in order to advance microelectromechanical systems (MEMS).[1–7] Although $(BH)_{max}$ of an isotropic Nd–Fe–B film is inferior to that of an anisotropic one,[8,9] the flexibility of magnetization is attractive in the practical applications such as miniaturized motors. For example, Töpfer *et al*. reported a multipolarly magnetized isotropic Nd–Fe–B film prepared by a screen printing method.[10] Yamashita *et al*. also indicated that a multipolarly magnetized isotropic thick film was effective to enhance the torque of a milli-size motor compared with that of a motor comprising an isotropic one.[11] We have already reported on the isotropic Nd–Fe–B thick film magnets prepared by using a PLD (pulsed laser deposition) method and applied them to several micro machines.[12] The characteristic of the method was the high deposition rate up to 90 μm/h on a 5×5 mm^2 substrate by taking advantage of small particles (droplets) emitted from an Nd–Fe–B target. Recently, we also reported on the vacuum arc deposition (VAD)-fabricated isotropic Nd–Fe–B thick film magnets.[13] The method also utilizes small particle, however, they are liquid droplets which are different from solid ones synthesized by PLD method.

This contribution reports that Nb additive enables us to enhance the coercivity value for the VAD-fabricated isotropic Nd–Fe–B films.

II. EXPERIMENTAL PROCEDURES

As shown in Fig. 1, the VAD method enables us to obtain Nd–Fe–B films thicker than 40 μm on Ta substrates by using several targets with the compositions of $Nd_{2.0}Fe_{14}B + Nb_X$ ($X = 0$, 0.1, 0.3, 0.5, 1.0, and 3.0 at. %). The observation of energy dispersive x-ray spectrometry (EDX) revealed that the Nb amount of the films increased according to the increase in Nb additive of each target. Before the deposition, the chamber was evacuated down to approximately $2–6 \times 10^{-5}$ Pa. The distance between the target and substrate

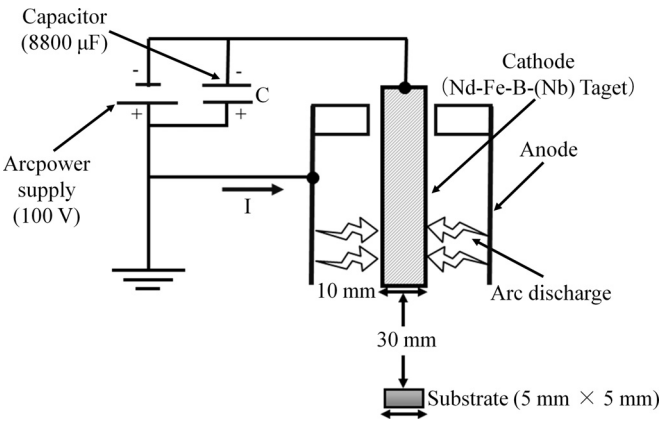

FIG. 1. Schematic diagram of vacuum arc deposition (VAD) method.

was fixed at 30 mm. The observation of the as-deposited film indicated the existence of liquid droplets which were different from solid droplets shown on the surface of PLD-made films.[12] In addition, the deposition rate reached approximately 10 μm/h as shown in Fig. 2. The compositional ratios of Nd to Fe atoms in the obtained films were almost the

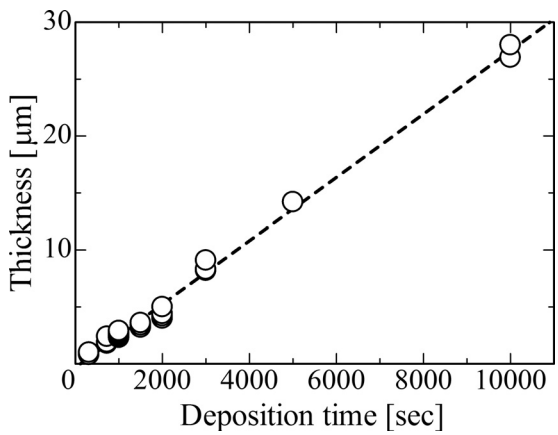

FIG. 2. Relationship between deposition time and thickness of Nd–Fe–B thick film magnets prepared by a VAD method. Deposition rate was approximately 10 μm/h.

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same as those of each target. The structure of all the as-deposited films was amorphous, therefore, they were crystallized by pulse-annealing process for periods of 3.5 or 3.6 s within an infrared furnace.[14] The annealing time of 3.5 or 3.6 s means that the electric current flows in the infrared furnace during the period. In order to cool the samples up to room temperature, the samples are hold in the furnace for approximately 5 min. After magnetizing the samples up to 7 T using a pulsed magnetic field created by a magnetization meter, the magnetic properties of each film were measured with a vibrating sample magnetometer (VSM) which could apply reversibly a magnetic field up to approximately 1800 kA/m. In this paper, only the in-plane magnetic properties will be shown because all the crystallized films are isotropic magnetically. The analysis of the crystal structure was carried out with an x-ray diffractometer (XRD) with Cu Kα radiation and the average thickness was estimated using hysteresis loops of as-deposited films.[15] The resulting thickness range of samples was from 50 to 70 μm.

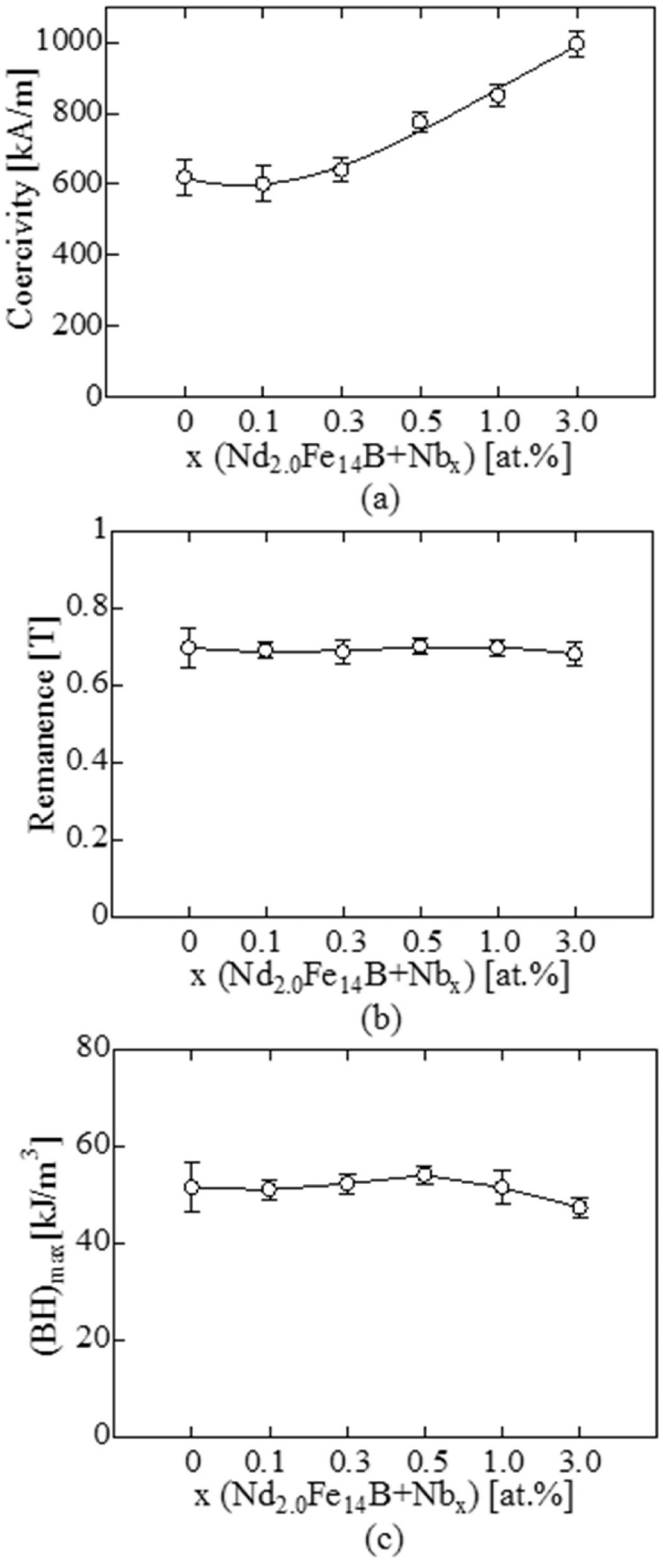

FIG. 3. (a) Coercivity, (b) remanence, and (c) $(BH)_{max}$ values of several samples as a function of the amount of Nb additive in each target. Coercivity increased with the increase in Nb contents.

III. RESULTS AND DISCUSSION

It is generally reported that the use of additives such as Zr, Nb, and Ga enables the obtaining of the homogeneous microstructure, and consequently the increase of the remanence and coercivity for Nd–Fe–B based ribbons and HDDR (Hydrogenation Decomposition Desorption Recombination) powders,[16–18] respectively. Among the three elements, we reported that Nb additive is the most effective in improving the magnetic properties and refining the grain size for PLD-fabricated Nd–Fe–B isotropic films by using a stoichiometric target.[19] We, therefore, selected Nb additive to improve the magnetic properties for isotropic Nd–Fe–B films prepared by VAD.

After the above-mentioned pulse-annealing process, the magnetic properties of samples were evaluated. Figure 3 shows the average values of coercivity, remanence, and $(BH)_{max}$ in several samples as a function of the amount of Nb additive in each target. Although the coercivity value increased with increasing the amount of Nb, the deterioration of $(BH)_{max}$ occurred as the amount exceeded 0.5 at. %. Figure 4 shows the in-plane M-H loops of sample prepared from $Nd_{2.0}Fe_{14}B + Nb_{0.5\ at.\ \%}$ and $Nd_{2.0}Fe_{14}B + Nb_{3.0\ at.\ \%}$ targets, respectively. For the sample shown in Fig. 4, the value of coecivity reaches approximately 1000 kA/m, however, the M-H loop presents a knick. It is considered that a larger amount of Nb additive reduces the interaction between the grains. On the other hand, the optimum amount of Nb additive enhances the coercivity without deteriorating the remanence and $(BH)_{max}$. Consequently, values of coercivity,

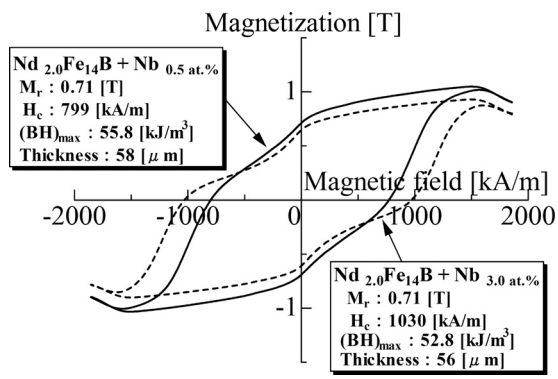

FIG. 4. In-plane M-H loops of samples prepared from $Nd_{2.0}Fe_{14}B + Nb_{0.5\ at.\ \%}$ and $Nd_{2.0}Fe_{14}B + Nb_{3.0\ at.\ \%}$ targets, respectively.

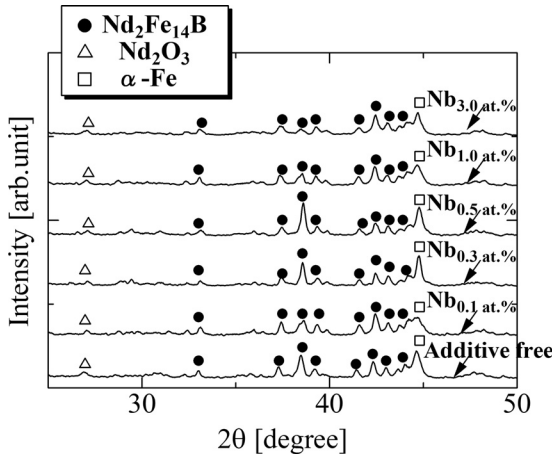

FIG. 5. X-ray diffraction patterns of Nd–Fe–B thick film magnets with and without Nb additives prepared by a VAD method. $Nd_2Fe_{14}B$, α-Fe, and Nd_2O_3 phases were observed for the films annealed 3.5 or 3.6 s.

remanence, and $(BH)_{max}$ of 800 kA/m, 0.71 T, and 56 kJ/m^3, respectively, could be obtained. We investigated the crystallized structure of Nb free and Nb added thick films by x-ray diffraction. As displayed in Fig. 5, the peaks corresponding to $Nd_2Fe_{14}B$, α-Fe, and Nd_2O_3 phases were observed in all samples. Although the intensity of $Nd_2Fe_{14}B$ phase peaks changes with the Nb content, the amount of α-Fe remains almost unchanged. It is considered that Nb additive mainly affects the control of grain size in $Nd_2Fe_{14}B$ phase. For example, it was found that the grain refinement occurs in each sample prepared by $Nd_{2.0}Fe_{14}B + Nb_X$ ($X = 1.0$ and 3.0 at. %) targets, respectively. Figure 6 shows the grain size of each sample estimated by Scherrer's equation.[20] As the amount of Nb in the target becomes larger, the grain size is smaller. These results suggest that the Nb additive is effective to increase the coercivity because of the reduction in the grain size for VAD-made isotropic Nd–Fe–B thick film magnets.

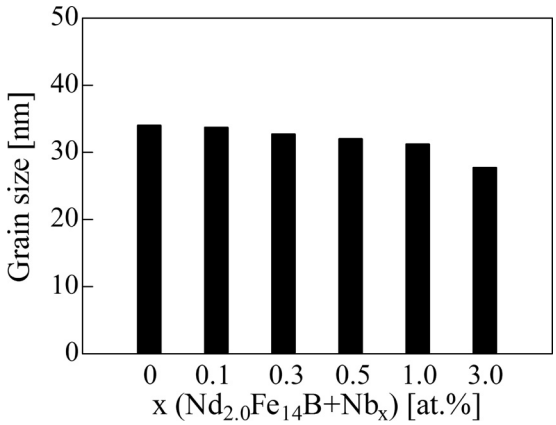

FIG. 6. Nanograin size of Nd–Fe–B thick film magnets prepared by using several targets with the composition of $Nd_{2.0}Fe_{14}B + Nb_X$ ($X = 0$, 0.1, 0.3, 0.5, 1.0, and 3.0 at. %). The size was estimated by Scherrer's equation.

IV. CONCLUSION

Nd–Fe–B thick film magnets with Nb additives were prepared by using a VAD method with the deposition rate of approximately 10 μm/h followed by pulse-annealing process. We succeeded in obtaining isotropic Nd–Fe–B thick films with coercivity, remanence, and $(BH)_{max}$ values up to 800 kA/m, 0.71 T, and 56 kJ/m^3, respectively, for Nb content of approximately 0.5 at. %.

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