

Nd-Fe-B film magnets with the thickness above 100 μm deposited on Si substrates

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Although increase in thickness of an Nd-Fe-B film magnet is indispensable to provide a sufficient magnetic field, it was difficult to suppress the peeling phenomenon due to the different values of a linear expansion coefficient for a Si substrate and a Nd-Fe-B film even if a buffer layer such as a Ta film was used. In this report, it was confirmed that a control of the microstructure for PLD (Pulsed Laser Deposition)-fabricated Nd-Fe-B films enabled us to increase the thickness up to approximately 160 μm without a buffer layer on a Si substrate. Namely, we found that the precipitation of Nd element at the boundary of Nd-Fe-B grains together with the triple junctions due to the composition adjustment is effective to suppress the destruction of the samples through an annealing process. The magnetic properties of the prepared films were comparable to those of previously reported ones deposited on metal substrates. Although the mechanism is under investigation, the above-mentioned film had stronger adhesive force compared with that of a sputtering-made film. Resultantly, no deterioration of mechanical together with magnetic properties could be observed after a dicing process.

Index Terms—Nd-Fe-B, film magnet, Si substrate, PLD (Pulsed Laser Deposition), MEMS

I. INTRODUCTION

In order to develop a micro-magnetic device prepared by taking account of a micromachining technology, several researchers have already reported sputtering-fabricated Nd-Fe-B thick-film magnets on Si wafers with Ta buffer layers [1-3]. Although the magnetic properties of all the anisotropic Nd-Fe-B were excellent, the thickness of each film was mainly less than 20 μm . On the other hand, we have reported isotropic Nd-Fe-B thick-film magnets with the thickness range from 10 to 1200 μm on metal substrates (Ta, Fe) by using a PLD (Pulsed Laser Deposition) method with the deposition rate of several-tens-microns per hour [4]. Furthermore, we demonstrated several magnetic devices such as a dc milli-size motor, a friction drive motor and a swimming machine in liquid, respectively, by using the PLD-made thick-films [5]. Recently, we deposited an isotropic 20 μm -thick Nd-Fe-B thick-film on a Si single crystal wafer coating with a 1.0 μm -thick Ta film by using the PLD, however the sample was broken after a dicing process [6]. It is considered that the phenomenon was attributed to the mechanical property for the Ta film with hard brittle.

This contribution reports that a control of microstructure of Nd-Fe-B thick-films enabled us to increase the thickness above 100 μm without a buffer layer on Si substrates. The magnetic properties of the samples were comparable to those of previously reported ones deposited on Ta substrates. In addition, it was confirmed that no deterioration of mechanical and magnetic properties could be obtained after a dicing process.

II. EXPERIMENTAL PROCEDURE

Each rotated target with the composition of $\text{Nd}_x\text{Fe}_{14}\text{B}$ ($x=2.0, 2.6, 3.0, 3.5$) was ablated by an Nd-YAG pulse laser at the laser energy density of approximately 4 J/cm^2 under the

distance between a target and a substrate of approximately 10 mm. All the as-deposited Nd-Fe-B thick-films on $\text{SiO}_2/\text{Si}(100)$ substrates (5 mm \times 5 mm : square) without a buffer layer had amorphous structure, therefore they were crystallized by a pulse annealing (PA) method in the vacuum atmosphere of $2\text{-}5 \times 10^{-5}$ Pa. The pulse-annealing time was approximately 1.7 s with an infrared furnace at output power of 8 kW, and then they were cooled down to room temperature [7]. The composition and the magnetic properties of the samples were measured with a SEM (Scanning Electron Microscope)-EDX (Energy Dispersive X-ray Spectroscopy) and a VSM (Vibrating Sample Magnetometer), respectively. The microstructure was observed with a TEM (Transmission Electron Microscope). Mechanical properties were evaluated by using a dicing process together with a scratch tester.

III. RESULTS AND DISCUSSION

A. Increase in thickness of Nd-Fe-B thick-film magnets deposited on Si substrates

Figure 1 shows the relationship between thickness and Nd contents in isotropic Nd-Fe-B thick-films deposited on SiO_2/Si substrates. After the annealing process, a lot of samples without destruction (the symbol of \circ seen in Fig.1) could be prepared. On the other hand, all the other samples plotted as “ \bullet ” were broken. As the Nd content exceeded approximately 22 at. %, the thickness could be enhanced up to approximately 160 μm without the deterioration of mechanical properties. Figure 2 shows the photographs of a partially broken sample observed by SEM. The sample broke from the inside of a Si substrate instead the Nd-Fe-B film didn't peel from the substrate, which did not agree with

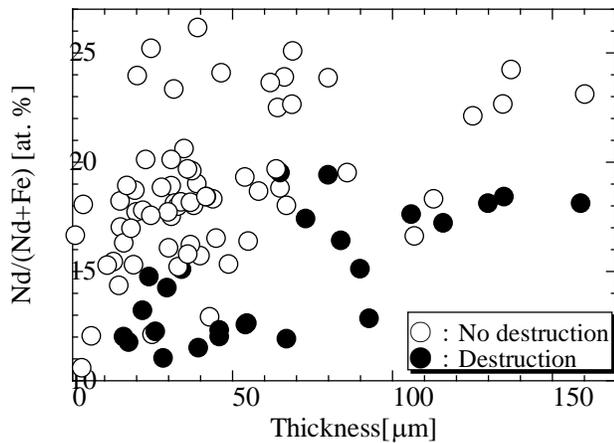


Fig. 1 Relationship between thickness and Nd contents in PLD-fabricated Nd-Fe-B thick-film magnets deposited on Si substrates. Increase in Nd contents enabled us to enhance the thickness up to 160 μm without mechanical destruction.

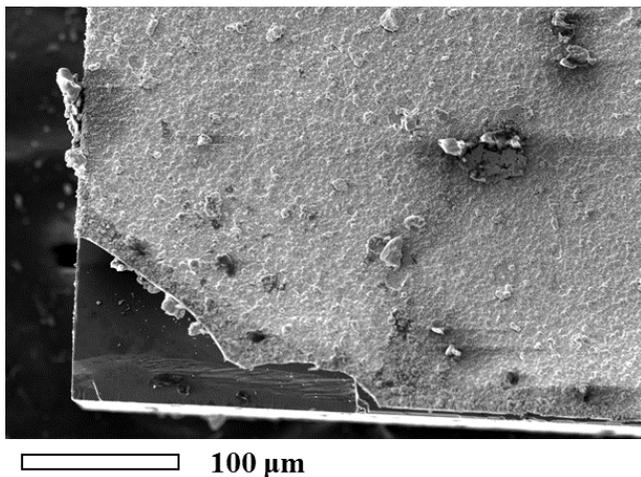


Fig. 2 Photographs of a sample after an annealing process. The sample partially broke from the inside of a Si substrate due to the different linear expansion coefficients of a Si substrate and an Nd-Fe-B film.

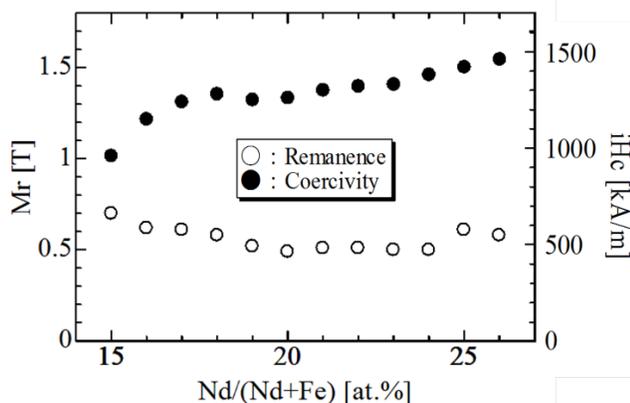


Fig. 3 Average remanence and coercivity values as a function of Nd contents in samples symbolized “○” in Fig. 1. As the Nd contents increased, remanence slightly decreased and coercivity increased.

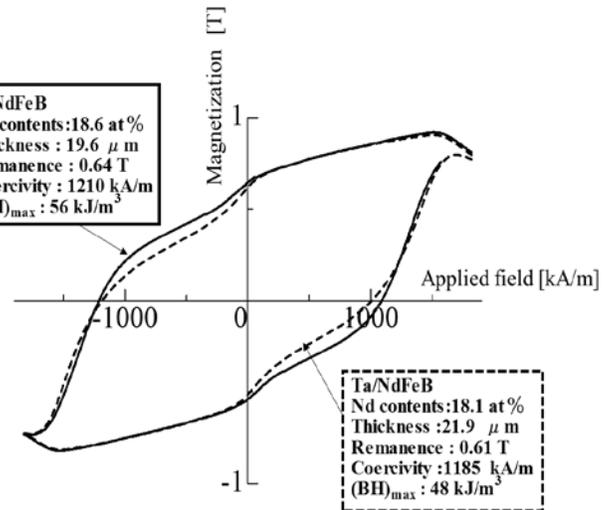


Fig. 4 M-H loops of two Nd-Fe-B films deposited on a Si and a Ta substrate, respectively. The Nd contents of both films were approximately 18 at. %. It was confirmed that the magnetic properties were almost the same.

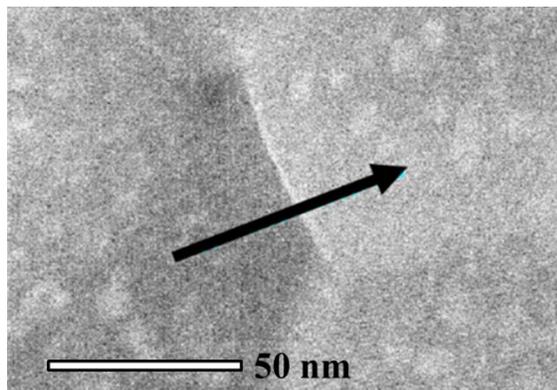
previously reported phenomenon for a sputtering-fabricated sample [3]. All the samples plotted as “●” displayed in Fig. 1 showed the same breaking phenomenon. The destruction is considered to be attributed to the different values of each linear expansion coefficient for a Si substrate ($2.6 \times 10^{-6} \text{K}^{-1}$) and an $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase ($14.7 \times 10^{-6} \text{K}^{-1}$). The phenomenon also indicates the strong adhesion between an Nd-Fe-B film and a SiO_2/Si substrate in PLD-fabricated films. Further investigations on the origin of the strong adhesion are required.

Average magnetic properties of the films displayed as “○” in Fig. 1 were measured (see Fig. 3) When the increase in Nd contents, remanence slightly decreased and coercivity increased from 1000 to 1500 kA/m. Moreover, an M-H loop of a sample plotted as “○” was compared to that of previously reported one deposited on a Ta substrate [1] as shown in Fig. 4. The Nd contents of both films were approximately 18 at. % and magnetic properties of the film deposited on a Si substrate were comparable to those of another one. The observation of the X-ray diffraction patterns of the films revealed that the samples were mainly composed of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase. Although each kink in the demagnetization curves is considered to be attributed to the existence of some soft magnetic phases, it was difficult to observe the soft magnetic phases in the present stage. The further investigation is required. In order to investigate the mechanism for enhancing the thickness up to 160 μm in the Nd-rich Nd-Fe-B films, the microstructure was observed by TEM-EDX shown in Figs. 5(a) and (b). We observed that Nd element precipitated around the grain boundary. In the present stage, we have difficulty in showing a solid evidence proving that the precipitation of the Nd at the $\text{Nd}_2\text{Fe}_{14}\text{B}$ grain boundary together with the triple junctions is the main reason why the thermal stress is suppressed for the Nd-Fe-B film. On the other hand, several researchers reported that the use of Ta buffer layer is effective to suppress the thermal stress between a Si substrate and Nd-Fe-B film because the linear expansion coefficient of a Ta layer is the intermediate value of $6.3 \times 10^{-6} \text{K}^{-1}$. We, therefore,

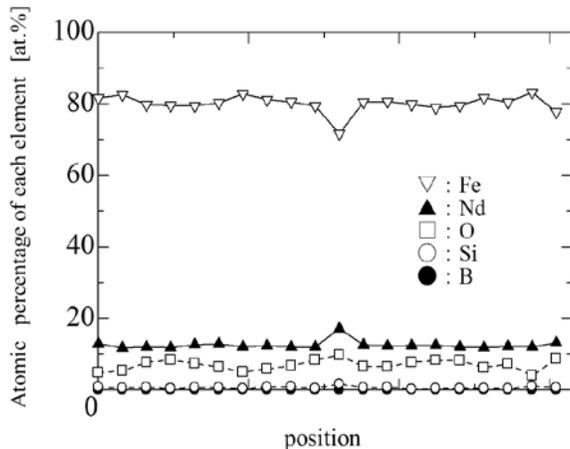
considered that the precipitation of Nd element is one of the reasons to suppress the stress in a sample and that resultantly the destruction didn't occur.

B. Mechanical properties of PLD-fabricated Nd-Fe-B thick-film magnets deposited on Si Substrates

A dicing process for a 113 μm -thick sample which had the remanence of 0.5 T and coercivity of 1160 kA/m was carried out as shown in Fig. 6. It was clarified that the sample could be diced smoothly without destruction. We also confirmed that the magnetic properties didn't degrade after the dicing. Furthermore, in order to evaluate the adhesion between a Nd-Fe-B film and a Si substrate, a scratch test was applied for a PLD-made 92 μm -thick Nd-Fe-B film on a Si substrate without a buffer layer. (see Fig. 7(a)) The scratch was traced with increasing the load shown in Fig. 7(b). No destruction and peeling could be observed as the scratch load changed from 0 to approximately 250 N. These results suggest an applicable property to MEMS in PLD-made Nd-Fe-B thick-films on Si substrates.



(a) Photograph of a microstructure for a 37 μm -thick Nd-Fe-B film deposited on a Si substrate. The arrow indicates the direction for the analysis of composition.



(b) Atomic percentage of each element depends on the position of analysis direction shown in Fig. 5(a).

Fig.5 TEM-EDX observation for a Nd-rich Nd-Fe-B film displayed as "○" in Fig. 1. Nd content of the sample was approximately 19.6 at. %. Around the position of grain boundary, it was confirmed that Nd content increased.

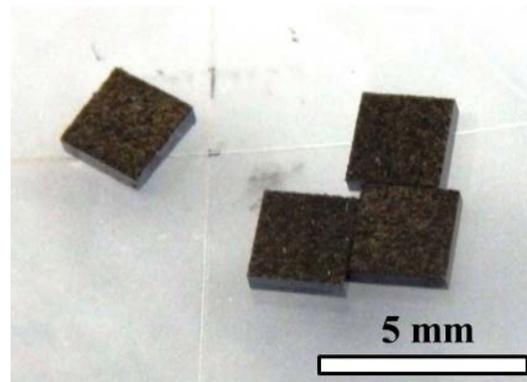
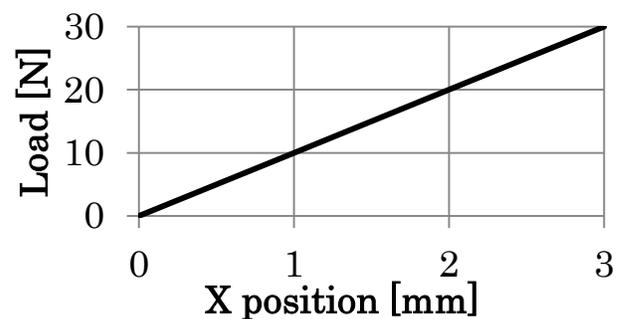


Fig. 6 Photograph of 113 μm -thick Nd-Fe-B film magnets deposited on Si substrates after a dicing process. No destruction could be observed after the dicing process.



(a) Scratched indentation



(b) Load of scratch as a function of trace position

Fig. 7 Scratched indentation for a PLD-made 92 μm -thick Nd-Fe-B film on a Si substrate without a buffer layer. The scratch was traced with increasing the load range from 0 to approximately 30 N shown in Fig. 7(b). Destruction and peeling didn't occur within the above-mentioned conditions.

IV. CONCLUSION

In this study, an enhancement in thickness of Nd-Fe-B film magnets deposited on Si substrates was carried out. The obtained results are summarized as follows;

- 1) The control of microstructure for Nd-Fe-B thick-films enabled us to increase the thickness up to approximately 160 μm without a buffer layer on a Si substrate. It is considered that the precipitation of Nd element at the boundary of Nd-Fe-B grains together with the triple junctions due to the composition adjustment is one of the reasons why the thermal stress is suppressed for the Nd-Fe-B film.
- 2) The magnetic properties of the samples deposited on Si substrates were comparable to those of previously reported ones deposited on Ta substrates.
- 3) No deterioration of mechanical and magnetic properties could be observed after a dicing process. In addition, a scratch test showed strong adhesion between a Nd-Fe-

B film and a Si substrate. Further investigations on the origin of the strong adhesion are required.

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