

PLD-fabricated isotropic Pr-Fe-B film magnets deposited on glass substrates

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Pr-Fe-B thick-film magnets were deposited on glass substrates without a buffer layer using a PLD (Pulsed Laser Deposition) method to apply the films to MEMS (Micro-Electro-Mechanical-Systems). Large particles with the average diameter of approximately 50 μm were emitted from a target and enabled us to obtain the relatively high deposition rate of 30 $\mu\text{m}/\text{h}$. An approximately 100 μm -thick Pr-Fe-B film with the $(BH)_{\text{max}}$ value of 70 kJ/m^3 could be obtained. After polishing a Pr-Fe-B thick-film deposited on a glass substrate with groove, the magnetic properties of the film didn't deteriorate.

Index Terms—Pr-Fe-B film magnet, Glass substrate, PLD (Pulsed Laser Deposition), MEMS (Micro-Electro-Mechanical-Systems)

I. INTRODUCTION

Film magnets, such as a Nd-Fe-B, Sm-Co or Fe-Pt film, were prepared by a lot of researchers using several deposition methods [1]-[18]. Our group has focused on a PLD method to obtain isotropic Nd-Fe-B thick-film magnets with the maximum thickness up to 1250 μm on metal substrates (Ta, Fe, W) under the relatively high deposition rate of several-ten-microns per hour and applied the films to several miniaturized electronic devices [19]-[21].

Although a Nd-Fe-B film thicker than 5 μm on a Si substrate without a buffer layer was fabricated using a sputtering method, the film was peeled out from the substrate through the preparation process [22]. In order to suppress the mechanical deterioration, a Ta buffer layer between a Nd-Fe-B film and a Si substrate has been deposited and MEMS applications comprising the Nd-Fe-B films have been demonstrated using a micromachining technology [23][24]. On the other hand, the mechanical destruction of PLD-made Nd-Fe-B films on a Si substrate without a buffer layer showed a different phenomenon, namely the samples were broken from the inside of a single crystalline Si substrate (not peeling) [25]. We, therefore, prepared a 20 μm -thick Nd-Fe-B film magnet on a Si substrate with an approximately 1 μm -thick Ta buffer layer. The sample, however, was broken through a dicing process. We also confirmed that the use of the thick Ta layer was not suitable for a chemical etching process due to the ultimately low etching rate. In order to overcome the difficulty, we increased the Nd content of a PLD-made Nd-Fe-B thick-film by taking account of the linear expansion coefficient for each material and succeeded in enhancing the thickness of the film up to 160 μm on a Si substrate without a buffer layer [25].

This contribution reports the deposition of a rare-earth film magnet on a glass substrate, namely we focused on a glass MEMS [24] instead of a Si one. Furthermore, in order to enhance ~~coercive force~~ coercivity, we prepared isotropic Pr-Fe-B film magnets instead of previously reported Nd-Fe-B ones because the magnetic crystalline anisotropy constant of a $\text{Pr}_2\text{Fe}_{14}\text{B}$ phase ($K_u = 6.8 \text{ MJ}/\text{m}^3$) is larger by approximately 2.3 MJ/m^3 than that of a $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase ($K_u = 4.5 \text{ MJ}/\text{m}^3$)

[26]-[28]. In this study, an investigation on the magnetic and mechanical properties of Pr-Fe-B films deposited on glass substrates without a buffer layer was carried out. Moreover, we evaluated the magnetic properties after polishing a Pr-Fe-B thick-film magnet on a glass substrate with groove.

II. EXPERIMENTAL

Each film was deposited on a glass substrate (TEMPAX Float, Schott AG : 5 mm \times 5 mm square) using a Nd-YAG pulse laser (wave length : 355 nm, frequency : 30 Hz) in the vacuum atmosphere of approximately 10^{-5} Pa. The laser beam was defocused on the surface of a $\text{Pr}_x\text{Fe}_{14}\text{B}$ ($X=1.8, 2.0, 2.2, 2.4$) target. The laser energy density was estimated at 50 mJ/mm^2 . The distance between a target and a glass substrate was fixed at 10 mm in order to obtain the deposition rate of approximately 30 $\mu\text{m}/\text{h}$.

All the as-deposited films had amorphous structure, a pulse annealing (PA) method was carried out to obtain $\text{Pr}_2\text{Fe}_{14}\text{B}$ phase [29]. The annealing time range was from 3.9 to 4.3 s with an infrared furnace at output power of 8 kW, and then they were cooled down to room temperature. The estimated value of annealing temperature is considered to be higher than 823 K according to the previous result [30]. The magnetic properties of the samples were measured with a vibrating sample magnetometer (VSM) under the maximum applied magnetic field of 2.5 T after magnetizing each sample with a pulsed magnetic field of 7 T. All the films had isotropic magnetic properties, therefore in-plane ones were only shown in the paper. The film thickness was measured with a micrometer, and the composition of each film was analyzed with an energy dispersive X-ray spectrometry (EDX). The crystalline structure of each film was observed by using a XRD (X-ray diffraction). The cross sectional view was observed by using a SEM (Scanning Electron Microscope).

III. RESULTS AND DISCUSSION

A. Preparation of Isotropic Pr-Fe-B Film Magnets Using Pr-Fe-B Targets with Various Compositions on Glass Substrate

Figure 1 shows the relationship between coercivity and residual magnetic polarization of annealed isotropic Pr-Fe-B and Nd-Fe-B films deposited on glass or Ta substrates. The rare earth (Pr or Nd) amount of each film was controlled between 15.5 and 17.5 at. % by using two targets of $\text{Pr}_{2.4}\text{Fe}_{14}\text{B}$ [11] and $\text{Nd}_{2.4}\text{Fe}_{14}\text{B}$ [19], respectively. The thickness range was between 20 and 40 μm . Use of a Pr-Fe-B target instead of a Nd-Fe-B one enabled us to increase the coercivity due to the larger crystalline anisotropy of $\text{Pr}_2\text{Fe}_{14}\text{B}$ phase compared to $\text{Nd}_2\text{Fe}_{14}\text{B}$ one [26]. Although the magnetic properties of Pr-Fe-B films deposited on glass substrates scattered, we could obtain similar magnetic properties of Pr-Fe-B films on both substrates. The result is considered to be attributed that the both samples were mainly composed of $\text{Pr}_2\text{Fe}_{14}\text{B}$ phase as shown in Fig. 2. In this experiment, all the films had isotropic magnetic properties and the magnetic properties such as the coercive force were inferior than that those of previously-reported sputtering-fabricated anisotropic Nd-Fe-B [2] together with Pr-Fe-B [27][28] films. We have already prepared PLD-made anisotropic Nd-Fe-B films on metal substrates using a substrate heating system [21][31]. The preparation of anisotropic Pr-Fe-B films deposited on Si substrates is required as a future work. Furthermore, the magnetic properties of Pr-Fe-B films thicker than 20 μm on glass substrates were evaluated. (see Fig. 3) Coercivity decreased and residual magnetic polarization increased with

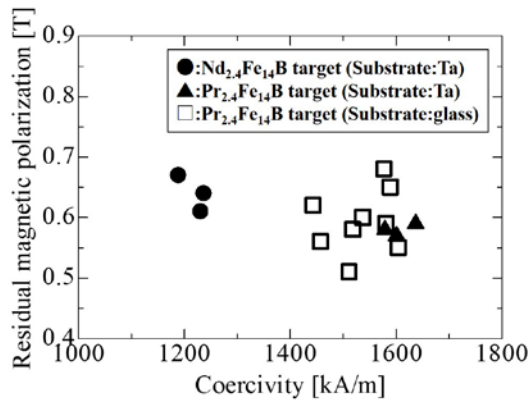


Fig. 1 Coercivity and residual magnetic polarization values of annealed Pr-Fe-B and Nd-Fe-B thick-films deposited on each substrate. The rare earth content range of all the films was from 15.5 to 17.5 at. %.

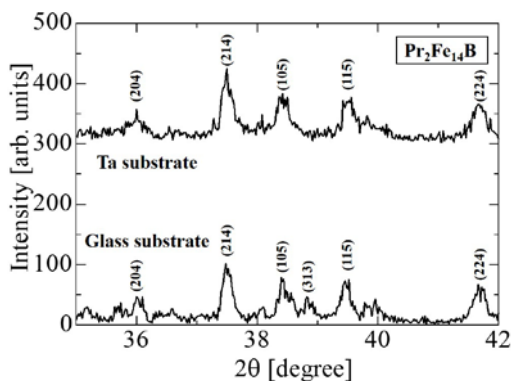
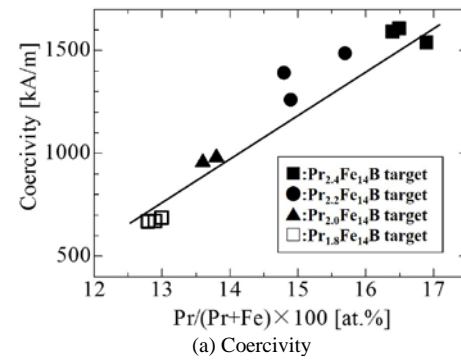
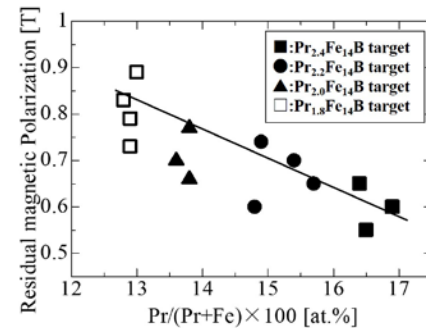


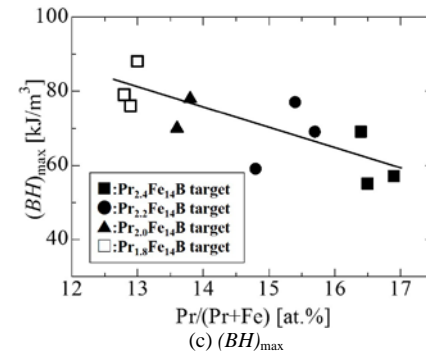
Fig. 2 X-ray diffraction patterns of Pr-Fe-B films deposited on glass and Ta substrates, respectively, using a $\text{Pr}_{2.4}\text{Fe}_{14}\text{B}$ target. All the diffraction peaks were corresponding to $\text{Pr}_2\text{Fe}_{14}\text{B}$ phase.



(a) Coercivity



(b) Residual magnetic polarization



(c) $(BH)_{\text{max}}$

Fig. 3 Coercivity, residual magnetic polarization and $(BH)_{\text{max}}$ values of annealed Pr-Fe-B thick-films deposited on glass substrates. Residual magnetic polarization reached up to approximately 0.8 T as $\text{Pr}/(\text{Pr}+\text{Fe})$ decreased down to approximately 13 at. %.

decreasing Pr contents of the samples. The residual magnetic polarization of approximately 0.8 T due to the reduction of Pr amount enabled us to obtain a Pr-Fe-B film with $(BH)_{\text{max}}$ above 70 kJ/m^3 on a glass substrate.

B. Enhancement in thickness of isotropic Pr-Fe-B film magnets deposited on glass substrates

In order to apply Pr-Fe-B films to MEMS, the thickness of Pr-Fe-B film magnets was enhanced by increasing the deposition time as shown in Fig. 4. The value of $\text{Pr}/(\text{Pr}+\text{Fe})$ for each film thicker than 50 μm varied from 13 to 15 at. %. It was confirmed that a mechanical destruction didn't occur in all the samples shown in Fig. 4. The phenomenon was different from the result of Nd-Fe-B films deposited on Si substrates [25]. We considered that it is attributed to the different structure of a glass (amorphous) and a Si substrate (a single crystalline structure).

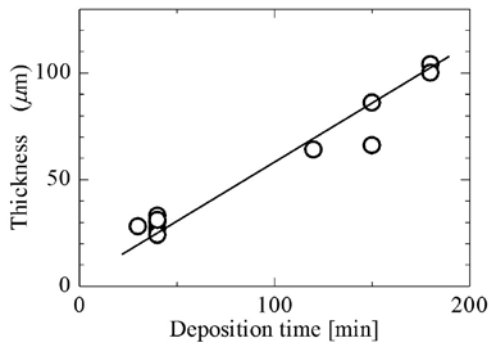


Fig. 4 Thickness of Pr-Fe-B films deposited on glass substrates as a function of deposition time. The deposition rate was estimated at about 30 $\mu\text{m}/\text{h}$.

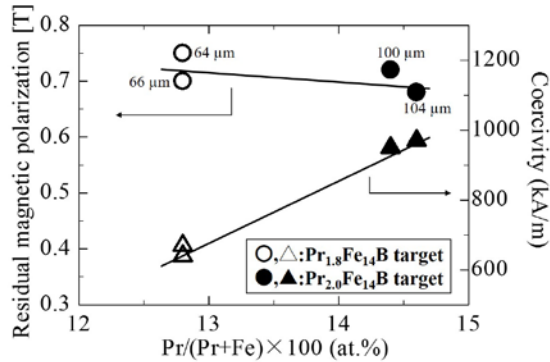


Fig. 5 Residual magnetic polarization (J_r) and coercivity (H_c) as a function of $\text{Pr}/(\text{Pr}+\text{Fe})$ in Pr-Fe-B film magnets thicker than 50 μm on glass substrates. J_r slightly decreased and H_c increased as the Pr contents increased.

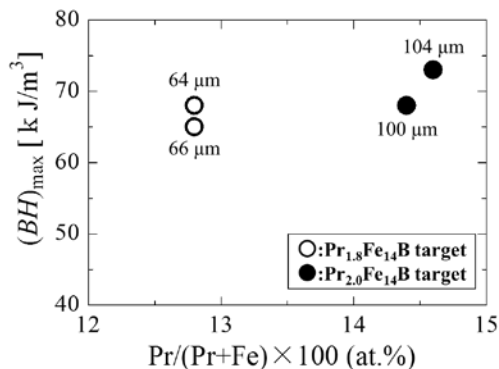


Fig. 6 Relationship between Pr contents and the $(BH)_{\text{max}}$ values of Pr-Fe-B films displayed in Fig. 5. $(BH)_{\text{max}}$ values of all the samples exceeded 60 kJ/m^3 .

Magnetic properties of Pr-Fe-B films thicker than 50 μm on glass substrates were displayed in Figs. 5 and 6. Increase in coercivity and slightly decrease in residual polarization occurred with increasing Pr contents. The $(BH)_{\text{max}}$ range of all the samples exceeded 60 kJ/m^3 . Figure 7 shows a J-H loop of an approximately 100 μm -thick Pr-Fe-B film. An observation on crystalline structure using an XRD revealed that the obtained hard magnetic properties is attributed to the formation of $\text{Pr}_2\text{Fe}_{14}\text{B}$ phase.

C. A 100 μm -thick Pr-Fe-B thick-film magnet deposited on glass mold substrates

Figure 8 (a) shows a Pr-Fe-B thick film magnet with the thickness of approximately 100 μm on a glass substrate with groove. Several vacancies due to the existence of large

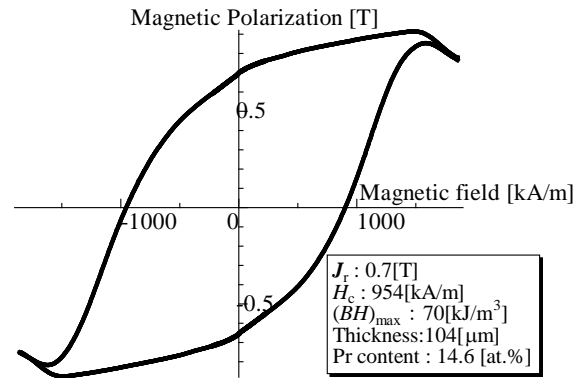


Fig. 7 A J-H loop of an approximately 100 μm -thick Pr-Fe-B film magnet with the $(BH)_{\text{max}}$ value of 70 kJ/m^3 .

aggregates emitted from the targets during the deposition could be observed. The depth and width of the grooves were 50 and 100 μm , respectively. As shown in Fig. 8 (b), the sample was polished in order to obtain the flat surface. The magnetic properties of each sample displayed in Figs. 8 (a) and (b) didn't deteriorate after the polishing. (see Fig. 9) We confirmed that the effect of above-mentioned large aggregates was little in the polishing process. Namely, it was clarified that the PLD method is effective to obtain a thick film magnet applicable to a glass MEMS.

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SUMMARY

In the study, isotropic Pr-Fe-B thick-film magnets could be prepared on glass substrates under the high deposition rate of 30 $\mu\text{m}/\text{h}$. The obtained results are as follows;

1. The obtained maximum thickness reached approximately 100 μm without mechanical destruction.
2. The $(BH)_{\text{max}}$ values of all the annealed Pr-Fe-B samples thicker than 50 μm exceeded 60 kJ/m^3 .
3. A Pr-Fe-B thick-film magnet prepared using large aggregates could be deposited on a glass substrate with groove. After polishing the sample, the magnetic properties didn't deteriorate.

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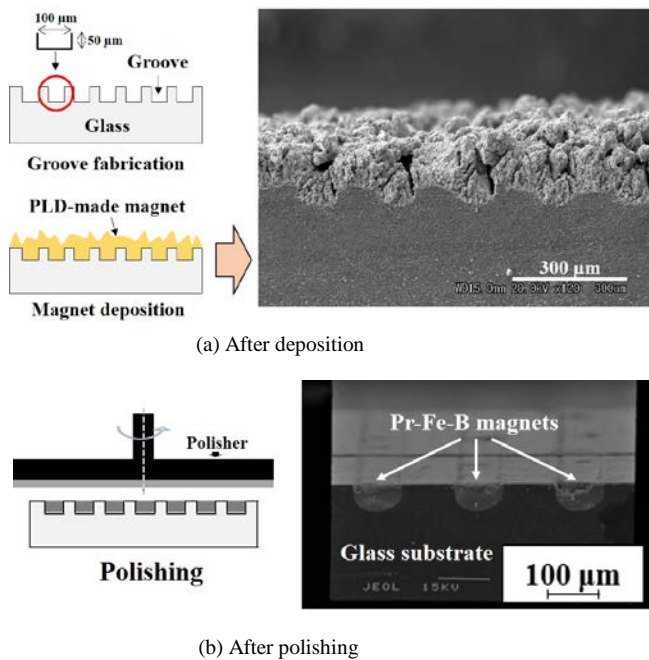


Fig. 8 Cross sectional view of a Pr-Fe-B thick-film magnets deposited on a substrate with groove (a) and the polished film (b).

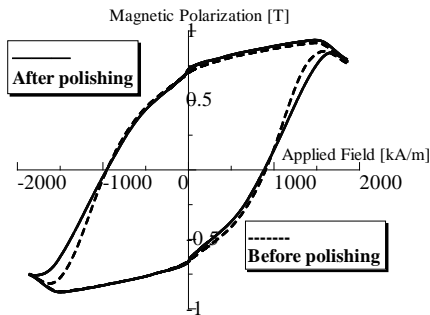


Fig. 9 J-H loop of a polished Pr-Fe-B film magnet with the thickness of approximately 100 μm on a glass substrate with groove. In order to compare the magnetic properties, a loop of the sample before polishing was also shown.

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