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Magnetic properties of Pr-Fe-B thick-film magnets deposited on Si substrates with glass buffer layer

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In order to improve the magnetic properties of PLD-made Pr-Fe-B thick-film magnets deposited on Si substrates, an adoption of a glass buffer layer was carried out. The glass layer could be fabricated under the deposition rate of approximately 70 μm/h on a Si substrate using a Nd-YAG pulse laser in the vacuum atmosphere. The use of the layer enabled us to reduce the Pr content without a mechanical destruction and enhance $(BH)_{max}$ value by approximately 20 kJ/m^3 compared with the average value of non-buffer layered Pr-Fe-B films with almost the same thickness. It is also considered that the layer is also effective to apply a micro magnetization to the films deposited on Si ones. © *2018 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).* https://doi.org/10.1063/1.5006988

I. INTRODUCTION

A microelectro–mechanical systems (MEMS) comprising an anisotropic Nd-Fe-B thick-film magnet has already been reported by a lot of researchers.[1–6] Although the magnetic properties of all the anisotropic Nd-Fe-B ones were excellent, the thickness of each film was mainly less than 20 μm. From a demagnetization field point of view, an increase in thickness of a film magnet is necessary to supply a sufficient magnetic field. Our group prepared isotropic Nd-Fe-B thick-film magnets with the thickness range from 10 to 160 μm on Si substrates using a PLD (Pulsed Laser Deposition) method with the deposition rate of several-ten-microns per hour.[7] On the other hand, in order to obtain a Nd-Fe-B film magnet thicker than 20 μm without a mechanical destruction during an annealing process, it was confirmed that the large amount of Nd above 15 at. % is required and subsequently $(BH)_{max}$ value showed only 40 kJ/m^3.[7] To overcome the difficulty, we demonstrated that the insert of a Nd-rich Nd-Fe-B buffer layer between a film magnet and a Si substrate enabled us to reduce the total amount of Nd and enhance the $(BH)_{max}$ value up to approximately 55 kJ/m^3. The obtained result is considered to be attributed that the thermal expansion coefficient of Nd (9.6×10^{-6} K^{-1}) is close to the value of Si (2.6×10^{-6} K^{-1}) compared with that (14.7×10^{-6} K^{-1}) of $Nd_2Fe_{14}B$.[8] In addition, we confirmed that the use of a glass substrate (10.0×10^{-6} K^{-1}) instead of a Si one is effective to reduce the amount of rare earth (Pr).[9]

In the study, we focused on the adoption of a glass buffer layer between a Pr-Fe-B film magnet and a Si substrate. The method suggests that the glass layer is more applicable to a micro magnetization[10] compared with the above-mentioned a Nd-rich Nd-Fe-B layer from a thermal conductivity point of view. This contribution reports that the use of a glass layer enabled us to reduce the amount of Pr in Pr-Fe-B thick-film magnets and enhance the $(BH)_{max}$ by above

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20 kJ/m^3 compared with the average value of Pr-Fe-B films deposited on non-buffer layered Si ones.

II. EXPERIMENTAL PROCEDURE

A rotated target was ablated using a Nd-YAG pulse laser (wave length: 355 nm, frequency: 30 Hz) in the vacuum atmosphere of approximately 10^{-5} Pa. In order to prepare a glass buffer layer on a Si substrate (5 mm × 5 mm square) using a Nd-YAG pulse laser in the vacuum atmosphere, a glass plate (MATSUNAMI GLASS IND., LTD, S1112) on a bulk metal was used as a target. Furthermore, a Pr-Fe-B thick-film magnet was fabricated using a $Pr_XFe_{14}B$ (X=1.8 ~ 2.8) target on a Si substrate with or without a glass layer. The laser beam was defocused on the surface of each target using *DF rate* = 0.1 (glass target) and *DF rate* = 0.3(Pr-Fe-B target), respectively[11] using the distance between a target and a Si substrate of 10 mm to exceed the deposition rate of several ten microns per hour. A pulse annealing (PA) was carried out after all the deposition to crystallize an as-deposited Pr-Fe-B film with amorphous structure,[12] and the annealing time was approximately 3.7-4.1 s with an infrared furnace at output power of 8 kW, and then they were cooled down to room temperature. 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 thickness of each film was measured with a micrometer and the compositions of each film were analyzed with an X-ray photoelectron spectroscopy (XPS) for an glass layer and an energy dispersive X-ray spectrometry (EDX) for a Pr-Fe-B film, respectively.

III. RESULTS AND DISCUSSION

A. Preparation of a glass buffer layer

Figs. 1(a) and (b) show the thickness of glass films prepared on Si substrates using the abovementioned PLD method as a function of deposition time and the surface morphology of a 18 µm-thick glass film. The deposition rate was estimated at approximately 70 µm/h, and it was clarified that glass thick-films could be obtained under the relatively high deposition rate. As shown in Fig 1(b), the existence of large particles with the size of several microns could be observed. Namely, the high deposition rate is considered to be attributed to the emission of the particles from a glass target. Figs. 2(a) and 2(b) show the composition of a glass film and a glass target

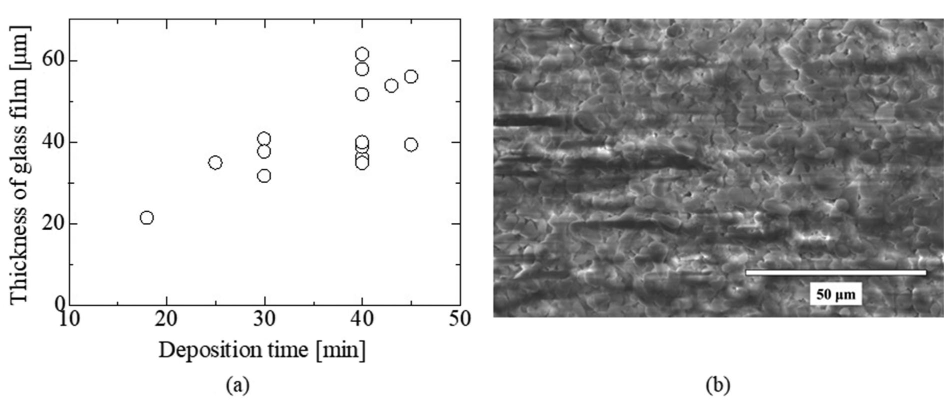

FIG. 1. Glass buffer layer prepared using PLD. Deposition rate was estimated at approximately 70 µm/h by taking advantage of the emission of large particles from a target. (a) Thickness of PLD-fabricated glass films as a function of deposition time. (b) Surface morphology of a 18 µm-thick glass film prepared using PLD.

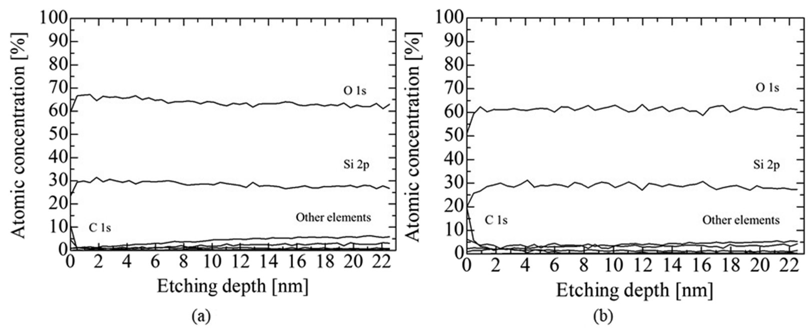

FIG. 2. Comparison of each composition for a glass target and a glass film, respectively, as a function of an etching depth. (a) Composition of a glass target. (b) Composition of a glass film prepared using PLD.

as a function of an etching depth, respectively, and it was confirmed that the both compositions showed similar tendency from each surface. These results suggest that we could achieve a good transfer composition from a target to a corresponding glass film under the high deposition rate.

B. Various properties of Pr-Fe-B thick-film magnets deposited on Si substrates with a glass buffer layer

In the section, we prepared Pr-Fe-B thick-films with a glass buffer layer on Si substrates. Figs. 3(a), (b) and (c) show the relationship between the breakdown phenomenon and the existence of a glass buffer layer in the film magnets with various thicknesses. Here, the thickness of the glass layers varied from 13 to 61 μm. As displayed in Fig. 3(a), the non-buffer layered samples

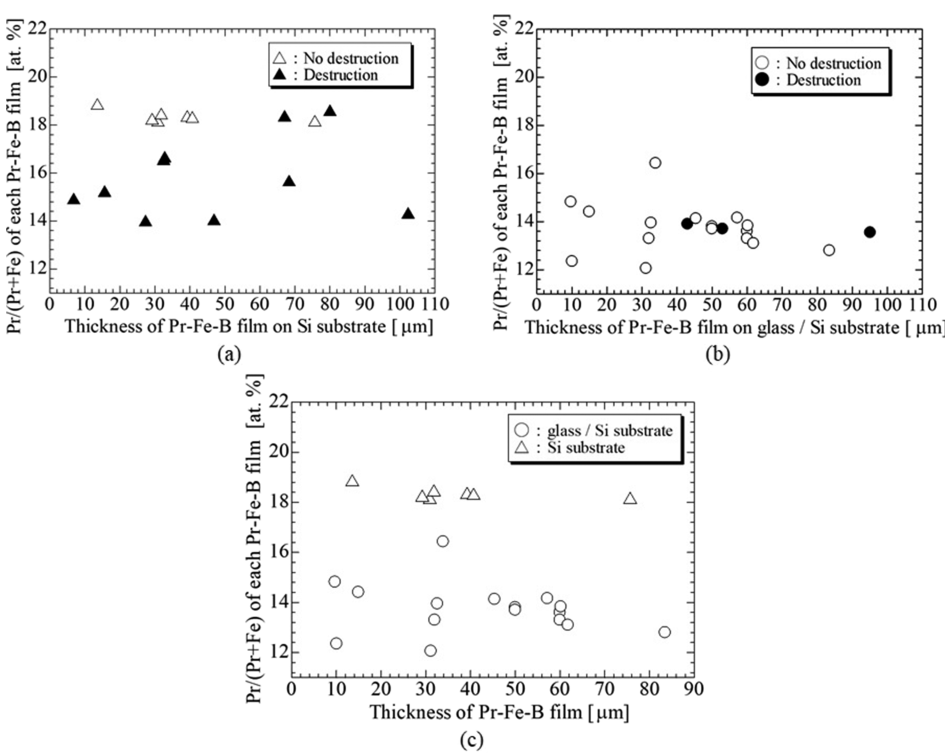

FIG. 3. Effect of glass buffer layer on mechanical destruction for Pr-Fe-B films deposited on Si substrates. (a) Non-buffer layered Pr-Fe-B film magnets. (b) Pr-Fe-B film magnets with a glass buffer layer. (c) Comparison of non-destroyed samples displayed in Figs. 3(a) and (b).

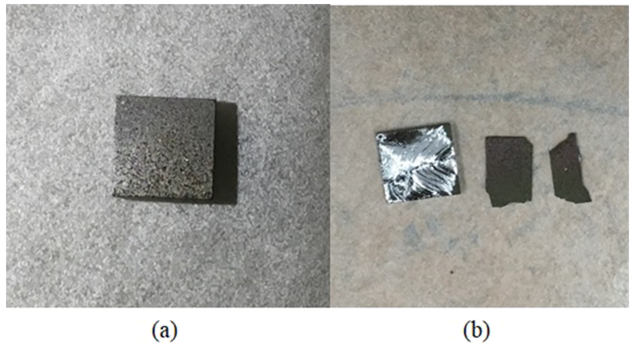

FIG. 4. Photos of a broken and a non-broken film through a post annealing process. The destruction occurred from the inside of a Si substrate. (a) No destruction (b) Destruction.

with Pr content less than 17 at. % were broken during an annealing process. We confirmed that all the destruction occurred from the inside of each Si substrate as shown in Fig. 4(b), which indicates that the samples had strong adhesion.[7] As seen in Fig. 3(b), the samples including the similar Pr contents with a glass layer could suppress the mechanical deterioration. Fig. 3(c) summarizes the non-destroyed samples displayed in Figs. 3(a) and 3(b). The use of a glass buffer layer is effective to maintain the shape in Pr-Fe-B thick-film magnets with Pr/(Pr+Fe) below 15 at. %. Figure 5 shows the effect of the glass layer on the breakdown phenomenon in various thicknesses of Pr-Fe-B film (Pr: 12.8 – 14.8 at.%). Increase in the thickness of a glass layer leaded to increase the thickness of Pr-Fe-B films without the destruction. We could obtain an approximately 80 μm-thick Pr-Fe-B thick film magnet with Pr content less than 15 at. % by taking advantage of an approximately 60 μm-thick glass layer.

Figure 6(a) show the relationship between coercivity and residual magnetic polarization of samples which were not broken through the pulse annealing (see Fig. 3(c)). The values of residual magnetic polarization of films with a buffer layer were higher by approximately 0.2 T than those of the samples without the layer. The tendency was opposite in the case of coercivity. It is considered that the obtained magnetic properties are attributed to the reduction in Pr contents as shown in Fig. 3(c). Resultantly, $(BH)_{max}$ values could be enhanced by above 20 kJ/m^3 using a glass buffer layer compared with those of non-buffer layered films as displayed in Fig. 6(b). An inset of Figure 6(b) shows a J - H loop of a film with $(BH)_{max}$ of approximately 65 kJ/m^3 consisting of a 60 μm-thick Pr-Fe-B film, a 47 μm-thick glass buffer layer, and a Si substrate. The value was also superior to those of previously reported Nd-Fe-B thick-films with a Nd-rich Nd-Fe-B buffer layer.[8] It is generally considered that we have difficulty in applying a micro magnetization technique to a thick-film magnet without a glass buffer layer deposited on a Si substrate because Si has a high

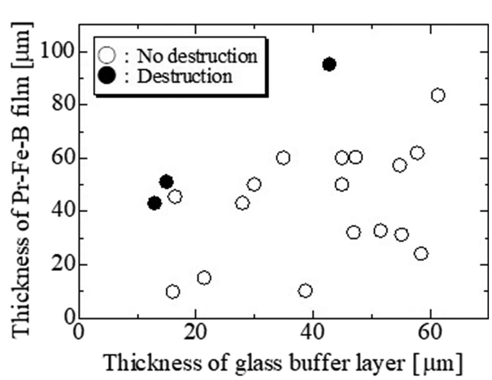

FIG. 5. Effect of thickness of glass buffer layer on mechanical destruction for Pr-Fe-B films on Si substrates.

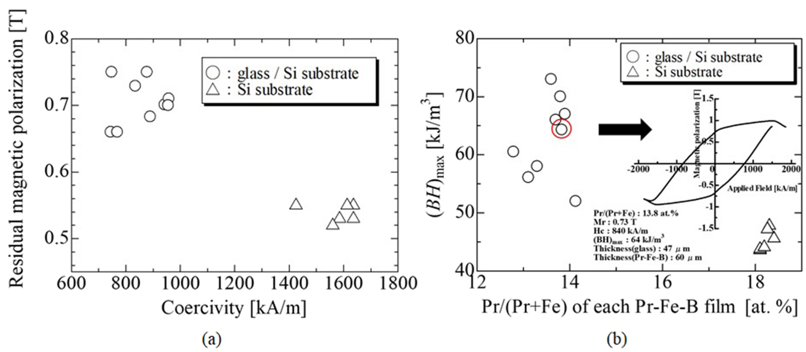

FIG. 6. Magnetic properties of Pr-Fe-B films with and w/o a glass buffer layer on Si substrates. The inset of FIG. 5(b) shows a J-H loop with $(BH)_{max}$ value of approximately 65 kJ/m^3 which was superior to those of non-buffer layered Pr-Fe-B thick-film magnets on Si substrates. (a) Relationship between coercivity and residual magnetic polarization of Pr-Fe-B films with and w/o a glass buffer layer. (b) Relationship between (BH)max and Pr content of Pr-Fe-B films with and w/o a glass buffer layer.

thermal conductivity.[10] Namely, these results indicate that a film magnet with a glass buffer layer on a Si substrate is a promising material applied to MEMS compared with previously reported PLD-made samples.[7,8]

IV. CONCLUSION

In the study, we investigated the effect of a glass buffer layer on the mechanical phenomenon together with magnetic properties of PLD-made Pr-Fe-B films on Si substrates. The obtained results are summarized as follows;

(1) The use of a glass layer enabled us to reduce the Pr content without a mechanical destruction and enhance $(BH)_{max}$ value compared with the average value of non-buffer layered Pr-Fe-B films with almost the same thickness deposited on Si ones.

(2) The use of a glass layer has a possibility to apply a micro magnetization technique to thick-film magnets deposited on Si substrates.

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