Preparation and deposition of Pr-Fe-B permanent magnet powder using pulsed laser

M. Nakano, S. Takeichi, K. Takashima, A. Yamashita, T. Yanai, and H. Fukunaga
Graduate School of Engineering, Nagasaki University, Nagasaki 852-8521, Japan

We have already prepared a thin permanent magnet with the thickness of sub millimeter by obtaining magnet powders using a PLD (Pulsed Laser Deposition) method. In the study, the PLD followed by a flash annealing enabled us to deposit isotropic Pr-Fe-B magnet powders with coercivity (H_{cj}) > 1000 kA/m on a stainless thin shaft applicable to a miniaturized motor. Observation on the surface of Pr-Fe-B magnets and evaluation on mechanical behavior was carried out. Since the surface of a Pr-Fe-B magnet was coated by a Pr oxide through an annealing process, their magnetic properties didn't degrade after one year. We also confirmed that the Pr-Fe-B magnet has possibility to be applied to a micro magnetization process. It was clarified that the powder technology using the PLD is useful to propose a thin magnet applicable to a next generation small motor.

Index Terms— PLD (Pulsed Laser Deposition), Pr-Fe-B permanent magnet powder, stainless shaft, coating

I. Introduction

A miniaturized permanent magnet is useful to develop the size reduction of devices in industrial and medical fields [1-3]. Although thinned Nd-Fe-B magnets prepared using a sputtering method have been applied to small electronic devices [4-8], we have difficulty obtaining small Nd-Fe-B magnets with the thickness of sub millimeter due to the low deposition rate. In order to overcome the difficulty, our group has used a PLD method with high deposition rate which is attributed to Nd-Fe-B magnet powders with the size of several microns [9]. Furthermore, small motors comprising sub-millimeter-thick PLD-made Nd-Fe-B magnets such as an axial gap type DC brushless motor and a friction drive motor have been demonstrated.

According to the demagnetization field due to the structure of a small motor aimed at development in future research, increase in coercivity (H_{cj}) up to 1000 kA/m is required because permeance coefficient (P_c) in a demagnetization curve is estimated at 1.0 [10]. Since the magnetic crystalline anisotropy constant of a $Pr_2Fe_{14}B$ phase (K_u= 6.8 MJ/m^3) is larger by approximately 2.3 MJ/m^3 than that of a $Nd_2Fe_{14}B$ phase (K_u= 4.5 MJ/m^3), Pr-Fe-B magnets instead of Nd-Fe-B ones were adopted in the study [11].

This contribution reports the deposition of Pr-Fe-B magnet powders with coercivity (H_{cj}) exceeding 1000 kA/m on a thin stainless shaft. Moreover, investigation on various properties was carried out to apply the PLD-made magnet to a miniaturized motor in future.

II. Experimental Procedure

A $Pr_{2.2}Fe_{14}B$ target was ablated with a Nd-YAG pulse laser (wavelength: 355 nm) at the repetition rate of 30 Hz in vacuum atmosphere. The laser power, which was measured with a power meter in front of the entrance lens of a chamber, was approximately 4 W. Before the ablation, the chamber was evacuated down to approximately 2.0~4.0×10^{-5} Pa by using a rotary pump together with a molecular turbo one. The distance between a target and a stainless shaft (SUS420J2) was fixed at 10 mm. As shown in Fig. 1, A target together with a shaft were rotated during the deposition. In some experiments, Pr-Fe-B powders were deposited on 100 μm-thick stainless (SUS420J2) plates. Average deposition rate exceeded approximately 40 μm/h. A pulse annealing (PA) [12] was carried out to crystallize the as-deposited sample.

After the annealing process, samples were magnetized up to 7 T with a pulse magnetizer. Then, J-H loops were measured with a VSM (Vibrating Sample Magnetometer) which could apply a magnetic field up to approximately 2000 kA/m reversibly. The composition of obtained films was evaluated by a SEM (Scanning Electron Microscope)-EDX (Energy Dispersive X-ray spectrometry), and the surface observation was also carried out by using a SEM. An average thickness was measured with a micrometer or estimated by measuring each weight.

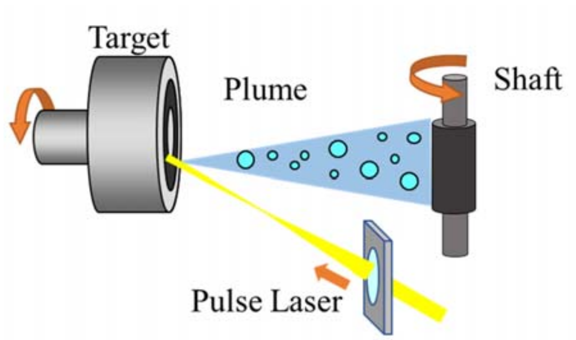


Fig. 1 Schematic diagram of the deposition of Pr-Fe-B powders using a pulsed laser on a thin stainless shaft with the diameter of approximately 0.5 mm.

III. Results and Discussion

A. Deposition of Pr-Fe-B magnet powders with high coercivity on a thin stainless shaft

Figure 2 shows photos of an isotropic ring-shaped Nd-Fe-B bonded magnet used for a conventional small motor. Overview photo of the 350 μm-thick bonded magnet was displayed as an inset. Surface observation indicated that Nd-Fe-B magnet flakes were bonded using a nonmagnetic phase of thermosetting resin. Comparison of magnetic properties between the conventional bonded magnet and a PLD-made Pr-Fe-B magnet (Pr content=Pr/(Pr +Fe) : approximately 15 at. %) on a stainless plate was carried out as shown in Fig. 3. The both values of coercivity and residual magnetic polarization of the PLD-made magnet were superior than those of the bonded one. This result

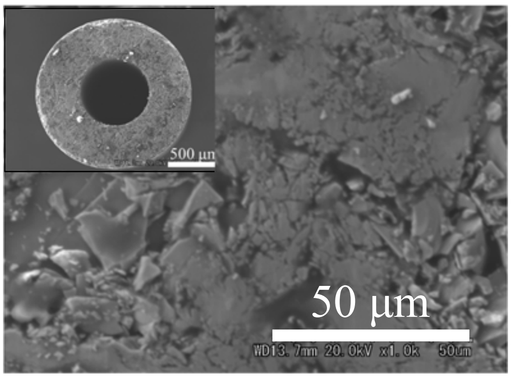


Fig. 2 Photos of a conventional ring-shaped Nd-Fe-B magnet. An overview photo was inserted. As shown in the surface observation, Nd-Fe-B magnet flakes were dispersed in thermosetting resin.

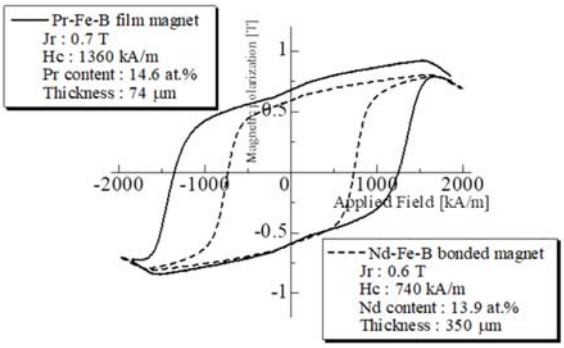


Fig. 3 Two J-H loops of a conventional ring-shaped Nd-Fe-B bonded magnet shown in Fig. 2 together with a PLD-made Pr-Fe-B magnet on a stainless plate. Pr-Fe-B magnet had superior magnetic properties compared with the bonded one.

indicated that the consolidation of the Pr-Fe-B magnet powders using a pulsed laser without nonmagnetic phase was effective to obtain the higher residual magnetic polarization. We, therefore, deposited Pr-Fe-B powders with the same Pr content of approximately 15 at. % on a thin stainless shaft with the diameter of approximately 0.5 mm. (see Figs. 4 and 5 (a)) Mechanical deterioration such as peeling phenomenon didn't occur after a deposition and an annealing process. In addition, the polish of the sample shown in Fig. 5 (b) enabled us to reduce the thickness down to 250 µm and improve the surface roughness as displayed in Fig. 5 (b).

B. Investigation on change with time and thermal exposure of magnetic properties in PLD-fabricated Pr-Fe-B magnet

Evaluation on magnetic properties of PLD-fabricated Pr-Fe-B magnets over one year was carried out to consider a practical application. A Pr-Fe-B magnet with Pr content of approximately 15 at. % on a stainless plate was hold in a desiccator with the vacuum of approximately 1 Pa and frequently exposed to the air more than 100 times through one year. Figure 6 shows the change with time of 2nd quadrant of *B-H* curves in the Pr-Fe-B magnet. A curve of the above-mentioned bonded magnet was also displayed [10]. The *B-H* curves of each Pr-Fe-B one were completely overlapped. It was clarified that magnetic properties didn't degrade after one year passed. In order to examine the mechanism, the surface of the

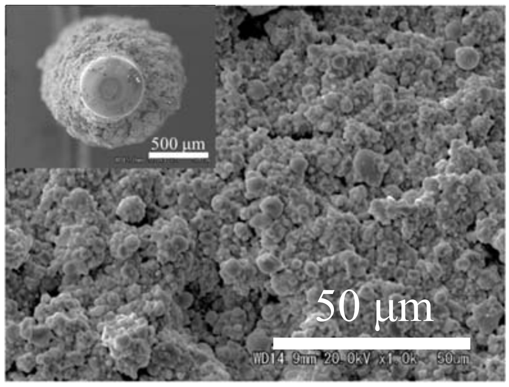
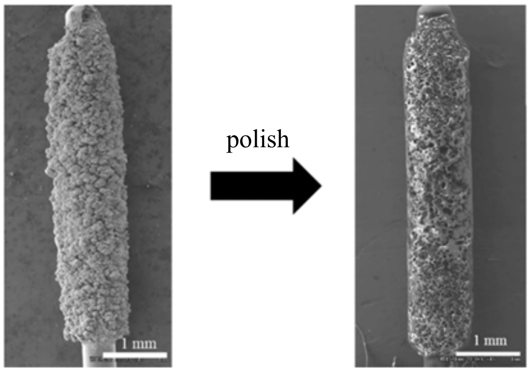


Fig. 4 Schematic diagram of the deposition of Pr-Fe-B powders on a thin stainless shaft. A cross-sectional overview photo was inserted. As shown in the surface observation, Pr-Fe- B magnet powders were consolidated without resin.



(a) after annealing (b) after polishing

Fig. 5 Polish was carried out to reduce the thickness less than 300 µm and improve the surface roughness of an annealed Pr-Fe-B magnet on a stainless shaft.

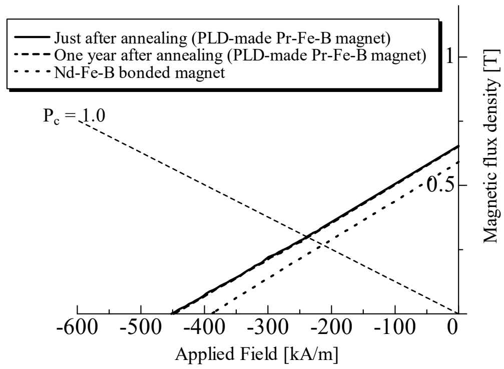
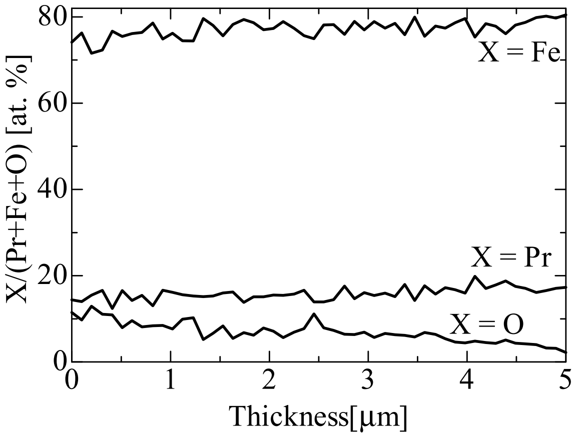
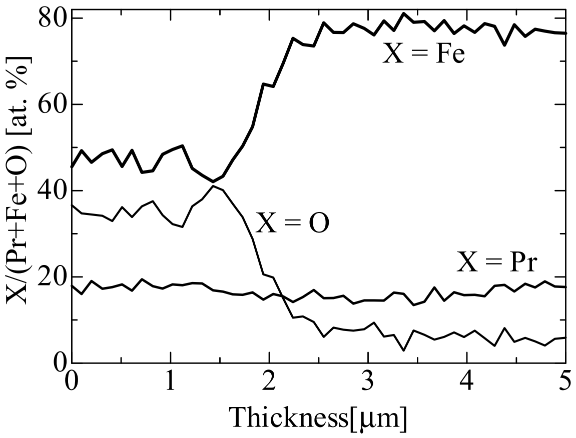


Fig. 6 2nd quadrant of *B-H* curves of a Pr-Fe-B magnet on a stainless plate and a conventional Nd-Fe-B bonded magnet, respectively. After one year passed, the curve of Pr-Fe-B one overlapped the original one.

sample was observed by using a SEM-EDX as shown in Fig. 7. The horizontal axis means the depth from the surface. The observation revealed that an approximately 2 µm-thick Pr oxide covered the surface of the sample after an annealing process. We suppose that an Pr-Fe-B film was naturally coated by the Pr oxide through an annealing process and the oxidation layer suppressed the deterioration of magnetic properties. In future,



(a) Before annealing (as-deposited)



(b) After annealing

Fig. 7 Composition analysis using SEM-EDX in an as-deposited and an annealed Pr-Fe-B magnet on a stainless plate, respectively. It was confirmed Pr oxide layer was formed in the surface with the thickness of approximately 2 μm through an annealing process.

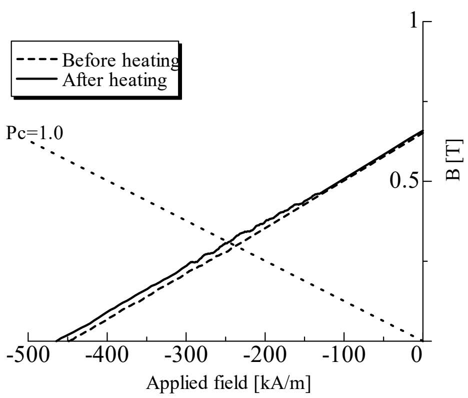


Fig. 8 2nd quadrant of *B-H* curves of a Pr-Fe-B magnet on a stainless plate before and after a heat treatment at 573 K in the air. The two curves were almost the same.

same observation for a magnet on a thin shaft is required.

Although it has already known that a micro magnetization method is useful to comprise an isotropic (Nd or Pr)-Fe-B magnet to a small motor [13], the magnet is needed to be heated up to approximately 573 K in the air. Figure 8 shows the *B-H* curves of a PLD-made Pr-Fe-B magnet with Pr content of approximately 15 at. % on a stainless plate before and after the heat treatment. Here, the annealed sample was re-magnetized with a pulse magnetizer. The *B-H* curves of each Pr-Fe-B one were almost the same. The magnetic properties of the PLD-made magnet didn't degrade after heating at 573 K in the air. It was found that an adoption of micro magnetization for the magnet is hopeful as a future work.

IV. Conclusion

Preparation of Pr-Fe-B powders using a PLD enabled us to obtain an isotropic magnet with coercivity (H_{cj}) > 1000 kA/m on a stainless thin shaft without mechanical deterioration. It was also confirmed that each change with time and thermal exposure of magnetic properties is little in the Pr-Fe-B magnets on a stainless plate. These results indicated that the powder technology using the PLD is effective to propose a next generation thin magnet.

Acknowledgment

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