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ABSTRACT

In this research, Fe-Pt thin sheets thicker than 10 microns with Fe contents ranging from 50 to 60 at. % were prepared. Isotropic Fe-Pt thin sheets could be obtained by taking advantage of the exfoliation behavior after depositing Fe-Pt films on Si substrates using a laser ablation technique. A post-annealing process was used to obtain the $L1_0$ phase, and the $(BH)_{max}$ value of Fe-Pt thin sheets showed approximately 70 kJ/m^3. Moreover, the test of a cantilever containing the obtained Fe-Pt thin sheet showed good mechanical characteristics.

I. INTRODUCTION

Since miniaturization of (Fe or Co)-Pt magnets with excellent biocompatibility is a hopeful material to develop the performance of small medical devices,[1] a lot of researchers prepared isotropic (Fe or Co)-Pt thick-film magnets with the thickness above several microns by sputtering, electrodeposition, and laser ablation technique.[2–9] Moreover, isotropic Fe-Pt thin sheets magnets with a thickness range from 10 to 100 microns were prepared using cold rolling and rapidly quenching technique.[10–12] The use of Fe-Pt thin sheets does not need to optimize a substrate for each application. However, it is generally hard to improve $(BH)_{max}$ by controlling the microstructure compared with Fe-Pt films prepared by the deposition methods mentioned above. For example, J. P. Liu reported synthesis of a nano-composite Fe-Pt/α-Fe film with excellent magnetic properties using sputtering technique.[13] In the study, a laser ablation technique was adopted to obtain Fe-Pt thin sheets by peeling Fe-Pt thick films from a Si substrate. As future work, we pay attention to the preparation of nano-composite Fe-Pt/α-Fe multilayered thin sheets because we have demonstrated rare-earth nano-composite films such as Nd-Fe-B/α-Fe and Sm-Co/α-Fe using the laser ablation technique.[14,15]

This contribution reports the possibility of preparing thin bulk magnets through the deposition process. In detail, an investigation on magnetic properties together with mechanical characteristics of Fe-Pt thin sheets was carried out.

II. EXPERIMENTAL PROCEDURE

A Fe-Pt target ($Fe_{70}Pt_{30}$ or $Fe_{60}Pt_{40}$) was ablated with a Nd-YAG pulse laser (wavelength: 355 nm) at a repetition rate of 30 Hz in a vacuum.[16] The laser power was approximately 4 W. Before the ablation, the chamber was evacuated down to about 2.0~4.0$\times 10^{-5}$ Pa using a rotary pump together with a molecular turbo pump. The distance between the target and a Si substrate used in the chamber was fixed at 10 mm. The area of all films obtained on the substrate was 5$\times$5 mm^2. In the deposition, laser energy density (LED) varied by a spot size of laser beam, which could be controlled by moving the distance between a focal lens and a target intentionally. The range of LED above 10 J/cm^2 was used under fixing the laser power of 4 W, which was measured with a power meter in front of the entrance lens of a chamber. Under the deposition condition, it was confirmed that the average composition of each film (5$\times$5 mm^2)

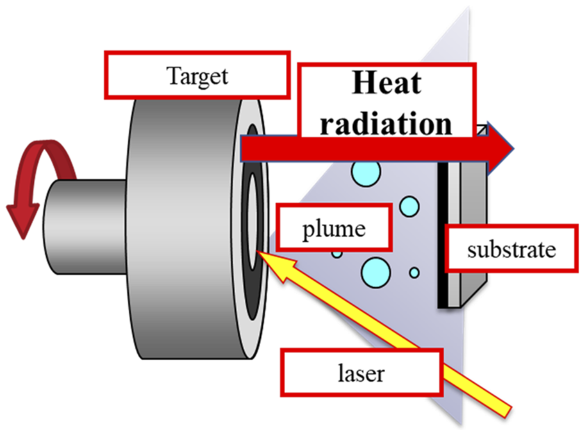

FIG. 1. Schematic diagram of a laser ablation technique to obtain a Fe-Pt thin sheet after deposition. Film and substrate were heated due to heat radiation from a target.

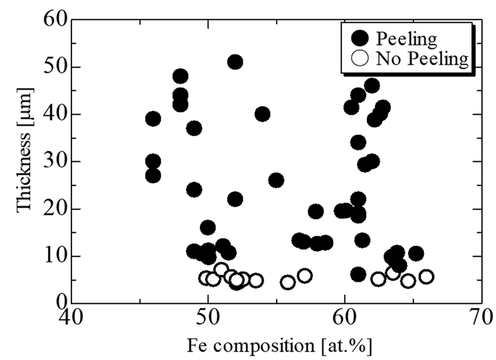

FIG. 2. Conditions of obtaining Fe-Pt thin sheet magnets through a peeling process using a laser ablation technique. When thickness exceeded approximately 7 μm, exfoliation behavior tended to occur.

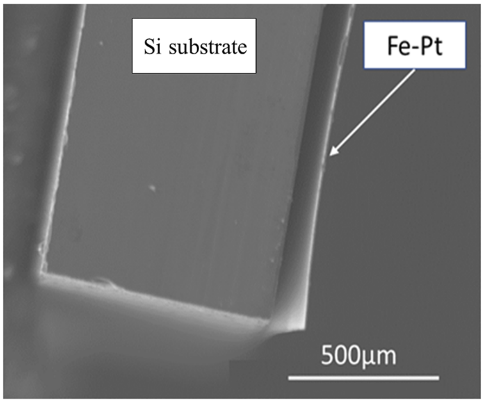

FIG. 3. Cross-sectional photo of an as-deposited $Fe_{50}Pt_{50}$ thin sheet and a Si substrate. The thickness of the sheet was approximately 10 μm.

depended on the position of a substrate. Although the temperature of substrate (Ta) in the previous experiment[16] was measured with a temperature-detecting tape commercially called "THERMO LABEL," the temperature of Si substrate has not been measured correctly in the present stage. It is presumed that a Fe-Pt film and a Si substrate were heated during the deposition because of heat radiation from the target based on the previous result[16] (Figure 1). As future work, the detection of substrate temperature is required.

A peeled Fe-Pt sheet from a substrate could be obtained after the deposition. To transform a disordered A1 (fcc) structure of Fe-Pt crystalline phase in the peeled sheets to an ordered $L1_0$ (fct) one, we annealed them at the temperature ranging from 573 to 973 K using an electric furnace (FULL-TECH, FT-01 VAC-30). The temperature was ramped from room temperature to the annealing temperature at the constant heating rate of 100 K/min, and then kept at constant for 30 min under a vacuum ($<4.0 \times 10^{-3}$ Pa). Finally, the samples were cooled down to room temperature.

After the annealing process, samples were magnetized up to 7 T with a pulse magnetizer. Hysteresis loops were measured with a VSM (Vibrating Sample Magnetometer) which could apply a magnetic field up to approximately 2000 kA/m reversibly. The in-plane magnetic field was applied to the substrate. The composition of the obtained films was evaluated with SEM (Scanning Electron Microscope)-EDX (Energy Dispersive X-ray spectrometry), and the film surface was also observed using an SEM. The average film thickness was measured with a micrometer.

III. RESULT AND DISCUSSION

A. Exfoliated Fe-Pt thin sheets from Si substrates

Figure 2 shows the exfoliation behavior in Fe-Pt films with various thicknesses and compositions after the deposition shown in Figure 1. For the deposited film with the Fe composition ranging between 45 and 65 at. %, the thickness was a dominant parameter for the exfoliation compared with the other compositions. All the Fe-Pt sheets shown in Figure 2 (●: peeling) could be fabricated through the exfoliation without defects (Figure 3). For example, it was observed that a 51 μm-thick $Fe_{51}Pt_{49}$ thin sheet (an as-deposited $Fe_{51}Pt_{49}$ film peeled from a Si substrate) was not brittle. It is generally known that an increase in the thickness enhances the internal stress of a film. As written in Experimental Procedure, it is presumed that both a sample and a substrate were heated during the deposition. Through the thermal exposure, the internal stress due to the large difference of linear expansion coefficient ($Fe_{50}Pt_{50}$ phase: 12×10^{-6} 1/K, Si: 2.6×10^{-6} 1/K) occurred. Moreover, Komine *et al.* reported about the weak adhesion between a Pt film and a Si substrate.[17] The exfoliation is probably attributed to the fact that the internal stress exceeded the adhesion between the film and the substrate as the thickness of a Fe-Pt film increased. Therefore, an increase in the thickness of the deposited Fe-Pt films beyond a certain thickness (approximately 7 μm) enabled the authors to obtain Fe-Pt sheets.

B. Magnetic properties and crystalline structure of Fe-Pt thin sheets

A 24 μm-thick $Fe_{49}Pt_{51}$ sheet did not show hard magnetic properties (Figure 4). It was confirmed that almost all the releasable samples displayed in Figure 2 had similar magnetic properties. As

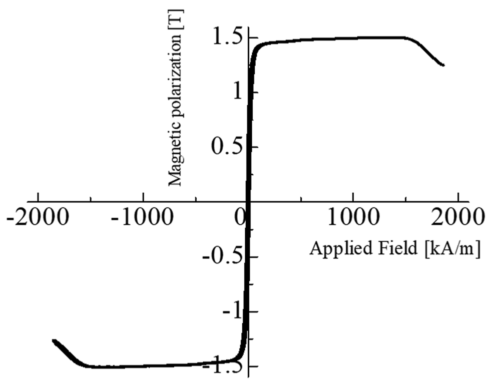

FIG. 4. J-H loop of a $Fe_{49}Pt_{51}$ sheet with the thickness of approximately 24 μm. After the deposition, a peeled thin sheet had soft magnetic properties.

displayed in Figure 5, observation on the crystalline structure of an as-deposited film shown in Figure 4 revealed the existence of the soft magnetic phase (disorder phase). Moreover, the maximum magnetic polarization at the applied magnetic field of 1600 kA/m was approximately 1.5 T. Our previous experiment indicated the maximum magnetic polarization of $Fe_{50}Pt_{50}$ fcc phase is approximately 1.5 T.[18] Based on both results in Figures 4 and 5, it was difficult to form fct structure in the peeled Fe-Pt sheets. The phenomenon mentioned above did not agree with that of the deposition of a Fe-Pt film on a Ta substrate.[13] In order to obtain hard magnetic properties of Fe-Pt thin sheets, the order-disorder transformation was carried out using post-annealing. Figure 6 shows the X-ray diffraction patterns of Fe-Pt sheets after annealing at various temperatures for 30 min. Fe compositions varied from 48 to 51 at. %. An increase in annealing temperature enabled us to obtain ordered phase such as fct (002) and fct (112) planes. Figure 7 shows the coercivity values of Fe-Pt sheets after annealing at each temperature. Moreover, the J-H loop of a sample annealed at 873 K was displayed in Figure 8. Since the hysteresis loop did not have a knick, it is possible that the large amount and/or volume of soft magnetic phase (fct) was not included in the sample. Although we could observe the transformation from fcc to fct phase, the obtained magnetic properties were inferior to those of Fe-Pt thin sheets reported by other groups.[10–12] Optimization of deposition and annealing conditions are required as future work. In particular, we have to focus on the different experimental

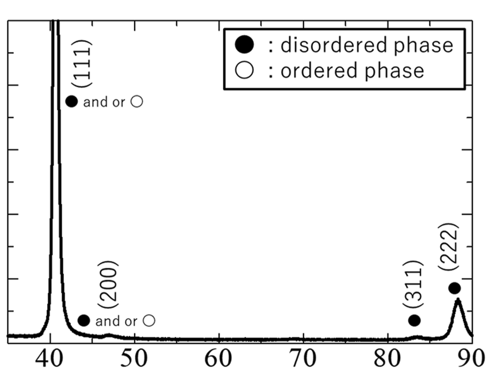

FIG. 5. X-ray diffraction pattern for a $Fe_{49}Pt_{51}$ sheet shown in Fig. 4. We had difficulty forming ordered phase through the heat radiation from a target during the deposition.

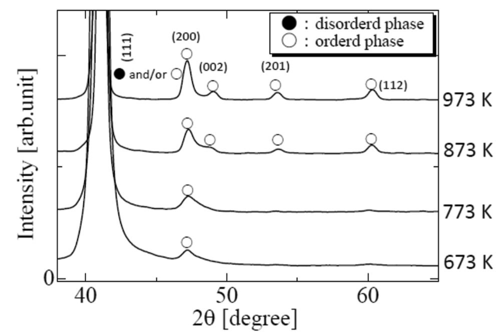

FIG. 6. X-ray diffraction patterns of annealed Fe-Pt sheets with almost the same composition. Namely, the average Fe composition of each film was between 48 and 52 at. %. The transformation from disordered to ordered phase due to the increase in annealing temperature could be observed.

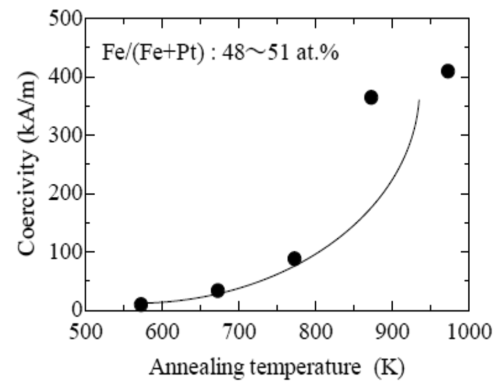

FIG. 7. Coercivity of Fe-Pt sheets with almost the same composition as a function of annealing temperature. Here, the average Fe composition of each film was between 48 and 51 at. %. In the present stage, the values of coercivity exceeded 400 kA/m using an annealing process.

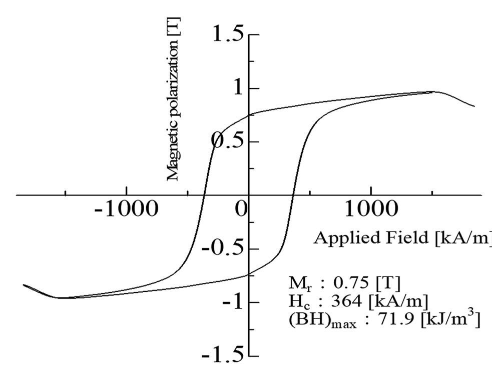

FIG. 8. J-H loop of a $Fe_{49}Pt_{51}$ sheet with the thickness of approximately 35 μm after annealing at 873 K for 30 min.

results about the Fe-Pt films deposited on Ta substrates by using the same laser ablation technique.[16]

C. Investigation on the mechanical characteristic of a Fe-Pt thin sheet using a cantilever test

In the previously reported Fe-Pt thin sheets,[10–12] we had difficulty confirming the mechanical characteristic. Examination of the mechanical strength of the obtained sheet is indispensable because the sheet was prepared using an unfamiliar method of releasable phenomenon. Instead of proposing a detailed device including the Fe-Pt sheet, we paid attention to the previous technique reported by Honda, who has demonstrated a flying machine by assembling a cantilever system using small Nd-Fe-B magnets.[19] The observation on the cantilever test is a possible approach as an evaluation of the mechanical characteristic. As shown in Figures 9 and 10, an approximately 10 μm-thick $Fe_{55}Pt_{45}$ sheet bonded on a polyimide film was vibrated by an external magnetic field (8 kA/m, 160 Hz). The values of coercivity, remanence, and $(BH)_{max}$ of the Fe-Pt sheet were 280 kA/m, 0.8 T, and 65 kJ/m^3, respectively (inset in Figure 10). In addition, the polyimide film measured 0.25 mm wide 4.5 mm long 25 micron thick. The driving force due to the movement of the cantilever could be observed, and it was confirmed that the sample was not damaged after the test. The result suggests that the Fe-Pt thin sheets have the potential to be material for a device from the mechanical strength point of view.

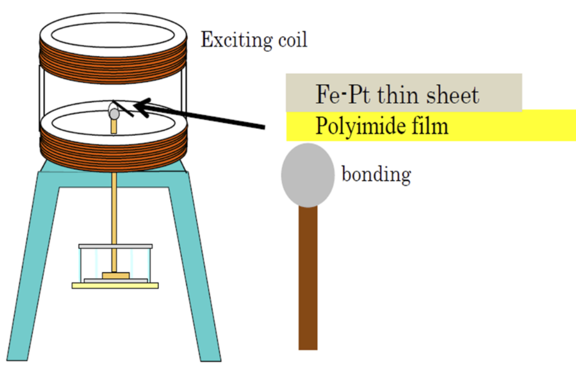

FIG. 9. Schematic diagram of a cantilever containing an annealed Fe-Pt thin sheet with the thickness of approximately 10 μm bonded on a polyimide film which measured 0.25 mm wide by 4.5 mm long by 25 μm thick. The lever was vibrated by the alternative magnetic field of 8 kA/m and 160 Hz.

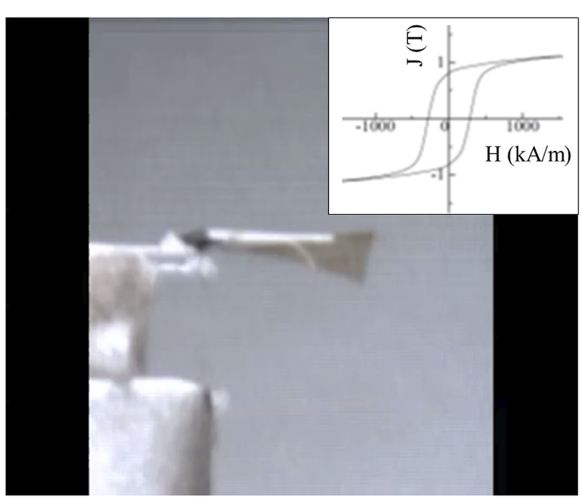

FIG. 10. Photo of a cantilever test cut from an original movie. The detail structure is shown in Fig. 9.

IV. CONCLUSIONS

In the study, it was clarified that the use of a thick film preparation process using a laser ablation technique together with exfoliation behavior is a possible approach to prepare a Fe-Pt thin sheet thicker than 10 μm. Moreover, improvement in magnetic properties of the obtained Fe-Pt sheets is required as additional work.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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