Magnetic Properties of Multilayer $[(\mathrm{FePt})_x/\mathrm{Os}]_n$ Films

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Multilayer $[(\mathrm{FePt})_x/\mathrm{Os}]_n$ films (with x being thickness in nm; Os with a fixed thickness 5 nm; n being the number of layers) have been deposited by ion-beam sputtering. Spacer layer was found to have substantial effects on the multilayers to exhibit different magnetic properties and microstructure. The average grain size of the multilayer films can be well controlled by both annealing temperature and thickness of the FePt layer with the Os space layer. The enhancement of H_c can be understood from the fact that for a FePt film with fixed thickness of Os spacer layer, the increasing number of Os spacer layer will inhibit the grain growth of FePt grains and enriches the grain boundary. The layer by layer structure of $[(\mathrm{FePt})_x/\mathrm{Os}]_n$ films can control the hard magnetic behaviors. Therefore, the multilayer $[(\mathrm{FePt})_x/\mathrm{Os}]_n$ films with Os as the spacer exhibit good hard magnetic properties and are attractive candidates for ultrahigh density magnetic recording media.

***Index Terms*—FePt, grain, multilayer, Os.**

I. Introduction

THE multilayer magnetic films are the essential ingredients of spintronic devices, whose properties, such as structure, technical magnetization, etc., have great influence on the overall performance of the spin devices. Several $\mathrm{L1_0}$ structure alloys such as FePt, CoPt, and FePd films are potential candidates for ultrahigh density magnetic recording media because of the high magnetocrystalline anisotropy [1]–[6]. Among the $\mathrm{L1_0}$ alloys, Fe-Pt system has attracted much attention due to its high K_u value ($\sim 7 \times 10^7$ erg/cm^3), high coercivity, good corrosion resistance, and large energy products $(\mathrm{BH})_{\max}$. Some nano-phased hard magnetic materials have also been extensively studied due to their potentially wide applications. Generally speaking, as-deposited FePt films have a disordered fcc structure with a soft magnetic phase. The formation of the ordered fct $\mathrm{L1_0}$ FePt hard magnetic phase requires preheating substrate or post-annealing the as-deposited films at a high temperature of above 500 °C [7]. However, this high temperature process results in the grain growth, poor surface roughness, inter diffusion between layers, which has the disadvantage of decreasing the recording density of the films and raises the production costs. So, it is a key challenge how to efficiently prevent the diffusion in a magnetic multilayer. From our recent works of the noble metal Osmium (Os) systems [8]–[10], Os has high melting and boiling point, which is predicted to have good effect on preventing inter diffusion between layers induced by sputtering process. We have reported that the Os layer is not a good layer for magnetic exchange coupling [8], but it may be an excellent candidate for space layer for a hard magnetic system [9]. This motivated us to fabricate and to study the FePt multilayers with inserted Os layer, especially on their magnetic properties and microstructures. In this investigation, we report the effect of inserted Os layers for the multilayer $[(\mathrm{FePt})_x/\mathrm{Os}]_n$ films (here, x being thickness in nm; Os with a fixed thickness 5 nm; n being the number of layers; total thickness of FePt layers were fixed roughly at 100 nm) from the variation of magnetic properties and microstructure of the films.

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II. Experimental

The $[(\mathrm{FePt})_x/\mathrm{Os}]_n$ multilayer films on glass substrate were prepared by ion beam sputtering with a high-purity argon source (99.999%). The base pressure of the main chamber was 1.0×10^{-8} Torr and the working pressure was 5×10^{-5} Torr. The thickness of FePt layer x (x = 10–100) in $[(\mathrm{FePt})_x/\mathrm{Os}]_n$ multilayer films was varied from 10, 20, 25, 50, to 100 nm with its associated n value of 10, 5, 4, 2, and 1, respectively. Os was used as a spacer layer with thickness of 5 nm, and the total thickness of the FePt hard layer was roughly kept at 100 nm. The FePt films were deposited by using $\mathrm{Fe_{50}Pt_{50}}$ alloying target and the composition of Pt and Fe checking by inductively coupled plasma spectra is 48.6% and 51.4%, respectively. Samples were deposited at room temperature and subsequently annealed for 1 hour. The annealing temperatures were varied between 300 °C and 800 °C. The Os layer was used to control the texture of hard magnetic layers and the grain size by proper annealing temperature and film thickness. The magnetic properties of the films were characterized at room temperature with a vibrating sample magnetometer (VSM) and a superconducting quantum interference device (SQUID). The structure of films was identified by an X-ray diffractometer (XRD) with $\mathrm{Cu-K_\alpha}$ radiation. The microstructure of films was investigated by high-resolution transmission electron microscopy (HRTEM). Specimens for HRTEM observation were prepared by standard sequential

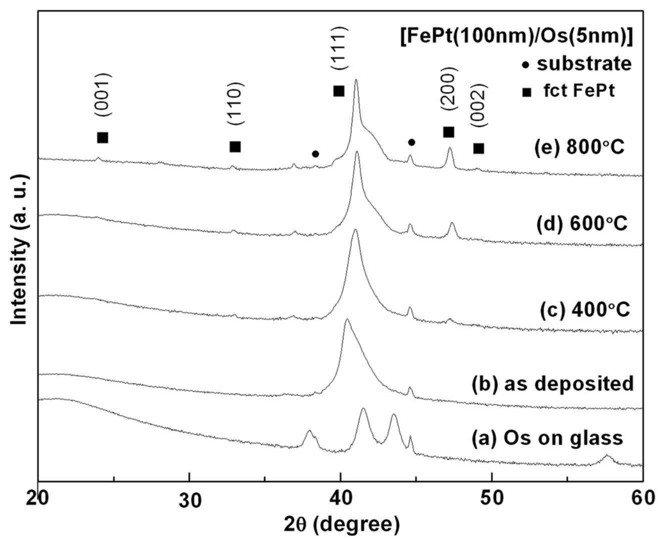


Fig. 1. XRD patterns of (a) a pure Os film with 50 nm thickness on glass substrate, (b) for as deposited sample, and (c) to (e) for the [FePt(100 nm)/Os(5 nm)] multilayer films with annealing temperatures at 400 °C, 600 °C, and 800 °C for one hour, respectively.

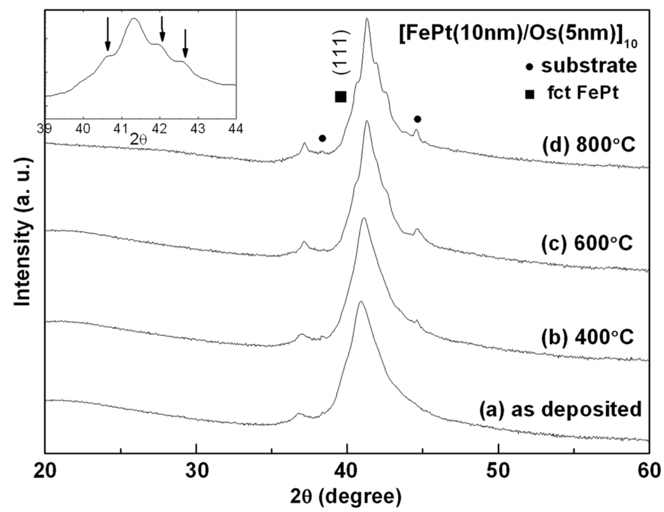


Fig. 2. XRD patterns of the $[\mathrm{FePt}(10\ \mathrm{nm})/\mathrm{Os}(5\ \mathrm{nm})]_n$ multilayer films with (a) for the as deposited sample, and (b) to (d) for samples with annealing temperatures at 400 °C, 600 °C, and 800 °C for one hour, respectively.

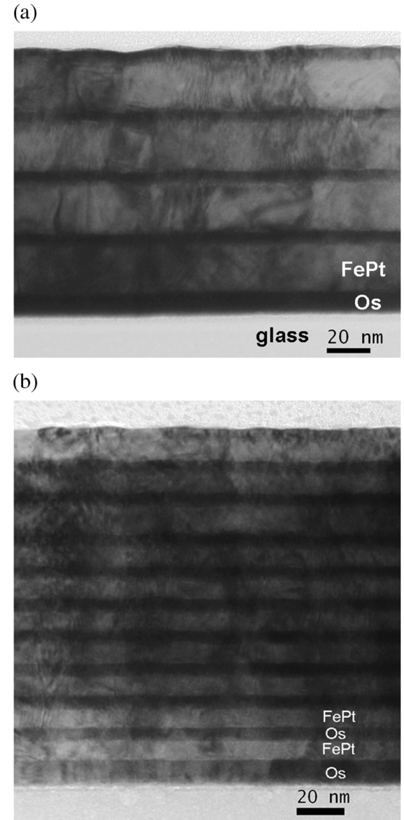
metallographic polishing with emery papers and diamond papers, and then treated with the ion beam etching method to produce a film thickness of less than 100 nm.

III. Results and Discussion

Fig. 1 shows the XRD patterns of (a) a pure Os film with 50 nm thickness on glass substrate, (b) for as deposited sample, and from (c) to (e) for the [FePt(100 nm)/Os(5 nm)] multilayer films with annealing temperatures at 400°C, 600°C, and 800 °C, respectively. It is clear that there is a shoulder at the right side of the fct FePt (111) peak near 41° for curves (c) to (e). By using a Lorentzian fit, a second peak at 42° could be obtained; this means that the Os (002) diffraction peak is embedded in this shoulder.

For the as deposited sample, curve (b) displays a disordered fcc phase with (111) orientation only. After annealing at 400 °C and 600°C, a peak for (200) was observed as shown in curves (c) and (d). In curve (e), additional peaks of (001), (110), and (002) were observed. These peaks were completely the same with a pure FePt film without an Os layer after annealing at 800 °C (data not shown). Kim *et al.* [11] reported that the origin of fct-FePt (001) texture evolution in FePt films was due to the occurrence of anisotropic strain during ordering transformation. Fig. 2 shows the XRD patterns of the $[\mathrm{FePt}(10\,\mathrm{nm})/\mathrm{Os}(5\,\mathrm{nm})]_n$ multilayer films with (a) for the as deposited sample, and (b) to (d) for samples with annealing temperatures at 400 °C, 600 °C, and 800 °C, respectively. By comparing with Fig. 1, the peaks associated with the hard magnetic phases were much weaker and appeared at higher annealing temperatures. It seems that the grain growth of FePt layers was inhibited by the Os space layers after the high temperature annealing. In other words, the grain growth is two-dimensional in FePt multilayer samples and is limited at the thickness direction. From the HRTEM cross-sectional study, the average grain size of the multilayer films can be well controlled by both annealing temperature and thickness of the FePt layers.

(a)



(b)

Fig. 3. Cross-section HRTEM images of the $[\mathrm{FePt}(x\ \mathrm{nm})/\mathrm{Os}(5\ \mathrm{nm})]_n$ multilayers after annealing at 700 °C for one hour, and with (a) $x = 25$ and $n = 4$, and (b) $x = 10$ and $n = 10$.

For identifying the inhibition effect of the grain growth in FePt layers by the Os space layer, as an example, Fig. 3 shows the cross-section HRTEM images of the $[\mathrm{FePt}(x\ \ \mathrm{nm})/\mathrm{Os}(5\ \ \mathrm{nm})]_n$ multilayers after annealing at 700 °C for one hour. Fig. 3(a) is for the multilayer film with

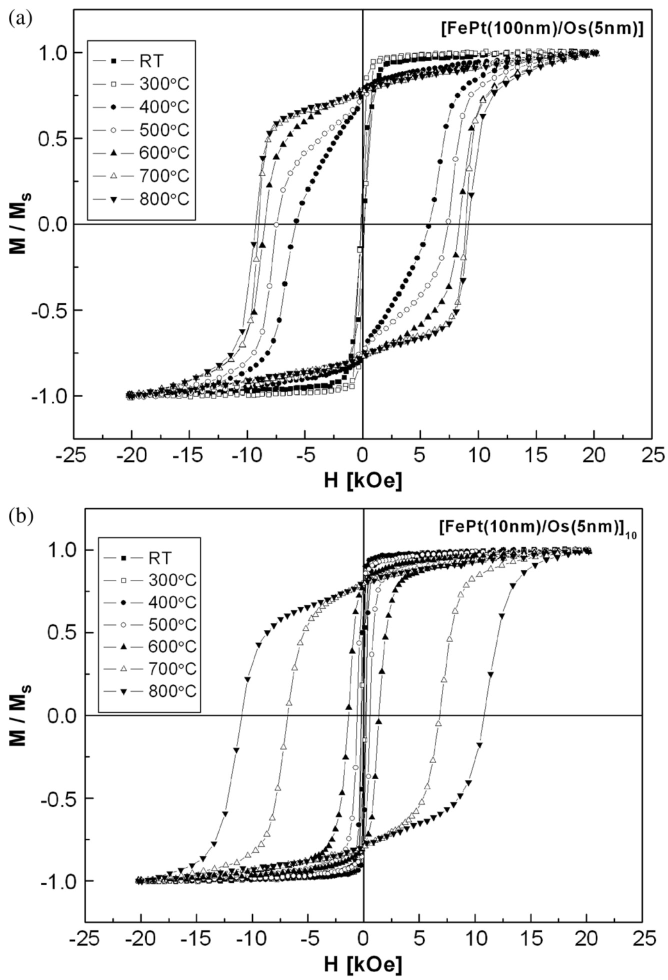


Fig. 4. (a) Hysteresis loops of [FePt(100 nm)/Os(5 nm)] films with annealing temperatures between 300 °C and 800 °C for one hour. (b) The hysteresis loops of $[\mathrm{FePt(10\,nm)/Os(5\,nm)}]_n$ films with annealing temperatures between 300 °C and 800 °C for one hour.

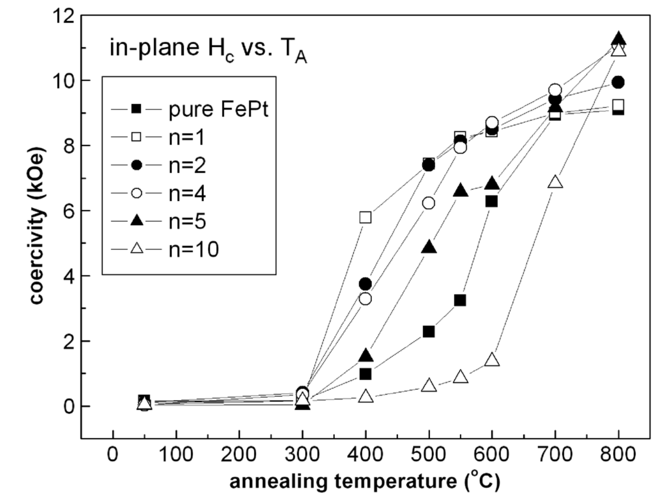


Fig. 5. Coercivity of a pure FePt film and the $[(\mathrm{FePt})_x/\mathrm{Os}_{5\ \mathrm{nm}}]_n$ films with $n = 1$, 2, 4, 5, and 10 as a function of the annealing temperature between 300 °C and 800 °C for one hour.

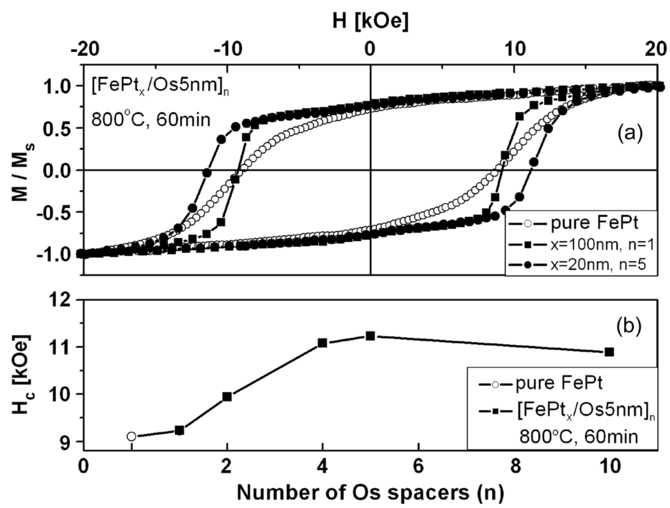


Fig. 6. (a) Hysteresis loops of a pure FePt film and the $[(\mathrm{FePt})_x/\mathrm{Os}_{5\ \mathrm{nm}}]_n$ films with $n = 1$ and 5, and (b) Os layer thickness dependence of the coercive force. All the samples were annealed at 800 °C for 1 hour.

$x = 25$ and $n = 4$, and Fig. 3(b) is for the multilayer film with $x = 10$ and $n = 10$. In general, the various gray regions are originated from the different crystallographic orientations of the isotropically distributed grains. By judging from some selected area of the HRTEM images, the average grain size of $[\mathrm{FePt(x\ nm)/Os(5\ nm)}]_n$ films after annealing at 600 °C was roughly 18 nm for $x = 10$ and $n = 10$, that is quite smaller than the average grain size of a single-layer FePt films after annealing at the same temperature (roughly 160 nm). But the 2-D grain growth in the FePt layers is clearly observed. This indicates that Os space layers can limit the grain growth at the thickness direction.

Fig. 4(a) shows the hysteresis loops of [FePt(100 nm)/Os(5 nm)] films annealed up to 800 °C for 1 hour. The coercivity increases very fast after annealing above 400 °C, but its value is roughly saturated near $\mathrm{H_c} = 9$ kOe for annealing temperature above 700 °C. Fig. 4(b) shows the hysteresis loops of $[\mathrm{FePt(10\ nm)/Os(5\ nm)}]_{10}$ films annealed up to 800 °C for 1 hour. The coercivity increases with increasing annealing temperature very slowly between 300 °C and 600 °C. However, between 600 °C and 800 °C, it increases very fast from roughly 1 kOe at 600 °C and up to roughly 11 kOe at 800 °C. This is explained by the different growth rate and grain size of the hard magnetic FePt $\mathrm{L1_o}$ controlled by both annealing temperature and thickness of the FePt layers. The coercivity as a function of annealing temperature for a pure FePt film and all the $[\mathrm{FePt(x\ nm)/Os(5\ nm)}]_n$ films with $n = 1$, 2, 4, 5, and 10 is depicted in Fig. 5. For samples with $n < 4$ and the pure FePt film, the $\mathrm{H_c}$ behaves roughly saturated after annealing above 700 °C, and the value of $\mathrm{H_c}$ is small than 10 kOe. But the value of $\mathrm{H_c}$ seems still increasing with increasing annealing temperature very fast for temperature above 700 °C. The highest $\mathrm{H_c}$ ($\sim$11.3 kOe) under this study was obtained in $[\mathrm{FePt(20\ nm)/Os(5\ nm)}]_5$ film as shown in Fig. 6(a). For comparison, Fig. 6(a) shows the hysteresis loops of a pure FePt film and $[\mathrm{FePt(xnm)/Os(5\ nm)}]_n$ films with $x = 20$ and 100, after annealing at 800 °C for 1 hour. The enhancement of $\mathrm{H_c}$ can be understood from the fact that for a FePt film with fixed thickness of Os spacer layers, the increasing number of Os spacer layer will inhibit the grain growth of FePt grains and enriches the grain boundary. The layer by layer structure of $[\mathrm{FePt(x\ nm)/Os(5\ nm)}]_n$ films can control the hard magnetic behaviors. As an example, Fig. 6(b) shows the H_c as a function of the number of Os space layer for samples with Os layer and the pure FePt sample without Os layer, after annealing at 800 °C for 1 hour. Therefore, the increase of coercivty of the films with Os spacer layers could be attributed to the pinning of magnetic domains at the grain boundary. In summary, the multilayer $[\mathrm{FePt(x\ nm)/Os(5\ nm)}]_n$ films with Os as the spacer exhibit good hard magnetic properties and are attractive candidates for ultrahigh density magnetic recording media.

Acknowledgment

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