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Microstructure and ordering parameter studies in multilayer $[FePt(x)/Os]_n$ films

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The microstructure, ordering parameter, and magnetic properties of multilayer $[FePt(x)/Os]_n$ films on glass substrate by dc-magnetron sputtering (with x being thickness in nm; Os with a fixed thickness 5 nm; n being the number of layers) have been studied as a function of the annealing temperatures between 300 and 900 °C. The grain size of multilayer films can be controlled by annealing temperature and thickness of the FePt layer with Os space layer. The coercivity as a function of the annealing temperature for samples with $n = 1$ and pure FePt behaves roughly saturated after annealing above 700 °C. However, for samples with $n > 4$ the value of H_c seems still increasing with increasing annealing temperature between 600 and 900 °C, and the ordering parameter decreases with increasing the number of Os layers. Our experimental results are reasonably well to describe the effect of strain-assisted transformation. © *2011 American Institute of Physics*. [doi:10.1063/1.3562524]

The ordered FePt, FePd, and CoPd films are potential candidates for ultrahigh density magnetic recording media because of the high magnetocrystalline anisotropy; among them, the high Ku value ($\sim 7 \times 10^7$ erg/cm^3) of ordered FePt films leads to excellent thermal stability of FePt grains scaled down below 5 nm.[1–4] Generally speaking, as-deposited FePt films have a disordered fcc structure with a soft magnetic phase. The formation of the ordered fct $L1_0$ FePt hard magnetic phase requires preheating substrate or post-annealing the as-deposited film at a high temperature of above 500 °C.[5] 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,[6–10] Os has high melting and boiling point, which is predicted to have good effect on preventing inter diffusion between layers induced by sputtering process. However, we have reported that the Os layer is not a good layer for magnetic exchange coupling,[7] but it may be an excellent candidate for buffer and space layers for a hard magnetic system.[9,10] In this investigation, we report the microstructure, ordering parameter, and magnetic properties in the FePt/Os multilayer films on a glass substrate.

The multilayer $[FePt(x)/Os]_n$ films were deposited on a glass substrate at room temperature by dc-magnetron sputtering system. Here, x varied from 2, 5, 10, 25, to 100 nm with its associated n value of 50, 20, 10, 4, and 1, respectively. All the films were deposited at room temperature and then annealed subsequently. A post annealing procedure with temperature ranged between 300 and 900 °C for one hour was taken such that the FePt layers could be converted into its magnetically hard phases. The magnetic properties were measured at room temperature with a vibrating sample magnetometer with a maximum applied field equal to 2 T. Specimens for cross-section transmission electron microscopy (TEM) observation were prepared by mechanical grinding to a thickness of about 15 μm, followed by appropriate ion milling. The microstructures of the films were observed by the TEM with a nano-beam energy dispersive spectrometer. Selected area electron diffraction patterns were collected by a camera attached to the TEM. The order parameter (S) was determined by comparing the measured diffracted intensity ratio. The crystal structure of the samples is characterized by a Philips PW3040/60 x-ray diffraction (XRD) with Cu-Kα radiation.

Heat treatments of the as-deposited FePt/Os films on glass substrates were studied between 300 and 900 °C. As an example, Fig. 1 shows the XRD patterns of the multilayer $[FePt(x)/Os]_n$ films after annealing at 600 °C for one hour; (a) for sample with $n = 1$, (b) for sample with $n = 4$, (c) for sample with $n = 10$, (d) for sample with $n = 20$, and (e) for sample with $n = 50$, respectively. For the films with $n = 50$, the XRD pattern displays a disordered face-centered-cubic (fcc) phase with (111) orientation roughly near 40.4° as shown in Fig. 1 curve (e). For sample with n decreasing from $n = 20$ to $n = 1$ [curves (d) to (a)], it is clear that the (111) peak of the post-annealing treated FePt film shifted to 41° and the superlattice

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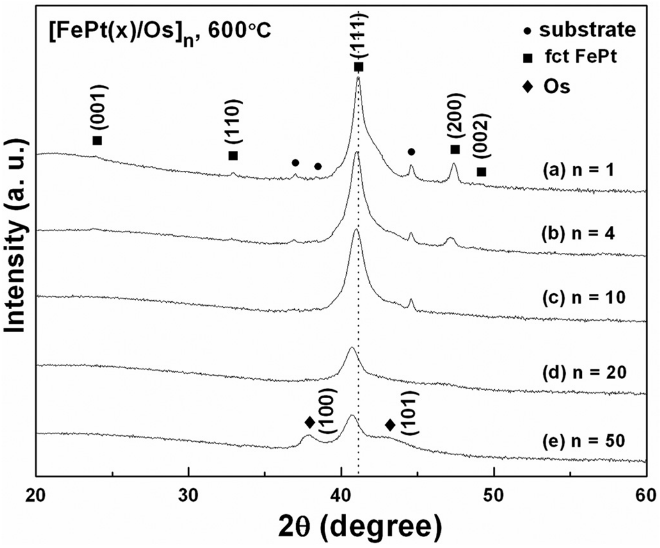

FIG. 1. The XRD patterns of the $[FePt(x)/Os]_n$ films after annealing at 600 °C for one hour; (a) for sample with $n = 1$, (b) for sample with $n = 4$, (c) for sample with $n = 10$, (d) for sample with $n = 20$, and (e) for sample with $n = 50$, respectively.

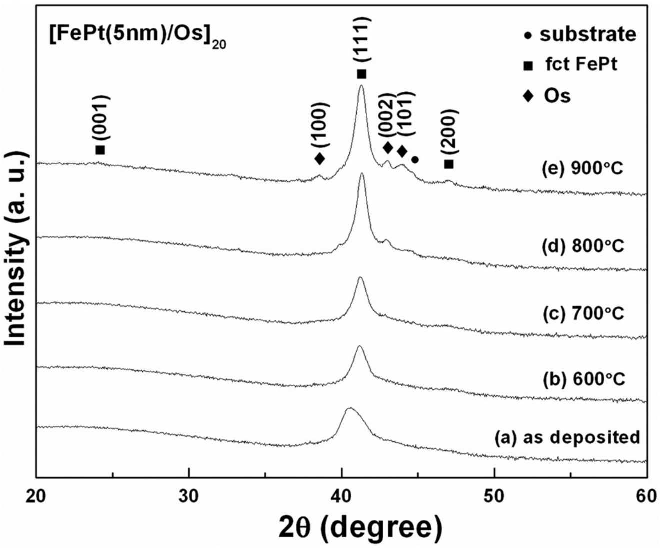

FIG. 2. The XRD patterns of the $[FePt(5nm)/Os]_{20}$ films with (a) for the as deposited sample, and (b) to (e) for samples annealed at 600, 700, 800, and 900 °C, respectively.

(001), (110), (200), and (002) peaks showed up with increasing the thickness of the single FePt layer in the multilayer FePt/Os films; this indicates that the formation of the $L1_0$ ordered phase is much better in $[FePt(x)/Os]_n$ samples with $n < 10$. But, for samples with $n = 20$ and 50, the annealing temperature at 600 °C is not high enough to form the $L1_0$ ordered phase, and the extra Os(100) and (101) peaks in sample with $n = 50$ are identified as shown in curves (e), it is due to the increasing thickness of the Os layers. It is clear that there is a shoulder at the right side of the peak near 41° for all the curves. 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.

Figure 2 shows the XRD patterns of the $[FePt(5nm)/Os]_{20}$ films with (a) for the as deposited sample, and (b) to (e) for samples annealed at 600, 700, 800, and 900 °C, respectively. For the as deposited sample, curve (a) displays a disordered fcc phase with (111) orientation only. After annealing at 900 °C, some additional peaks of (001) and (200) were observed as shown in curve (e). 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.

The coercivity of a pure FePt film and all the $[FePt(x\ nm)/Os]_n$ films with $n = 1$, 4, 10, 20, and 50 as a function of the annealing temperature between 300 and 900 °C for one hour is depicted in Fig. 3. For samples with $n \leqq 4$, the Hc increases very fast than that of the pure FePt film after annealing above 400 °C, This behavior could be explained by the strain-assisted transformation effect. From our TEM studies, the addition of Os layers actually causes an obvious strain. For samples with $n = 1$ and 4 (the thickness of FePt layers is 100 and 25 nm, respectively), due to the large strain effect of lattice-mismatch between the Os and FePt layers, the FePt $L1_0$ structure in the FePt layers can be enhanced by straining along the c axis for the fcc structure. Therefore, it can assist the growth of hard FePt $L1_0$ phase in this two cases, and results with higher Hc. The remained disordered fcc FePt phase is associated with large strain as being analyzed by TEM, which further indicate the strain-assisted transformation effect.

However, for samples with $n = 10$, 20, and 50 (the thickness of FePt layers is 10, 5, and 2 nm, respectively), the strain can be partially released and less degree of average strain was applied to the layers (similar to the prestrained growth technique).[12] Therefore the amount of FePt L10 phase becomes less. The number of Os layers is then the main cause to reduce the ordering parameter and much higher annealing temperature should be applied to form the L10 FePt phase. Therefore, the Hc increases with increasing annealing temperature very slowly between 300 and 600 °C; but, it increases very fast with increasing annealing temperature between 600 and 900 °C. The enhancement of Hc can be understood from the fact that for a FePt film with fixed thickness of Os spacer layer, the

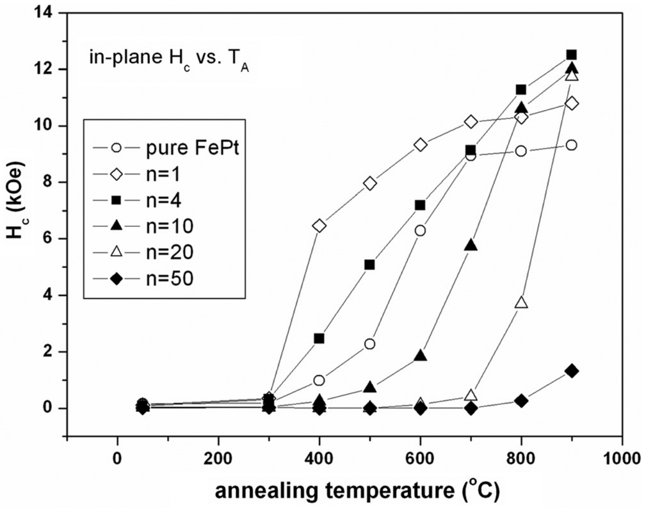

FIG. 3. Coercivity of a pure FePt film and all the $[FePt(x\ nm)/Os]_n$ films with $n = 1$, 4, 10, 20, and 50 as a function of the annealing temperature between 300 and 900 °C for one hour.

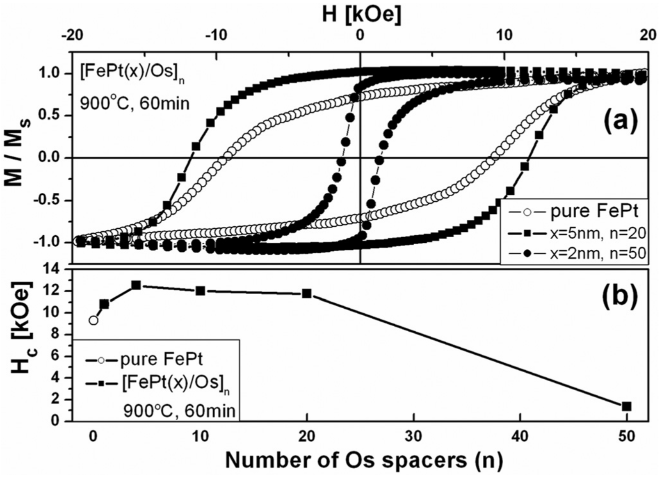

FIG. 4. (a) Hysteresis loops of a pure FePt film and [FePt(x nm)/Os]$_n$ films with x = 5 and 2, and (b) Os layer thickness dependence of H$_c$. All the samples were annealed at 900 °C for 1 h.

increasing number of Os spacer layer will inhibit the grain growth of FePt grains and enriches the grain boundary. As an example, Fig. 4(a) shows the hysteresis loops of a pure FePt film and [FePt(x nm)/Os]$_n$ films with x = 5 and 2, after annealing at 900 °C for 1 h. The value of Hc for the sample with x = 2 is quite small than that of the pure FePt film. This means that a FePt layer with 2 nm thickness is not thick enough for the inter diffusion and the formation of the ordered FCT phase in this film. Figure 4(b) plots the Hc as a function of the number of Os space layer for all the samples after annealing at 900 °C for 1 h. The highest Hc (~12.3 kOe) under this study was obtained in [FePt(25nm)/Os]$_4$ film. However, the annealing temperature of 900 °C is still not high enough for sample of [FePt(2nm)/Os]$_{50}$, because the grain of the hard phase in a 2 nm FePt layer is inhibited by the Os space layers. The ordering parameter studied from the diffracted intensity of a selected area electron diffraction as a function of the thickness of FePt layer is plotted in Fig. 5 for samples annealed at 600 and 900 °C. It shows that annealing temperature at 900 °C is high enough to get above 0.6 degree of order for all the samples except the sample with n = 50 and x = 2. But, for annealing temperature at 600 °C the ordering parameter is below roughly 0.1 for samples with $n > 10$ and $x < 10$.

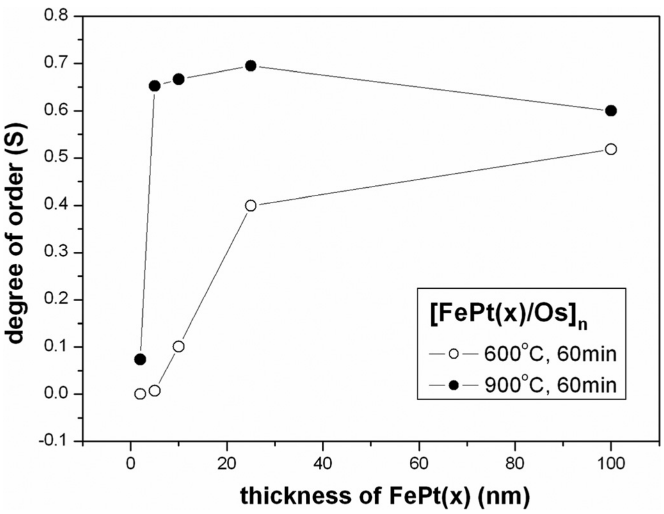

FIG. 5. The ordering parameter for samples annealed at 600 and 900 °C as a function of the thickness of FePt layers.

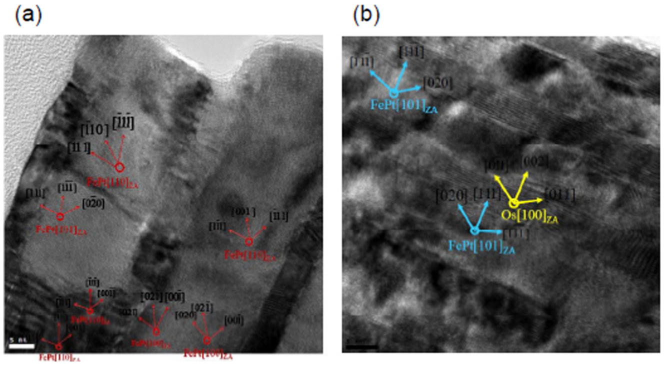

FIG. 6. (Color online) Cross-section HRTEM images of the [FePt(x)/Os]$_n$ films after annealing at 700 °C for one hour, and with (a) n = 4 and x = 25, and (b) n = 10 and x = 10.

Figures 6(a) and 6(b) show the cross-section HRTEM images of the [FePt(x)/Os]$_n$ films with (a) n = 4 and x = 25, and (b) n = 10 and x = 10 after annealing at 700 °C for 1 h. From this TEM analysis, we did see more L1$_0$ superlattice of FePt in sample with n = 4 than that in sample with n = 10. The various gray regions are originated from the different crystallographic orientations of the isotropically distributed grains. The grain size of [FePt(x)/Os]$_n$ films after high temperature annealing is smaller than the grain size of a single-layer FePt films after annealing at the same temperature, and the two-dimensional grain growth in the FePt layers is clearly observed. The inter diffusion behavior between the FePt layers can also be studied by this HRTEM cross-sectional observation. As shown in Fig. 6, well defined interfaces between all the FePt and Os layers were observed. This means the FePt diffusion could be effectively blocked by the inserted Os layers. Again, we have experimentally demonstrated that the diffusion effect can efficiently prevent in the hard magnetic FePt films by the Os space layers and the Os space layers can limit the grain growth at the thickness direction.

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