Nanoscience and
Nanotechnology Letters
Vol. 3, 285–288, 2011

Microstructure and Magnetic Properties of Multilayer $[FePt/Os]_n$ Films

D. P. Chiang[1,2], S. Y. Chen[3], Y. D. Yao[4,*], C. C. Yu[5], Y. Y. Chen[3], and H. M. Lin[2]

[1]Center of General Education, Ming Hsin University of Science and Technology, Hsinchu 304, Taiwan
[2]Department of Materials Engineering, Tatung University, Taipei 104, Taiwan
[3]Institute of Physics, Academia Sinica, Taipei 115, Taiwan
[4]Institute of Applied Science and Engineering, Fu Jen Catholic University, Taipei 242, Taiwan
[5]Department of Applied Physics, University of Kaohsiung, Kaohsiung 811, Taiwan

The microstructure and magnetic properties of multilayer $[FePt(x)/Os]_n$ films on glass and Si substrates by dc-magnetron sputtering technique have been studied as a function of the annealing temperatures between 300 and 800 °C. Here, x varied from 10, 20, 25, 50, to 100 nm with its associated n value of 10, 5, 4, 2, and 1, respectively. On glass, no diffusion evidence was found in all the samples. However, on Si, the insertion of a 10 nm Os layer into the FePt and Si interface results in better thermal stability. The Os underlayer can effectively prevent the diffusion of the intermixing between FePt layer and the Si(100) substrate for temperatures up to 800 °C. The grain size of the multilayer films can be well controlled by both the annealing temperature and the thickness of the FePt layers between the Os layers. The Os layers can effectively prevent the diffusion of the intermixing among the FePt layers and the Si(100) substrate. This means that the diffusion effect can be efficiently prevent in the multilayer $[FePt(x)/Os]_n$ films by the Os layers.

Keywords: Thermal Stability, Diffusion, Annealing, FePt and Os Layer.

1. INTRODUCTION

Several $L1_0$ structure alloys, such as FePt, FePd, and CoPd films, are potential candidates for ultrahigh density magnetic recording media because of the high magnetocrystalline anisotropy;[1–7] among them, the ordered $L1_0$ phase of FePt exhibits one of the highest magnetic anisotropy energy and is a promising candidate for high density magnetic recording media and high energy product materials in microelectromechanical systems. 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.[8] 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,[9–12] 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,[9] but it may be an excellent candidate for buffer and space layers for a hard magnetic system.[10,11] These motivated us to study the thermal stability of magnetic FePt/Os films on Si and glass substrates with/without insertion of Os spacer layers, especially on the magnetic properties and microstructure. In this investigation, we report the Os buffer layer and inserted layer effects for FePt/Os multilayers on Si(100) and glass substrates from the variation of magnetic properties of the films.

2. EXPERIMENTAL DETAILS

The multilayer $[FePt(x)/Os]_n$ films were deposited on a Si(100) and glass substrates with/without a 10-nm-thick Os underlayer at room temperature by dc-magnetron sputtering system. Here, x varied from 10, 20, 25, 50, to 100 nm with its associated n value of 10, 5, 4, 2, and 1, respectively. The Os layers were used to control the texture of hard magnetic layers and the grain size by proper annealing temperature and film thickness. The chamber

*Author to whom correspondence should be addressed.

base pressure was less than 1.0×10^{-8} torr and the working pressure was 5.0×10^{-5} torr with the high purity argon source (99.999%). The FePt films were deposited by using $Fe_{50}Pt_{50}$ alloying target and the composition of Pt and Fe checking by inductively coupled plasma spectra is 48.6 and 51.4%, respectively. All the films were deposited at room temperature and then annealed subsequently. A post annealing procedure with temperature (T_A) ranged between 300 and 800 °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 (VSM) with a maximum applied field equal to 2 Tesla. 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. The crystal structure of the samples is characterized by a Philips PW3040/60 X-ray diffraction (XRD) with Cu-Kα radiation. The following analyses will be focused on the thermal stability of films structures on glasses or on Si substrates with/without an Os underlayer, especially on the magnetic properties and diffusion between the layers.

3. RESULTS AND DISCUSSION

Heat treatments of the as-deposited FePt films with/without an Os underlayer on both Si and glass substrates were studied between 300 and 800 °C. Figure 1 shows the XRD patterns of the multilayer $[FePt(x)/Os(5)]_n$ films on the Si substrate (a) for as-deposited Os(10)/FePt(100)/Os(5) samples, (b) for Os(10)/FePt(100)/Os(5) samples after annealing at $T_A = 700$ °C for 1 hour, (c) for FePt(100)/Os(5) samples annealed at $T_A = 700$ °C for 1 hour, and (d) for $[FePt(25)/Os(5)]_4$ samples annealed at $T_A = 700$ °C for 1 hour. For the as-deposited films with a 10 nm Os underlayer, the XRD pattern displays a disordered face-centered-cubic (fcc) phase with (111) orientation only at 40.4° as shown in Figure 1 curve (a). For sample with Os underlayer (curve b), it is clear that the (111) peak of the post-annealing treated FePt flim shifted to 41° and the superlattice (001), (200), and (002) peaks showed up after the annealing at 700 °C; this indicates the formation of the $L1_0$ ordered phase. However, for samples without an 10 nm Os underlayer after annealing at 700 °C, the FePt(111) peak is vanished as shown in curves (c) and (d), and some extra Pt_2Si_3(004), Pt_2Si_3(101), and Fe_5Si_3(100) peaks are identified according to the JCPDS file 89-2047 and 41-0874 as shown in curves (c) and (d). This means that no hard magnetic phases exist in these samples. Figure 2 shows the XRD patterns of the $[FePt(20)/Os(5)]_5$ films with a 10 nm Os underlayer, and (a) for the as deposited sample, (b) to (d) for samples annealed at $T_A = 400$, 600, and 800 °C, respectively. 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. For the as deposited sample, curve (a) displays a disordered fcc phase with (111) orientation only. After annealing at 400, 600, and 800 °C, a peak for (200) was observed as shown in curves (b) to (d). Some additional peaks of (001), (200), and (002) were observed. Kim et al.[13] reported that the origin of fct-FePt (001) texture evolution in FePt films was due to the occurrence of anisotropic strain during ordering transformation.

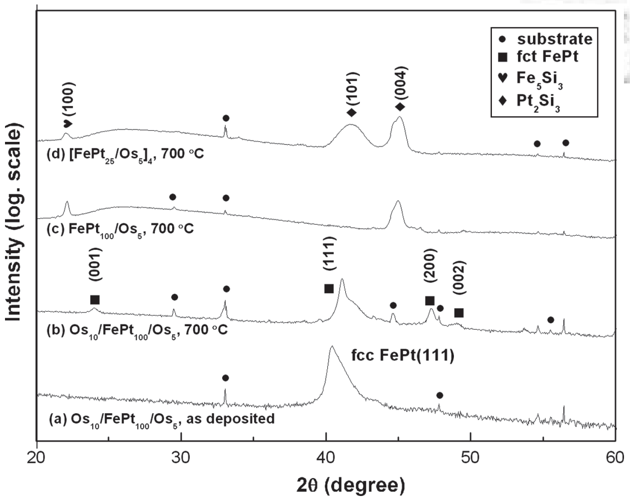

Fig. 1. The XRD patterns of the FePt single-layer films on the Si(100) substrate. (a) as-deposited samples with/without the Os underlayer, (b) sample with a 10 nm Os underlayer after annealing at 700 °C for 1 hour, (c) FePt single-layer films without the Os underlayer after annealing at 700 °C for 1 hour, and (d) $[FePt(25)/Os(5)]_4$ films without a Os underlayer after annealing at 700 °C for 1 hour.

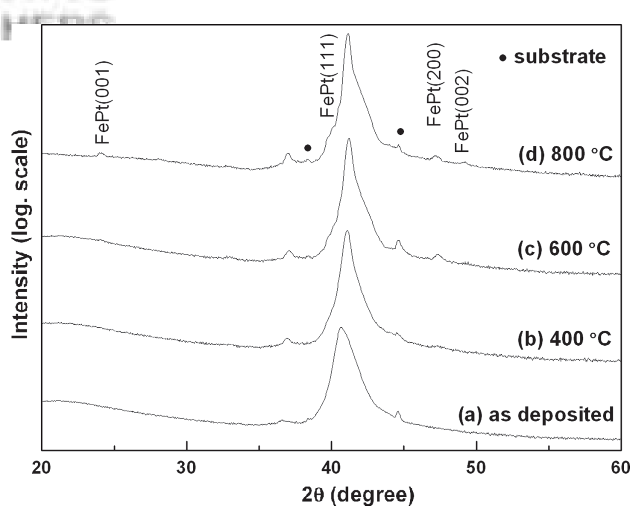

Fig. 2. The XRD patterns of the $[FePt(20)/Os(5)]_5$ films on Si substrate with a 10 nm Os underlayer. (a) is for the as-deposited film. (b), (c), and (d) are for the films annealed at $T_A = 400$ °C, 600 °C, and 800 °C, respectively.

The inter diffusion behavior between the FePt layers and the Si(100) substrate can be studied by a HRTEM cross-sectional observation. As an example, Figure 3 shows the HRTEM cross-sectional image of a multilayer $[FePt(25)/Os(5)]_4$ on the Si(100) substrate (a) without and (b) with a 10 nm Os underlayer after annealing at 700 °C for 1 h, respectively. As shown in Figure 3(a) for the sample without an Os underlayer, the interfaces between the layers were destroyed significantly due to the heavy interdiffusion between the layers. Blurred interfaces between the FePt layers and the Si(100) substrate after high temperature annealing were displayed at the initial stage, but the FePt diffusion could be effectively blocked by the inserted Os layers. Roughly speaking, only the two topmost FePt layers can be recognized in Figure 3(a). However, for the multilayer $[FePt(25)/Os(5)]_4$ sample with a 10 nm Os underlayer, well defined interfaces between all the FePt, Os, and Si(100) layers were observed, as depicted in Figure 3(b). Again, we have experimentally demonstrated that the diffusion effect can efficiently prevent in the hard magnetic FePt films by the Os buffer and space layers. For identifying the inhibition effect of the grain growth in FePt layers by the Os space layer, again as an example, Figure 4 shows the cross-section HRTEM images of a multilayer $[FePt(25)/Os(5)]_4$ sample on glass substrate after annealing at 700 C for one hour. In general, the various gray regions are originated from the different crystallographic orientations of the isotropically distributed grains. From Figures 3 and 4, It is clear that the Os space layers can limit the grain growth at the thickness direction.

To study the effect of Os layers on the magnetic property of the multilayer FePt/Os samples, as an example, Figure 5 shows the hysteresis loops of the multilayer $[FePt(10)/Os(5)]_{10}$ films on Si(100) substrate without an Os buffer layer annealed up to 700 C for 1 hour. For all the samples without an Os underlayer, the interfaces between the layers were destroyed significantly due to the heavy interdiffusion between the layers. Therefore, the coercivity increases quite small even after annealing above 700 C, its value is roughly near 2 kOe. For comparison, Figure 6 plots the hysteresis loops of the multilayer $[FePt(20\ nm)/Os(5\ nm)]_5$ films on glass substrate annealed up to 800 C for 1 hour. The coercivity increases with increasing annealing temperature very slowly below roughly 400 C. However, between 400 C and 600 C, it increases very fast from roughly 2 kOe at 400 C and up to roughly 9 kOe at 600 C. After 800 C annealing, the coercivity can up to roughly 11 kOe. This is explained by the different growth rate and grain size of the hard magnetic FePt controlled by both annealing temperature and thickness of the FePt layers. This enhancement of coercivity is related to the increase of the FePt $L1_0$ phase after annealing. The enhancement of coercivity can be understood from the fact that for a FePt/Os films 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 the films can control the hard magnetic

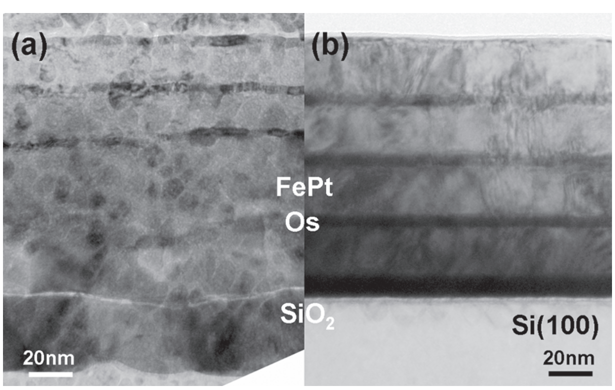

Fig. 3. Cross-section HRTEM images of the $[Os(5)/FePt(25)]_4$ films on the Si(100) substrate (a) without, and (b) with a 10 nm Os underlayer after annealing at 700 °C for one hour, respectively.

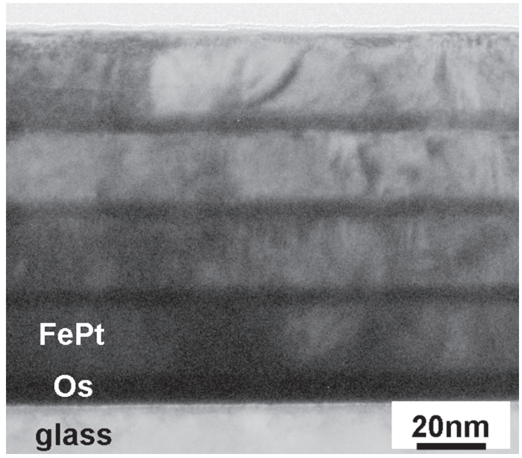

Fig. 4. Cross-section HRTEM images of the $[Os(5)/FePt(25)]_4$ films on the glass substrate after annealing at 700 °C for one hour.

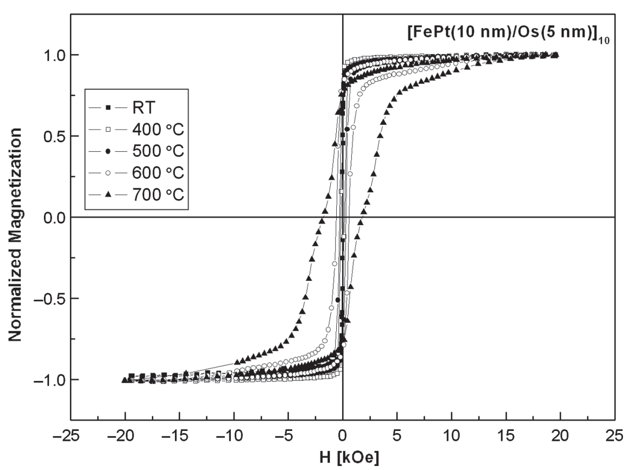

Fig. 5. The coercivity versus the annealing temperature of the $[Os(5)/FePt(10)]_{10}$ films on the Si(100) substrate without an Os underlayer. The external magnetic field is parallel to the film plane.

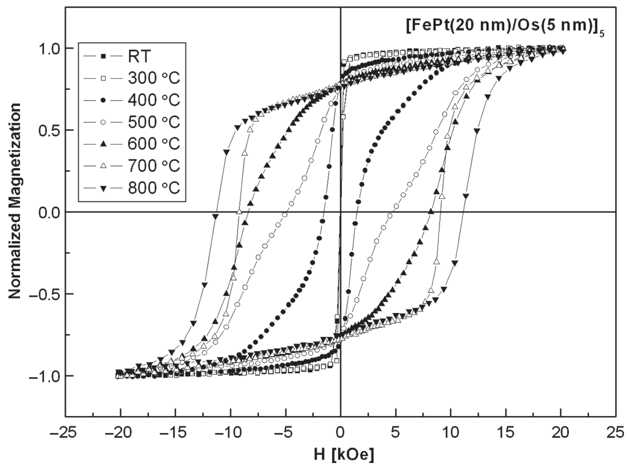

Fig. 6. The coercivity versus the annealing temperature of the [Os(5)/FePt(20)]$_5$ films on the glass substrate. The external magnetic field is parallel to the film plane.

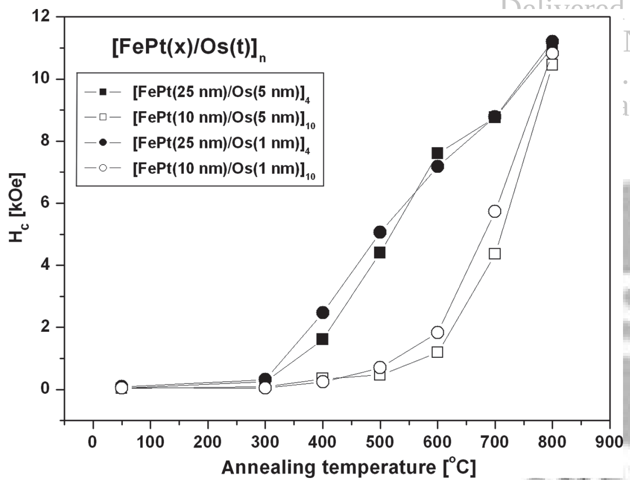

Fig. 7. The coercivity of the [Os(t)/FePt(x)]$_n$ films with $n = 4$ and 10; and $t = 1$ and 5 nm as a function of the annealing temperature between 300 and 800 °C for one hour.

behaviors and the increase of coercivity of the films with Os spacer layers could be attributed to the pinning of magnetic domains at the grain boundary.

The coercivity as a function of annealing temperature for all the [FePt(x)/Os(t)]$_n$ films with $n = 4$ and 10; $t = 1$ and 5 nm is depicted in Figure 7. From this figure, we demonstrate experimentally that the magnetic behavior of the multilayer films with the thickness of the inserted Os layers varied between 1 and 5 nm is almost the same; and it also indicated that the multilayer [FePt(x)/Os(t)]$_n$ films even with a very thin Os spacer layer of 1 nm can efficiently prevent the diffusion of the FePt layers and exhibit good hard magnetic properties. Because Os is the densest known element (22.59 g/cm^3), that the Os layers could prevent the diffusion in the multilayer [FePt(x)/Os(t)]$_n$ films is related to its dense atomic packing in the lattice.

In summary, we have reported experimentally the thermal stability, the interlayer diffusion behavior and the magnetic properties of the multilayer [FePt(x)/Os(t)]$_n$ films on both Si(100) and glass substrates with/without a 10-nm-thick Os underlayer as functions of the annealing temperatures between 300 and 800 °C. The insertion of the Os layers into the FePt films on a Si(100) substrate results in better thermal stability as seen from the X-ray, TEM, and magnetic studies. The Os layers can effectively prevent the diffusion of the intermixing among the FePt layers and the Si(100) substrate for temperatures below 800 °C. This means that the diffusion effect can be efficiently prevent in the hard magnetic FePt films by the Os buffer and inter layers.

Acknowledgment: We are grateful for the financial support by Fu Jen Catholic University, the Sapintia Education Foundation and the National Science Council of Taiwan under Grant No. NSC99-2119-M-030-001.

References and Notes

1. D. Weller, A. Moser, L. Folks, M. E. Best, W. Lee, M. F. Toney, M. Schwickert, J. U. Thiele, and M. F. Doerner, *IEEE Trans. Magn.* 36, 10 (**2000**).
2. J. P. Liu, C. P. Luo, Y. Liu, and D. J. Sellmyer, *Appl. Phys. Lett.* 72, 483 (**1998**).
3. V. Nandwana, G. S. Chaubey, Y. Zhang, and J. P. Liu, *J. Nanosci. Nanotechnol.* 10, 2979 (**2010**).
4. D. Goll and S. Macke, *Appl. Phys. Lett.* 93, 152512 (**2008**).
5. A. Perumal, Y. K. Takahashi, T. O. Seki, and K. Hono, *Appl. Phys. Lett.* 92, 132508 (**2008**).
6. T. Seki, Y. Hasegawa, S. Mitani, S. Takahashi, H. Imamura, S. Maekawa, J. Nitta, and K. Takanashi, *Nature Mater.* 7, 125 (**2008**).
7. X. D. Liu, Z. Xu, R. X. Gao, Z. F. Chen, T. S. Lai, J. Du, and S. M. Zhou, *J. Appl. Phys.* 106, 053907 (**2009**).
8. C. M. Kuo, P. C. Kuo, H. C. Wu, Y. D. Yao, and C. H. Lin, *J. Appl. Phys.* 85, 4886 (**1999**).
9. S. Y. Chen, Y. D. Yao, and J. M. Wu, *J. Magn. Magn. Mater.* 310, 1914 (**2007**).
10. T. Y. Peng, C. K. Lo, S. Y. Chen, and Y. D. Yao, *J. Appl. Phys.* 99, 08C907-1 (**2006**).
11. T. Y. Peng, C. K. Lo, S. Y. Chen, and Y. D. Yao, *IEEE Trans. Magn.* 43, 894 (**2007**).
12. T. Y. Peng, C. K. Lo, Y. D. Yao, and S. Y. Chen, *Appl. Phys. Lett.* 90, 121904 (**2007**).
13. J. S. Kim, Y. M. Koo, and B. J. Lee, *J. Appl. Phys.* 99, 053906 (**2006**).

Received: 20 July 2010. Accepted: 20 November 2010.