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Highly oriented nonepitaxially grown $L1_0$ FePt films

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A method of preparing nonepitaxially grown, highly textured $L1_0$ FePt thin films is described. A nearly perfect (001) texture was obtained by direct deposition of FePt films on Corning 7059 glass substrates and subsequent rapid thermal annealing. The ordering and orientation of the $L1_0$-phase FePt grains were controlled by the initial as-deposited film structure, and also by the annealing process. Magnetic measurements reveal large perpendicular anisotropy for these (001) textured films. The substrates and processes used for nonepitaxial growth of $L1_0$ ordered FePt films are much more compatible with practical applications than those grown epitaxially. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1556257]

I. INTRODUCTION

$L1_0$-phase FePt films have attracted much attention for hard and semihard thin-film applications because of their high magnetocrystalline anisotropy constant that promises thermal stability of material with small grain size. Under normal growth conditions, $L1_0$-phase FePt films often possess (111) preferred or random orientations. In order to control the crystal orientation, epitaxial growth of FePt film was investigated by several deposition techniques including sputtering[1–3] and molecular-beam epitaxy (MBE).[4] The most common methods by which to obtain the *c* axis normal to the film plane [(001) texture] are to use seed or buffer layers between a single-crystal substrate, such as MgO, and the FePt thin films. In most cases, however, it is not convenient or practical to use such methods. In the present study, we report nonepitaxial, highly textured $L1_0$ FePt thin films. (001) texture has been successfully obtained by direct deposition of films on glass substrates and subsequent rapid thermal annealing (RTA). The annealing temperature controls ordering of the FePt grains, and the annealing time controls the orientation of the FePt grains. The orientation mechanism of FePt films deposited on a glass substrate is totally different from that of epitaxial films. We are able to fabricate $L1_0$ ordered FePt films in a very short annealing time and control the orientation by adjusting the initial as-deposited film structures and annealing processes.

II. EXPERIMENT

The samples were magnetron sputtered on Corning 7059 glass substrates with a multilayer structure of Fe/Pt. Targets of Fe and Pt were commercial products with purity higher than 99.9 at. %. The base pressure of the chamber was less than 3×10^{-7} Torr and a working pressure of 4 mTorr Ar was used during sputtering. The substrates were mounted on a water-cooled rotating table. The composition of the film was well controlled by adjusting the thickness ratio of the Fe and Pt layers. The typical total film thickness was 10 nm. The as-deposited films were annealed by RTA from 350 to 650 °C for 2–600 s. The microstructural evolution of the nonepitaxial growth and magnetic properties of these films was investigated by x-ray diffraction (XRD), transmission electron microscopy (TEM), alternating gradient magnetometer (AGFM) and a superconducting quantum interference device (SQUID).

III. RESULTS AND DISCUSSION

The ordering and orientation of the FePt films are dependent on the annealing temperature and time, and (001) texture is obtained for the film that is less thick. Figure 1 shows the ordering process of 10 nm FePt film as the annealing temperature increases, characterized by the increase in intensity of the superlattice peaks. FePt films were prepared by Fe/Pt multilayer deposition with initial layer thickness of 0.47 nm Fe and 0.4 nm Pt. All films were annealed by RTA

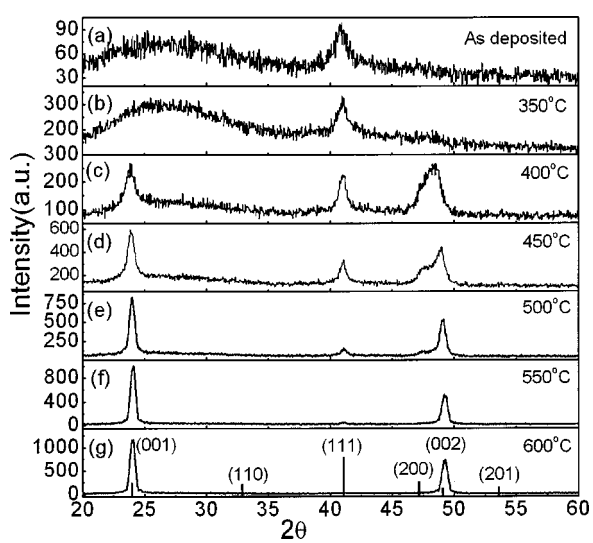

FIG. 1. XRD patterns of [Fe (0.47 nm)/Pt (0.4 nm)]$_{12}$ film annealed at different temperatures T_A for 600 s. T_A=(a) as deposited and (b) 350, (c) 400, (d) 450, (e) 500, (f) 550, and (g) 600 °C.

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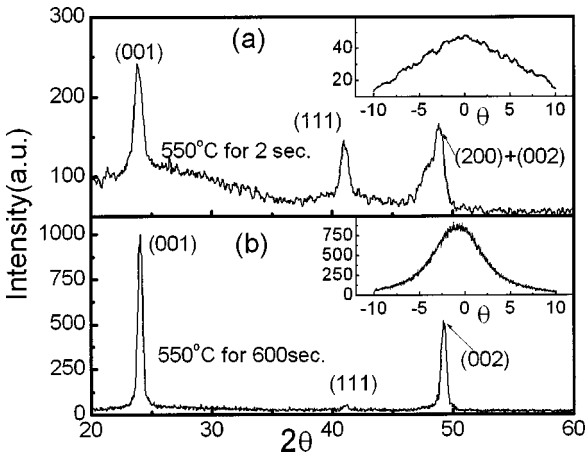

FIG. 2. XRD patterns of [Fe (0.47 nm)/Pt (0.4 nm)]$_{12}$ film annealed at 550 °C for different times: (a) 2 s and (b) 600 s. The insets show the (001) rocking curves.

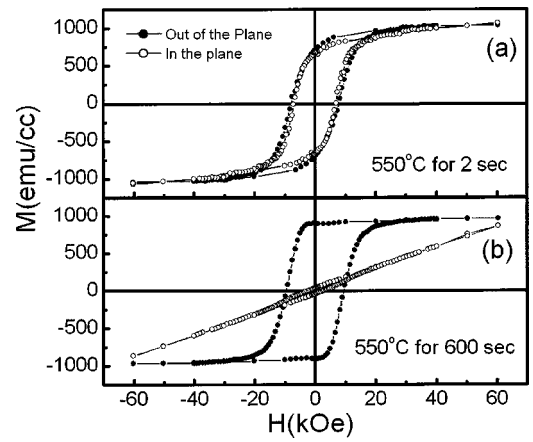

FIG. 3. Hysteresis loops of [Fe (0.47 nm)/Pt (0.4 nm)]$_{12}$ film annealed at 550 °C for (a) 2 s and (b) 600 s. Closed circles represent out of plane loops and open circles in plane loops.

for 300 s at different temperatures. As can be seen in Fig. 1(a), the film is disordered face-centered-cubic (fcc) phase in the as-deposited film. When annealing temperature T_A is lower than 350 °C [Fig. 1(b)], the film is still disordered fcc phase. As T_A increases to 400–450 °C [Figs. 2(c) and 2(d)], (001) and (002) peaks begin to appear, but they are broad and their relative intensity is low, which means that the ordering process has just started. The overlap of the (200) and (002) peaks means that the film is still a mixture of fcc and face-centered-tetragonal (fct) phases. These results indicate that the fcc to fct phase transition is incomplete when the samples are annealed at lower temperature. For T_A above 500 °C, the (001) superlattice peak becomes stronger and the (200) and (002) peaks are separated [Fig. 1(e)], indicating a completely ordered phase. We also see that the intensities of the (001) and (002) peaks increase very quickly, whereas the intensity of the (111) peak decreases as the annealing temperature increases [Figs. 1(f) and 1(g)]. When T_A is higher than 550 °C, the (001) and (002) peaks become dominant and the (111) peak has almost disappeared [Fig. 1(g)], which indicates the high degree of (001) texture. This preferential (001) orientation is quite significant since the [001] direction is the easy axis of FePt, and usually FePt films with an easy axis perpendicular to the film plane can be obtained only by an epitaxial growth technique. Earlier we reported this preferential (001) orientation in thinner CoPt,[5] FePt:B_2O_3,[6] and CoPt:B_2O_3 (Ref. 7) nanocomposite films, and the degree of orientation decreases as the film thickness increases. This preferential (001) orientation may be affected by the properties of the interface between the substrate and the FePt multilayers. The first stage of growth of as-deposited films may determine the initial nanostructure, and then the orientation of postannealed films.[6]

Figure 2 shows XRD patterns of [Fe (0.47 nm)/Pt (0.4 nm)]$_{12}$ films annealed at 550 °C for different amounts of time. The insets in Fig. 2 are the (001) peak rocking curves. Superlattice peaks are clearly seen in Fig. 2(a), indicating the $L1_0$ ordered structure of FePt grains formed after only 2 s of annealing. The broad (001) rocking curve and relatively higher intensity of the (111) peak indicate degraded (001) texture and randomly oriented FePt grains. For the sample annealed for 600 s, the (001) and (002) peaks are dominant and the (111) peak is almost invisible [Fig. 2(b)]. The rocking curve shows the narrow full width at half maximum (FWHM) (=5.4°), which indicates the high degree of (001) texture in the film with a suitable amount of annealing. Our results show that ordering of the FePt grains is very sensitive to annealing temperature T_A, and that orientation of the FePt grains is controlled by the annealing time. Figure 3 shows corresponding hysteresis loops of these two samples measured by the SQUID with a field applied either in the film plane or perpendicular to the film plane. Almost identical hysteresis loops were measured in both directions for the sample annealed for 2 s [Fig. 3(a)], which is consistent with randomly oriented FePt grains. When annealed for 600 s [Fig. 3(b)], the film exhibits a square perpendicular loop with a high remanence ratio, indicating the development of perpendicular anisotropy due to preferential (001) orientation. The magnetic measurements are clearly compatible with the XRD results.

Another experimental finding is that the orientations of $L1_0$-phase FePt films are also dependent on the initial as-deposited film structure. Figure 4 shows XRD patterns of FePt films with different bilayer thicknesses. The films were of the form of $(ND_{Fe}/ND_{Pt})_n$, in which D_{Fe} was a Fe layer of 0.23 nm, and D_{Pt} was Pt layer of 0.2 nm. N was changed from 1 to 8 and n ranged from 24 to 3 in order to keep the nominal film thickness around 10 nm. All films were annealed by RTA at 550 °C for 5 s. It is clearly seen that the texture of the film is affected by the initial multilayer structure. Films are (001) textured when the Fe/Pt bilayer thickness is small. When the bilayer thickness increases, the relative intensity of the (001) peak decreases and the intensity of the (111) peak increases. When N increases to 8, the intensity of (111) increases and becomes dominant. Relatively high (111) intensity and low (001) intensity indicate the development of (111) texture. This result shows that the texture of the film changed from (001) to (111) with the increase in bilayer thickness. This preferential (001) texture appears to be related to the configuration of the nearly monatomic lay-

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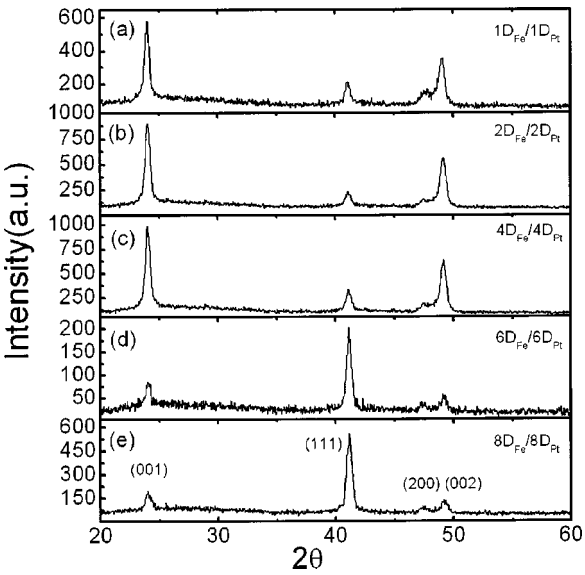

FIG. 4. Ordering and texture development of FePt thin films with different bilayer (D_{Fe}/D_{Pt}) thicknesses. The films were annealed at 550 °C for 5 s. $D_{Fe}=0.23$ nm and $D_{Pt}=0.2$ nm. (a) $1D_{Fe}/1D_{Pt}$, (b) $2D_{Fe}/2D_{Pt}$, (c) $4D_{Fe}/4D_{Pt}$, (d) $6D_{Fe}/6D_{Pt}$, and (e) $8D_{Fe}/8D_{Pt}$.

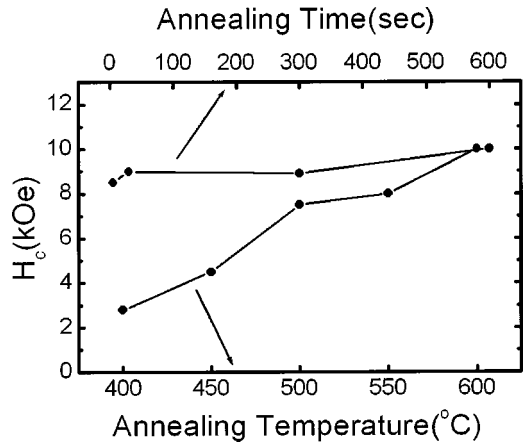

FIG. 5. Coercivity behavior of [Fe (0.47 nm)/Pt (0.4 nm)]$_{12}$ film as a function of the annealing time and the annealing temperature. The annealing temperature was fixed at 550 °C when the annealing time was changed and the annealing time was 600 s when the annealing temperature was changed.

ers. Alternate monatomic-layer deposited FePt films with perpendicular anisotropy deposited at relatively low temperatures have been reported.[8] In our case, one can see from Fig. 4 that samples comprised of thinner bilayers (in these cases the layers are only a few atoms thick) acquire a strong (001) texture upon annealing. In these cases the multilayers already emulate an ordered (001) $L1_0$ phase. During subsequent annealing this requires that the atoms need only move slightly to obtain a stable fct configuration. This preserves the orientation prescribed by the multilayer geometry, namely (001). In the case of the samples with greater bilayer thickness, however, the atoms of each layer must undergo longer random walks to achieve $L1_0$ phase, meaning the pattern of the original layer structuring is lost, resulting in (111) orientation. Although monatomic layering to achieve orientation may be ideal, as suggested by the work of Shima *et al.*,[8] our experiment shows that good orientation can be obtained with a multilayer Fe/Pt configuration deposited by a more conventional, nonepitaxial method that employs magnetron sputtering.

Figure 5 summarizes the systematic trends in perpendicular coercivity of [Fe (0.47 nm)/Pt (0.4 nm)]$_{12}$ films as a function of the annealing temperature and time. For films annealed at 400 °C, the coercivity is around 2–3 kOe. The relatively small coercivity indicates that the film is not fully chemically ordered at this annealing temperature. As the annealing temperature increases, the transformed fraction of the $L1_0$ ordered phase increases and the highest coercivity value obtained (10 kOe) occurs when the film is annealed at 550 °C. The coercivity remains almost unchanged as the annealing time increases. The average grain sizes estimated from TEM grow from 8 to 12 nm when the annealing time is changed from 5 s to 600 s.

IV. CONCLUSION

$L1_0$ ordered FePt films with perpendicular anisotropy was successfully obtained by sputtering Fe/Pt multilayers directly onto a glass substrate and subsequent rapid thermal annealing. This nonepitaxial (001) texture of FePt films can be controlled by the initial multilayer structure and annealing processes. Nonepitaxially grown $L1_0$ FePt films are much more compatible with practical applications than those grown epitaxially.

ACKNOWLEDGMENTS

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