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$L1_0$,(001)-oriented FePt:B_2O_3 composite films for perpendicular recording

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CoPt, FePt, and Other Hard Magnetic Materials Ralph Skomski, Chairman

$L1_0$,(001)-oriented $FePt:B_2O_3$ composite films for perpendicular recording

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A multilayered deposition structure was developed for fabricating $FePt:B_2O_3$ films. We successfully obtained nanostructured $FePt:B_2O_3$ films with FePt grains aligned perpendicular to film plane by postannealing the as-deposited multilayers at 550 °C for 30 min. It was found that development of (001) texture depends strongly on the total film thickness, initial B_2O_3 layer thickness, and Fe concentration. Nearly perfect (001) orientation of FePt-ordered grains can be obtained in the films with small total film thickness, large initial B_2O_3 layer thickness, and slightly higher Fe concentration. Our results show that highly (001) oriented films with ordered fct phase have significant potential as perpendicular media for extremely high-density recording. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1451902]

Much effort has been made to explore recording media for extremely high density recording. Basic requirements for these media are high coercivity, small and uniform grain size, and thermal stability. Most recently, nanocomposite films with $L1_0$ structured CoPt and FePt nanoparticles imbedded in nonmagnetic matrix have received attention.[1–4] These films possess high magnetocrystalline anisotropy ($K_u > 3\times10^7$ erg/cm^3), from which adjustable coercivities (from 4 to 12 kOe) and high thermal stability ($K_uV/k_BT \geqslant 60$) can be obtained, similar to our previously reported CoPt:C,[5,6] $FePt:SiO_2$,[7] and $FePt:B_2O_3$ (Ref. 8) nanocomposite films. Usually, FePt and CoPt thin films deposited by magnetron sputtering tend to grow with random orientations or with (111) texture that place the *c* axes of grains 37° out of the film plane. For magnetic recording media, the magnetic easy axes are preferred to lie either in the film plane or perpendicular to the film plane. Therefore, control of orientation is very important for magnetic recording media. In our previous study,[8] by using large initial B_2O_3 layer thickness, we successfully obtained $FePt:B_2O_3$ films with *c* axes of FePt grains to be aligned with the film normal direction, which normally can only be obtained by the molecular beam epitaxy technique epitaxially on orientated single-crystal substrate. However, a large initial B_2O_3 layer thickness will result in the high nonmagnetic matrix concentration for postannealed films. As we have determined, a high concentration of nonmagnetic matrix materials could deteriorate magnetic and recording properties.[9] On the other hand, a larger B_2O_3 layer thickness causes the film to retain a layered structure after annealing, which will make the $FePt:B_2O_3$ film a ''nonideal'' composite structure. In this study, a multilayered structure was used to deposit $FePt:B_2O_3$ films in order to improve the film properties and better understand the orientation mechanism. A series of x-ray diffraction (XRD) results are presented to show the influence of different initial layer thickness, total film thickness, and Fe concentration on structure, ordering, orientation, and magnetic properties of annealed films. We show that an excellent orientation of *c* axes normal to film plane can be achieved in a low matrix concentration, which has significant potential for perpendicular recording at extremely high density.

$[(Fe/Pt)_x/B_2O_3]_n$ multilayers were deposited on 7059 glass substrates by dc- and rf-magnetron sputtering. The base pressure of the chamber was 2×10^{-7} Torr and working pressure for deposition was 4 mTorr. The Fe atomic composition was varied from 40% to 65% and B_2O_3 volume fraction was 11% to 43%, which were easily controlled by changing the ratio of initial Fe and Pt layer thickness and the ratio of $(Fe/Pt)_x$ and B_2O_3 layer, respectively. Film thickness in this study ranged from 10 to 100 nm. The as-deposited

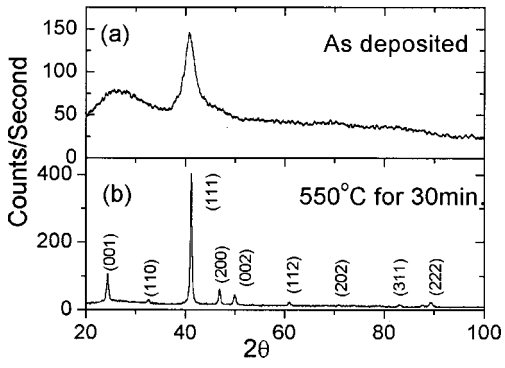

FIG. 1. XRD patterns of $[(Fe(3\ Å)/Pt(4\ Å))_2/B_2O_3(12\ Å)]_{36}$ film: (a) as-deposited and (b) annealed at 550 °C for 30 min.

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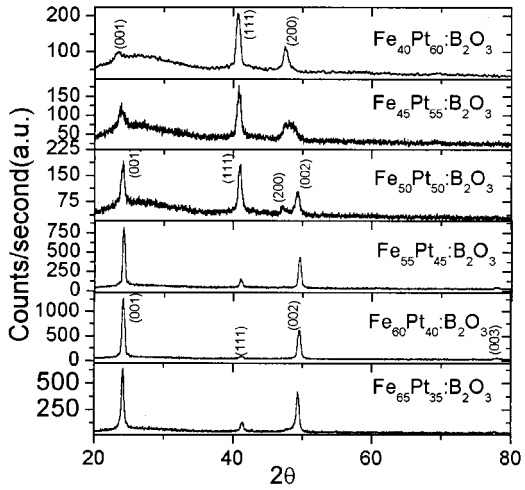

FIG. 2. XRD patterns of the $Fe_xPt_{1-x}:B_2O_3$(11v/o) films annealed at 550 °C for 30 min.

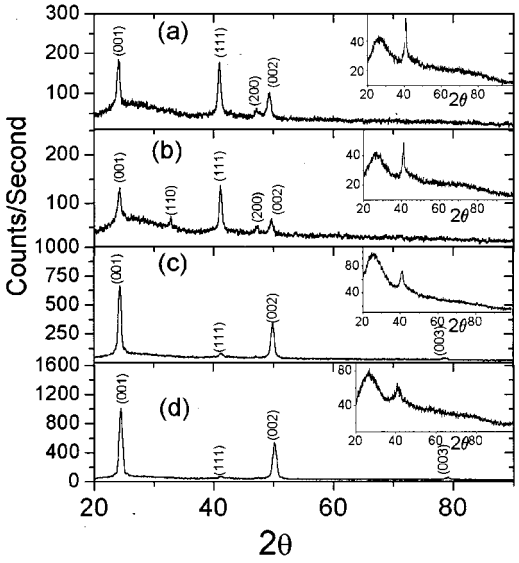

FIG. 3. XRD patterns of $[(Fe(3\ Å)/Pt(4\ Å))_2/B_2O_3(x\ Å)]_n$ films annealed at 550 °C for 30 min. Insets are XRD patterns of the corresponding as-deposited films: (a) $x=2$ Å, $n=10$; (b) $x=4$ Å, $n=9$; (c) $x=8$ Å, $n=7$; (d) $x=12$ Å, $n=6$.

films were annealed in vacuum at 550 °C for 30 min. The structure of the films was investigated by XRD with Cu $K\alpha$ radiation. Magnetic properties were measured by an alternating gradient force magnetometer (AGFM) and a superconducting quantum interference device.

Figure 1 shows the XRD patterns of the as-deposited and postannealed $[(Fe(3\ Å)/Pt(4\ Å)_2/B_2O_3(12\ Å)]_{36}$ film. The as-deposited film consists of disordered fcc FePt [Fig. 1(a)] with a (111) peak predominating. This fcc phase has very low anisotropy energy due to the high symmetry of the crystal structure. Therefore, the film is magnetically soft with a coercivity less than 5 Oe. After annealing at 550 °C for 30 min, the FePt undergoes a phase transition from the disordered fcc to ordered fct structure, which is characterized by (001), (002) superlattice peaks [Fig. 1(b)]. From the intensity ratio of the XRD peaks, the FePt grains are indicated to be randomly oriented. Magnetic measurements show that postannealed film is magnetically hard with coercivities larger than 10 kOe. In the following discussion, we will show that perfect (001) texture can be obtained in the annealed film by adjust Fe concentration, initial B_2O_3 layer thickness and total film thickness.

Figure 2 shows the XRD patterns of the $[Fe(x\ Å)/Pt(4\ Å)_2/B_2O_3(2\ Å)]_{10}$ films with x varying from 2 to 6 Å. The Fe composition of corresponding film changes from 40% to 65 at. % accordingly. All films were annealed at 550 °C for 30 min. One interesting feature is that orientation of FePt grains changes with the Fe concentration. In the Fe-40 at. % film, XRD pattern shows (111), (200) peaks and relatively weak peak of (001). In the Fe-45 at. % film, relative intensity of $(00n)$ begins to increase and intensity of (111) and (200) decreases. The (200) and (002) peaks merge into one because of the close intensity and position of (200) and (002) peaks. This means the film is partially ordered. As Fe concentration further increases, the relative intensity of (111) peak decreases more. In Fe-60 at. % film, the $(00n)$ peak became predominant. The (110) peak is restrained and the (200) disappears. The high intensity ratio of $I_{(001)}/I_{(111)}$ indicates that (001) texture is favored and the c axis prefers to orient perpendicularly to the film plane in slightly Fe-rich films.

Figure 3 shows the XRD patterns of 550 °C-annealed films with initial multilayer structure of $[(Fe(3\ Å)/Pt(4\ Å))_2/B_2O_3(x\ Å)]_n$, where x varies from 2 to 12 Å. Variation of n from 10 to 6 is to keep the total film thickness constant. One can see that the relative intensity of the (111) peak decreases when the initial B_2O_3 layer increases. As the B_2O_3 layer thickness increases to 8 Å and above, the (111) peak nearly disappears. The $(00n)$ peaks become dominant. This result indicates the alignment of the

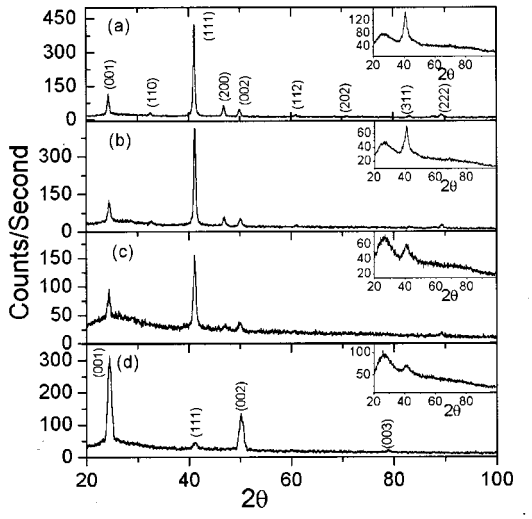

FIG. 4. XRD patterns of the $[(Fe(4\ Å)/Pt(4\ Å))_2/B_2O_3(12\ Å)]_n$ films with different thickness. Films annealed at 550 °C for 30 min. Insets are XRD patterns of the corresponding as-deposited films: (a) 100 nm, (b) 60 nm, (c) 20 nm, and (d) 10 nm.

c axes of FePt grains along the film-normal direction. Comparing these results with our previous report,[8] the big improvement is that no superlattice peaks were observed in postannealed films by low-angle XRD scan. This means that no compositional modulation exists in the postannealed film. The texture of annealed films also depends on the total film thickness. Figure 4 shows XRD patterns of the $[(\mathrm{Fe}(4\,\text{Å})/\mathrm{Pt}(4\,\text{Å}))_2/\mathrm{B_2O_3}(12\,\text{Å})]_n$ films with n varying from 36 to 4, corresponding to film thickness ranging from 100 to 10 nm. In Fig. 4(a), superstructure lines and other peaks can be clearly seen. This means that the film consists of $L1_0$ structure with FePt grains randomly oriented. As the film thickness decreases, the relative intensity of (111) decreases and the intensity of $(00n)$ increases. As the film thickness decreases to 10 nm, $(00n)$ peaks show strong relative intensity and (111) peak shows a weak one [Fig. 4(d)]. The ratio of $I_{(001)}/I_{(111)}$ is a measure of orientation degree while the ratio $I_{(001)}/I_{(002)}$ reveals the ordering degree. It is obvious that (001) texture developed in thinner films, and the orientation degree increases as film thickness decreases. The degree of long-range order does not change in the whole film thickness because the ratio of $I_{(001)}/I_{(002)}$ does not change very much.

The growth mechanism of (001) texture, which depends on initial B_2O_3 layer thickness, total film thickness, and Fe concentration, seems to be related strongly to the first stages of growth. The inset in Fig. 4 shows the corresponding XRD pattern of as-deposited films. The inset in Fig. 4(a) shows that the as-deposited film 100 nm thick has a disordered fcc structure with (111) peak predominated. In this case, FePt grains grow with (111) texture preferred when the film was annealed. As the film thickness decreases, the relative intensity of (111) peak decreases in the as-deposited film. As film thickness decreases to 10 nm, no obvious sharp peaks were observed. The broad peaks mean that the film is disordered FePt nanocrystallites or amorphous [inset of Fig. 4(d)]. Because the intensity of the broad peak between 20° and 30° is higher than that between 35° and 45°, it seems that tendency of (001) oriented nanocrystallites predominated although the film consists of nanocrystallites. In this case, FePt grains grow along the (001) direction when the film is annealed. The insets in Fig. 3 show the same case. The nanocrystallites in the $\mathrm{FePt}/\mathrm{B_2O_3}$ film with larger initial B_2O_3 layer thickness show the tendency of (001) orientation in the as-deposited film. The relatively high intensity, broad peak located between 20° and 30° is from FePt in (001)-oriented nanocrystallites. Therefore we conclude that the orientation of the postannealed films is determined by initial nanocrostructures, which are developed by first stage of growth of as deposited film. A more complete analysis of the orientation mechanism is under way.

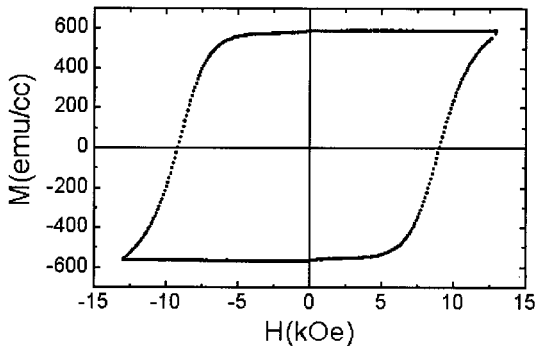

FIG. 5. The hysteresis loops of the $[(\mathrm{Fe}(3\,\text{Å})/\mathrm{Pt}(4\,\text{Å}))_2/\mathrm{B_2O_3}(6\,\text{Å})]_6$ films annealed at 550 °C for 30 min.

Figure 5 shows the hysteresis loop of the $[(\mathrm{Pt}(4\,\text{Å})/\mathrm{Fe}(3\,\text{Å}))_2/\mathrm{B_2O_3}(12\,\text{Å})]_6$ film annealed at 550 °C for 30 min. The film was measured by AGFM with the applied field perpendicular to the film plane. The loop exhibits a perpendicular coercivity that is larger than 9200 Oe and remanence ratio M_r/M_s is nearly 1. The detailed magnetic properties have been discussed in our previous work.[8] These properties come from the (001) orientation of $L1_0$ FePt grains and provide a good choice for perpendicular recording media.

In summary, we have fabricated $\mathrm{FePt}:\mathrm{B_2O_3}$ composite films by a novel deposition structure. It was found that (001) orientation of FePt grains was closely related to the first growth stage of as deposited films. By controlling initial deposition structure of the films, B_2O_3 layer thickness, and Fe concentration, the tendency of (001) oriented FePt nanocrystallites can be formed in the as deposited films. After annealing, composite $\mathrm{FePt}:\mathrm{B_2O_3}$ films with perfect (001) orientation were successfully obtained. These tailorable properties make $\mathrm{FePt}:\mathrm{B_2O_3}$ nanocomposite films very attractive for perpendicular recording at extremely high area densities.

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