Journal of Magnetism and Magnetic Materials 320 (2008) 3071–3074

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Microstructural and magnetic studies of $L1_0$ FePt–SiO_2 nano-composite thin film with columnar structure for perpendicular magnetic recording

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ARTICLE INFO

Available online 8 August 2008

PACS:
75.50.Bb
74.25.Ha
75.50.Ss
74.81.Bd
75.75.+a

Keywords:
FePt
Granular media
Perpendicular magnetic recording
Columnar structure

ABSTRACT

$(Fe_{50}Pt_{50})_{100-x}$-$(SiO_2)_x$ films ($x = 0$–30 vol%) were grown on a textured Pt(0 0 1)/CrRu(0 0 2) bilayer at 420 °C using glass substrates. FePt(0 0 1) preferred orientation was obtained in the films. Interconnected microstructure with an average grain size of about 30 nm is observed in the binary FePt film. As SiO_2 is incorporated, it precipitates as particles are dispersed at FePt grain boundaries. When the content of SiO_2 is increased to 13 vol%, columnar FePt with (0 0 1) texture separated by SiO_2 is attained. The FePt columns have a length/radius ratio of 2:1. Additionally, the mean grain size is reduced to about 13 nm. The development of this well-isolated columnar structure leads to an enhancement in coercivity by about 44% from 210 to 315 kA/m. As the SiO_2 content exceeds 20 vol%, a significant ordering reduction is found accompanied by a transformation of preferred orientation from (0 0 1) to (2 0 0) and the columnar structure disappears, resulting in a drastic degradation in magnetism. The results of our study suggest that isolated columnar, grain refined, (0 0 1)-textured FePt film can be achieved via the fine control of SiO_2 content. This may provide useful information for the design of FePt perpendicular recording media.

1. Introduction

Since 2005, the magnetic recording industry has produced hard disk drivers based on the perpendicular magnetic recording (PMR) technology [1]. Conventional longitudinal magnetic recording is expected to be obsolete by 2011, and completely replaced by PMR [2]. Granular perpendicular CoCrPt-based thin film is presently used in PMR media [3,4], yielding, as demonstrated in 2006, a recording density of 345 Gb/in^2 [5]. In a granular film structure, non-magnetic oxide is segregated at the magnetic grain boundary, to reduce magnetic coupling between the magnetic grains. However, when the recording density reaches approximately 500 Gb/in^2 in the granular CoCrPt-based thin film, superparamagnetism degrades thermal stability [3] because the magnetocrystalline anisotropy constant (K_u) is small. The $L1_0$ FePt thin film with high K_u has been extensively investigated in the last decade in order to overcome this issue and continue to increase the recording density.

Several issues have to be addressed before the magnetic recording industry can fully exploit FePt. First, the temperature of the order-disorder phase transformation should be reduced to less than 300 °C, preventing grain growth. The size of the FePt grains must be controlled within a few nanometers—with a narrow distribution of 15% or less. [4] Secondly, the FePt grains must be isolated to reduce inter-grain interactions, reducing media noise. Third, columnar magnetic grains are required for PMR, because shape anisotropy and sufficient grain volume can increase perpendicular anisotropy, stabilizing the magnetization in the normal direction [6]. Recently, control of the microstructure, especially the grain size and the grain isolation, to achieve an acceptable level of media noise, has attracted considerable attention. Various papers have described the incorporation of oxides (or nitrides) such as B_2O_3, BN, SiO_2, Al_2O_3, Si_3N_4, and MgO [7–12] to isolate FePt grains by co-sputtering or annealing the as-deposited multilayer FePt/oxide thin film [13]. However, only few works have achieved the isolated FePt columnar structure with (0 0 1) texture using C and MgO as isolation materials [14,15].

Since the formation of isolated columnar structure is necessary for PMR, the purpose of this study is to explore this feasibility. SiO_2 was adopted as the isolation material because of the clear phase separation between SiO_2 and FePt derived from the large difference of surface energy of amorphous SiO_2 ($\sim 3\times10^{-5}$ J/cm^2) and the crystalline FePt ($\sim 2.1\times10^{-4}$ J/cm^2) [16]. Our investigations indicate that a distinct phase separation can be realized in the $L1_0$ FePt–SiO_2 co-sputtered films prepared on Pt(0 0 1)/CrRu(0 0 2) bilayer at 420 °C if the SiO_2 content is between 0 and 30 vol%. It is further demonstrated that isolated FePt–SiO_2 columnar structure can be attained with the proper control of SiO_2 content. The magnetic behaviors of the FePt–SiO_2 film are correlated with the microstructure herein.

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doi:10.1016/j.jmmm.2008.08.032

2. Experiment

Composite $(FePt)_{100-x}$-$(SiO_2)_x$ films ($x = 0$–30 vol%.) were prepared on Pt/$Cr_{90}Ru_{10}$ bilayer by magnetron sputtering using glass substrates. The base pressure of the sputtering chamber was under 6.7×10^{-7} Pa. Before sputtering the composite FePt–SiO_2 films, the substrate was heated to 300 °C in order to deposit CrRu(0 0 2) underlayer and Pt(0 0 1) buffer layer. Afterwards, the temperature was increased to 420 °C for deposition of composite FePt–SiO_2 films [17]. The thicknesses of the FePt–SiO_2, Pt, and CrRu layers were fixed at 20, 3, and 70 nm, respectively. Various rf sputtering powers of SiO_2 were used to adjust the volume fraction of SiO_2 in the composite FePt–SiO_2 film. A simple formula, $[R_{SiO_2}/(R_{FePt} + R_{SiO_2})] \times 100$ vol%, was used to calculate the SiO_2 content in the composite FePt–SiO_2 film, where R denotes the sputtering rate. After deposition, the power of the substrate heater was shut off and the FePt–SiO_2/Pt/CrRu trialyer samples were cooled to room temperature in the sputtering chamber before being removed. The microstructures and crystalline phases of the films were identified by transmission electron microscopy (TEM) and X-ray diffraction (XRD) using Cu-K_α radiation, respectively. Magnetic properties were investigated with a vibrating sample magnetometer at room temperature with a maximum applied field of 1000 kA/m. Magnetic domain structure was studied using a magnetic force microscope (MFM). The composition of the FePt magnetic layer and the CrRu underlayer were determined by a energy dispersive spectrometer (EDS). The results indicate that the composition of FePt and CrRu are $Fe_{50}Pt_{50}$ and $Cr_{90}Ru_{10}$ in at%, respectively.

3. Results and discussion

Fig. 1(a) presents the XRD patterns of the $(FePt)_{100-x}$-$(SiO_2)_x$ samples with $x = 0$, 13, 18, 21, and 30. It is noted that the intensities of FePt(0 0 1) and (0 0 2) peaks decrease and (2 0 0) peak increases as x is increased, suggesting a texture change of FePt Ll_0 phase from (0 0 1) to (2 0 0). Additionally, the position of (0 0 1) peak does not shift with the increase in the content of SiO_2, indicating a good phase separation between FePt and SiO_2. Fig. 1(b) plots the dependence of ordering parameter (S) on x. The value of S is determined from $0.85 \times [I_{(001)}/I_{(002)}]^{0.5}$, where $I_{(001)}$ and $I_{(002)}$ denote the intensities of (0 0 1) and (0 0 2) peaks, respectively [17]. The overlapped (2 0 0) and (0 0 2) peaks can be separated using Gaussian fitting procedure with fixed peak positions. As the SiO_2 content is increased from 0 to 19.5, S decreases from 0.81 to 0.67. Further increasing x beyond 20, S drops rapidly below 0.5 and the lowest S is present in the sample with $x = 30$.

Fig. 2(a) shows the perpendicular coercivity ($H_{c\perp}$) as a function of the SiO_2 content. $H_{c\perp}$ is found to increase from 215 to 310 kA/m when x is increased from 0 to 18. With the further increase of x, $H_{c\perp}$ decreases rapidly to smaller than 100 kA/m. The significant drop can be attributed to the decrease of S as well as the change of preferred orientation of the Ll_0 phase. Fig. 2(b) and (c) displays the in-plane and out-of-plane hysteresis loops of the films with $x = 0$ and 30. The film with $x = 0$ exhibits a good perpendicular magnetic anisotropy, while the film with $x = 30$ have large longitudinal anisotropy. This confirms the change of easy-axis alignment as observed by XRD.

Fig. 3(a)–(d) illustrate the TEM images of the samples with $x = 0$, 13, 18, and 30, respectively. Without incorporating SiO_2, FePt grains are connected with each other. The average grain size is found to be around 30 nm with a wide distribution. In the films with small x, SiO_2 precipitates as small particles are dispersed at FePt grain boundaries. When 13 vol% SiO_2 is added to the FePt

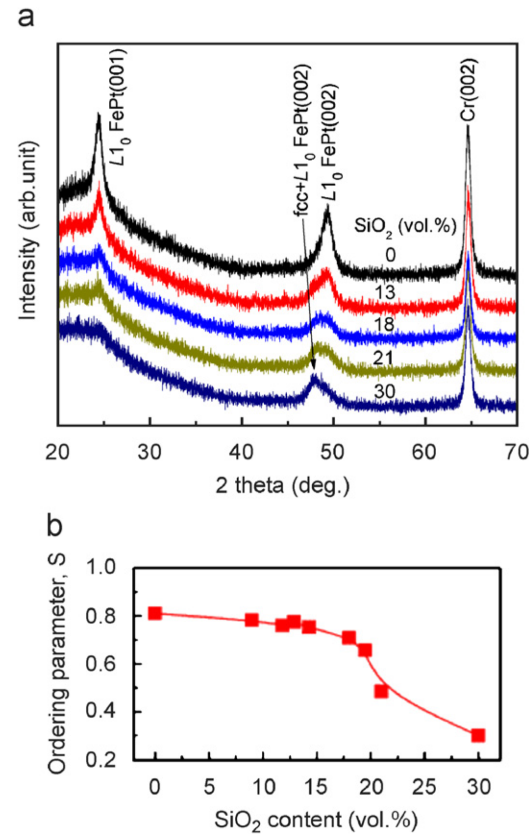

Fig. 1. (a) XRD patterns and (b) ordering parameter (S) of $(FePt)_{100-x}$-$(SiO_2)_x$/Pt/CrRu trilayer films with various SiO_2 volume fraction.

film, the average grain size is markedly reduced to around 13 nm as revealed in Fig. 3(b). The grains are separated by clear boundaries with a narrow size distribution. The thickness of the oxide was approximately 3–4 nm. Nanobeam EDS was used to determine the composition of the grains and boundaries. The composition of the grains is close to $Fe_{50}Pt_{50}$ and the boundary material of the amorphous structure is found to be SiO_2. The film with $x = 18$ has a similar isolated microstructure as with $x = 13$. As x is increased to 30, the average size of the isolated FePt grain is reduced to below 5 nm as indicated in Fig. 3(d). The TEM results further confirmed the distinct phase separation between FePt and SiO_2 when $0<x<30$. From the microstructure evolution by increasing the content of SiO_2, the enhancement of $H_{c\perp}$ in Fig. 2(a) is believed due to the formation of isolation(granular) structure. The non-magnetic SiO_2 hinders the continuous motion of the domain wall during magnetic reversal leading to the increase in $H_{c\perp}$.

In order to further examine the profile structure of the films, cross-sectional TEM analysis was preformed. Fig. 4(a) displays the image of the binary FePt film. Its plane-view image is presented in Fig. 3(a). No clear grain boundary is observed. Instead, in the $(FePt)_{87}$-$(SiO_2)_{13}$ film, clear columnar structure separated by SiO_2 with a length/radius ratio of about 2:1 is observed as revealed in Fig. 4(b). As x exceeds 20, the grain size is reduced to about 10 nm and the columnar structure disappears with the formation of isolated particulate nanocomposite microstructure.

From the TEM results presented above, it can be concluded that the formation of columnar structure is very sensitive to the content of SiO_2. In those samples with low content, SiO_2 precipitates as particles and the microstructure remains continuous. As the SiO_2 content is increased to an optimum level, it surrounds the FePt grains and constrains the lateral growth of Ll_0

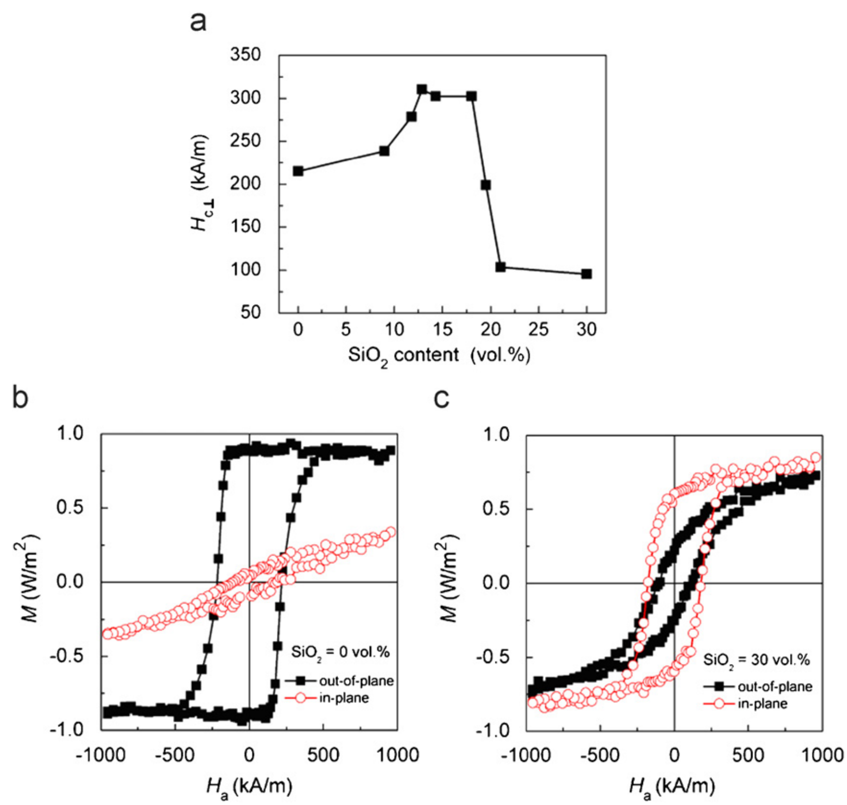

Fig. 2. (a) Dependence of perpendicular coercivity ($H_{c\perp}$) on volume fraction of SiO_2 for the (FePt-SiO_2)/Pt/CrRu trilayer films; (b) and (c) are the in-plane and out-of-plane hysteresis loops of FePt/Pt/CrRu and $(FePt)_{70}$-$(SiO_2)_{30}$/Pt/CrRu trilayer films, respectively.

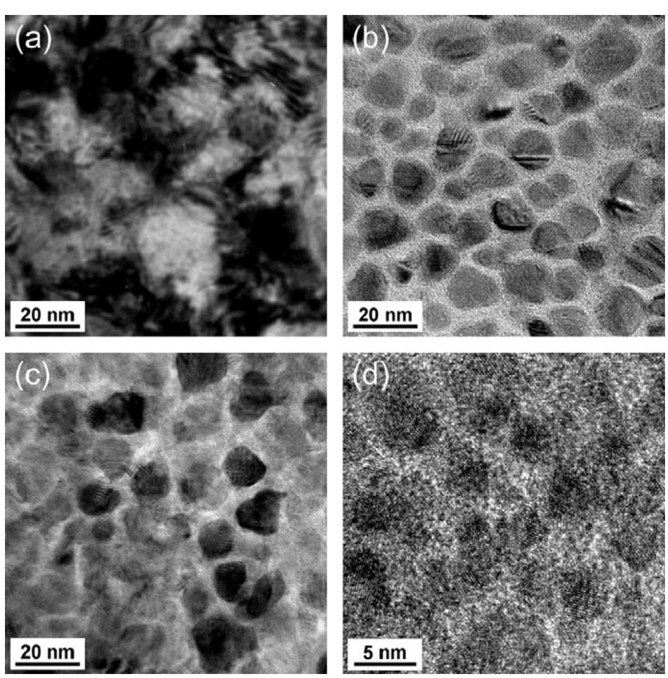

Fig. 3. Plane-view TEM images of $(FePt)_{100-x}$-$(SiO_2)x$/Pt/CrRu trilayer films with (a) $x = 0$, (b) $x = 13$, (c) $x = 18$, and (d) $x = 30$.

phase, resulting in the formation of two-dimensional granular structure with refined size. Furthering increasing the SiO_2 content will promote the formation of three-dimensional granular structure and destroy the columnar characteristics.

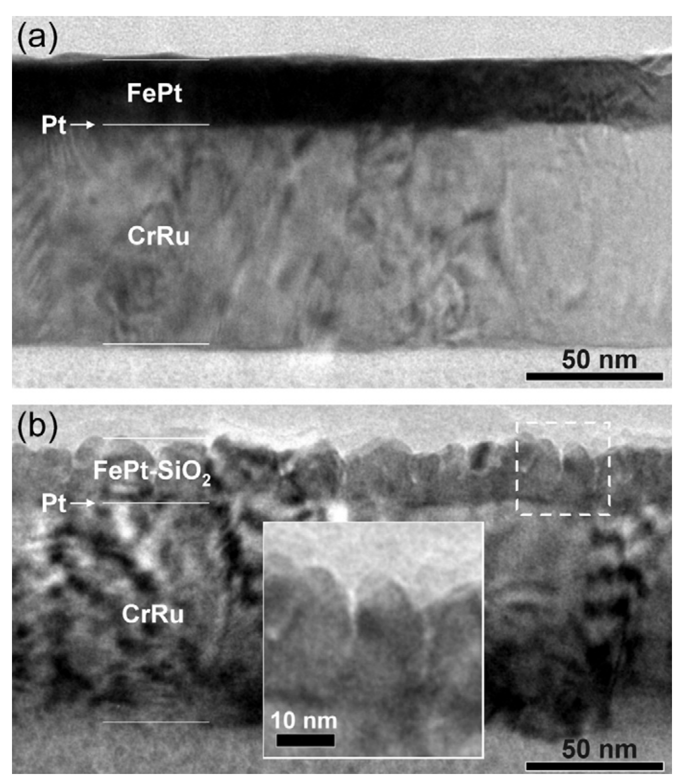

Fig. 4. Cross-sectional TEM images of $(FePt)_{100-x}$-$(SiO_2)_x$/Pt/CrRu trilayer films with (a) $x = 0$ and (b) $x = 13$. The inset is the enlarged image corresponding to the dashed-square region in Fig. 4(b).

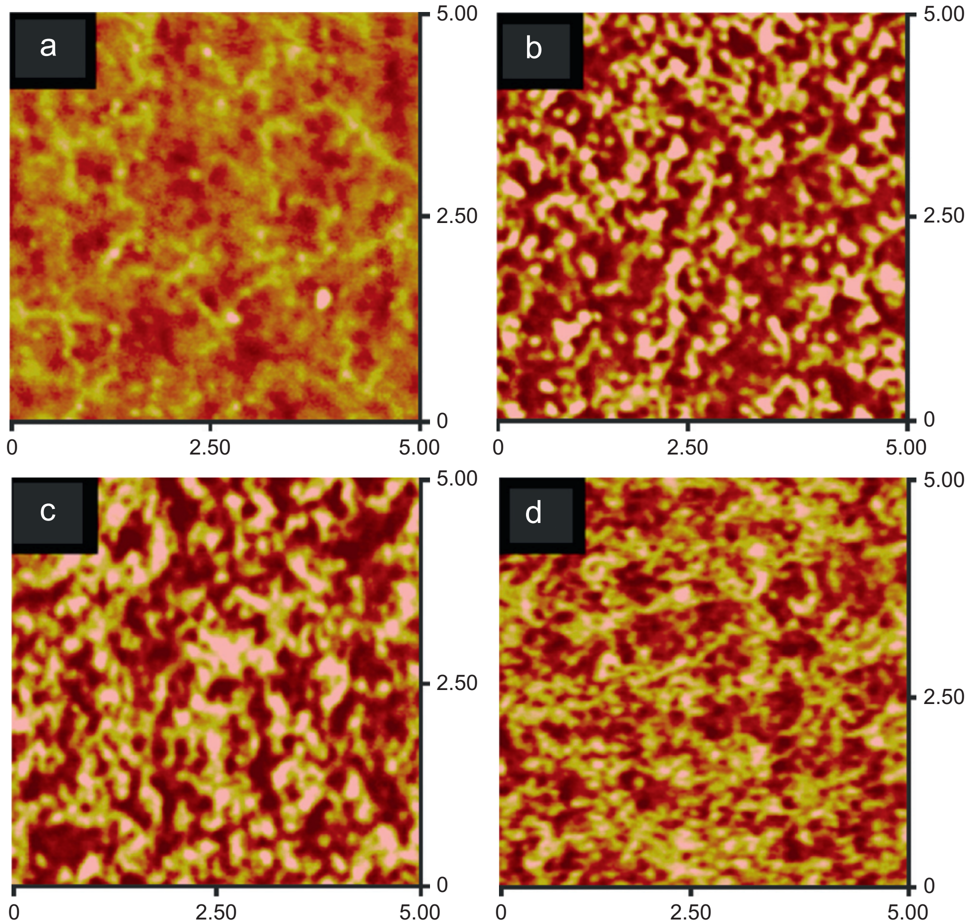

Fig. 5. MFM images of $(FePt)_{100-x}$-$(SiO_2)_x$/Pt/CrRu trilayer films with (a) $x = 0$, (b) $x = 13$, (c) $x = 18$, and (d) $x = 30$.

Fig. 5(a)–(d) shows the MFM images for the $(FePt)_{100-x}$-$(SiO_2)_x$/Pt(0 0 1)/CrRu(0 0 2) films with $x = 0$, 13, 18, and 30, respectively. The samples were dc-demagnetized before imaging. All four images are with the same data scale of 10° and same size of 5 μm × 5 μm. In the binary FePt film, large domain size with weak contrast is found, a typical feature of a magnetic film with interconnected(continuous) microstructure. In the film with $x = 13$, significantly reduced domain size of about 200 nm is observed. Great contrast is present, indicating strong stray field from the isolating magnetic grains. The film with $x = 18$ exhibits similar domain structure and contrast. As x reaches 30, the domain size is further refined and the contrast is weakened again. It can be associated with the reduced grain size and the transition of preferred orientation of Ll_0 phase from out-of-plane to in-plane direction as described earlier.

In summary, the columnar FePt(0 0 1)-textured grains with refined average size surrounded by SiO_2 were fabricated on the textured Pt(0 0 1)/CrRu(0 0 2) bilayer film. Without doping SiO_2, the FePt films that have an interconnected microstructure exhibit typical perpendicular magnetic anisotropy. Adding 13 vol% of SiO_2 will markedly increase $H_{c\perp}$ and lead to the formation of columnar structure with reduced grain size by precipitating SiO_2 surrounding the Ll_0 phase. When the content of SiO_2 exceeds 20 vol%, columnar structure disappears accompanied by a preferred orientation transition from (0 0 1) to (2 0 0). In this study we demonstrate that the isolated FePt(0 0 1) columns can be obtained by the fine control of the content of SiO_2 during co-sputtering, which may be helpful to the design of FePt perpendicular recording media.

Acknowledgement

This work was supported by the Ministry of Economic Affairs of Taiwan.

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