Microstructure and magnetic properties of (001) textured $L1_0$ FePt films on amorphous glass substrate

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ABSTRACT

$L1_0$ FePt thin films with an island-like morphology and magnetic perpendicular anisotropy were grown at low temperature ($300 < T_{dep} < 375$ °C) by magnetron sputtering on Hoya glass substrates using a 30 nm thick Cr (200) underlayer. An MgO buffer layer with a thickness of 2 nm was used to inhibit the diffusion from the Cr underlayer and promote the growth of (001) oriented $L1_0$ FePt films by inducing an in-plane lattice distortion.

By varying the substrate temperature and the Ar sputter pressure ($3.5 < P_{Ar} < 15$ mTorr) during the deposition, the degree of chemical order, the microstructure and the magnetic properties were tuned and the best properties in term of squareness ratio ($M_r/M_s \sim 0.95$) and coercive field ($H_c \sim 14$ kOe) were observed for films deposited at $T_{dep} = 350$ °C and $P_{Ar} = 5$ mTorr, due to the appearance of a tensile strain, which favors the perpendicular anisotropy. The analysis of the angular dependence of remanent magnetization curves on the optimized sample suggests that the magnetization reversal is highly incoherent due to the inter-island interactions. .

Our results provide useful information on the low temperature growth of FePt films with perpendicular anisotropy onto glass substrates, which are relevant for a variety of technological applications, such as magnetic recording and spintronic devices.

Index Terms — $L1_0$ FePt, Glass substrate, magnetization reversal, thin film growth

1. INTRODUCTION

High anisotropy $L1_0$ FePt thin films ($K_u = 4 - 7 \times 10^7$ erg/cc) are of great interest for both fundamental studies and applications such as magnetic recording [1-3] and spintronic devices [4-6]. In magnetic recording, the $L1_0$ FePt alloy is currently considered the best candidate for future granular perpendicular magnetic recording media (> 1 Tbits/in²) due to its high uniaxial magnetocrystalline anisotropy, which can ensure thermal stability of grains with size down to 3 nm [7]. $L1_0$ FePt films with a (001) texture (i.e. c-axis perpendicular to the film plane) are commonly prepared by epitaxial growth on single crystal (100) MgO substrates [8-10]. High temperature processes, such as in-situ substrate heating ($T > 400$ °C) and/or post-deposition thermal treatments ($T > 550$ °C) are required to promote the conversion from the magnetically soft *fcc* phase to the magnetically hard *fct* $L1_0$ phase [11-13]. However, the use of MgO single crystals as substrates is not industrially viable because of their high cost and many efforts have been devoted to the fabrication of ordered $L1_0$ (001) films on more suitable substrates, like glass or Si by using different underlayers such as MgO, CrRu, RuAl and TiN [14-16]. Furthermore high temperature processes are not desirable as they promote the degradation of the (001) texture of single layered FePt films and cause grains coarsening and formation of large particles [17]. To favor the ordering process at low temperatures (< 400 °C) different approaches have been investigated, including i) deposition of a pre-ordered film consisting of a stack of very thin and alternate Fe and Pt layers [18-20], ii) doping the FePt films with a third element such as Au, Ag, and Cu [21-24] or even Ti, Zr and Hf as reported in [25], iii) applying a stress/strain by using either a high gas-pressure sputtering process (resulting in a tensile stress along the *c*-axis) [26] or a suitable underlayer such as CrX (X = Ru, Mo, W, Ti), which induces an in-plane tensile strain by lattice mismatch [27, 28], or Mo, for which an incoherent FePt/Mo interface was found to induce lattice misfit dislocations that favors the *fcc-fct* ordering transformation [29].

In this work we present a low temperature method for inducing the growth of $L1_0$ FePt films with an island-like morphology with a strong (001) texture by using a very thin MgO (100) seed layer grown on a (200) Cr underlayer deposited on a Hoya glass substrate. The MgO seed layer was introduced to prevent the chemical diffusion and favor the transition to the $L1_0$ phase by producing a tensile strain in the first layers, while the Cr underlayer was added to favor the (001) texture of the MgO seed layer, when depositing on an amorphous substrate. According to the literature for similar systems, a Cr thickness of 30 nm ensures a good crystallographic texture on glass [28] and a 2 nm MgO thickness is expected to be sufficient to achieve a good texture while avoiding the Cr diffusion [28, 30].

Moreover, we have studied the effect of the variation of Ar deposition pressure and deposition temperature on film morphology, grain size and magnetic properties and demonstrated that it is possible, by varying such process parameters, to obtain highly textured and high magnetic anisotropy FePt films on glass, with optimized structural and microstructural properties.

2. EXPERIMENTAL

Samples with a FePt/MgO/Cr/Glass stacking sequence were prepared by using a ultra high vacuum AJA Int.-magnetron sputtering system (*ATC 2200-V*) with a base pressure of 1×10^{-9} Torr, which rises up to 2×10^{-8} Torr during substrate heating. The Ar flow was 62 sccm and the Ar deposition pressure (P_{Ar}) varied from 1.5 to 10 mTorr. A Cr underlayer with a nominal thickness of 30 nm was deposited by DC magnetron sputtering at a deposition temperature (T_{dep}) ranging between 100 and 400 $^{\circ}\text{C}$, by applying a target power density of 9.9 W/cm^2 , with $P_{Ar} = 1.5$ mTorr and a deposition rate of 0.083 nm/s. After the deposition of the Cr underlayer, the substrate was cooled down to room temperature and a thin MgO seed layer with a nominal thickness of 2 nm was deposited using an RF magnetron sputtering and an

MgO target. The target power density, the Ar pressure and the deposition rate were set to 9.9 W/cm², 2 mTorr and 0.007 nm/s, respectively. Finally, the FePt magnetic layer with a nominal thickness of 10 nm was deposited with a DC magnetron sputtering using a target power density of 0.6 W/cm², an Ar pressure ranging from 3.5 to 15 mTorr and a substrate temperature varying from 300 to 375 °C. At $P_{Ar} = 5$ mTorr the deposition rate was 0.008 nm/s. The nominal atomic composition of the target was Fe₅₅Pt₄₅, since for that stoichiometry, when the thickness of FePt layer is close to the grain diameter, the strain relaxation along the *c*-axis leads to the (001) texture [31]. Fe₅₅Pt₄₅ films are transformed from *fcc* to *fcc* before grain growth and the *L1*₀ superstructure can affect the subsequent grain growth [31].

The structural analysis was performed by X-ray diffraction using a Siemens D500 diffractometer with Cu-K α radiation. Microstructural investigation was carried out combining field emission scanning electron microscopy (FE-SEM, JEOL 7401F) and transmission electron microscopy (TEM, JEOL 3010 TEM). Magnetic characterization was performed at room temperature by combining an MPMS SQUID magnetometer (Quantum Design, $H_{max} = 50$ kOe) and a vector vibrating sample magnetometer (vVSM, ADE-Technologies Model 10, $H_{max} = 20$ kOe).

3. RESULTS AND DISCUSSION

Structure and Microstructure

Figure 1 shows the XRD θ - 2θ scans of Cr films for different substrate temperatures. On increasing T_{dep} from 100 to 200 °C the intensity of Cr (200) peak first increases, reaching its maximum at 150°C, then decreases and disappears at temperature above 300 °C. At the same time, the intensity of Cr (110) peak, which is absent at low temperature and up to 200°C, weakly increases with the temperature up to 400 °C. It should be noted that at $T_{dep} = 100$ °C, an additional peak is present at $2\theta = 37.83$, which was associated to the (210) reflection of a CrO₃ phase. In figure 2 the rocking curves of the (200) peak are reported for the three samples

with substrate temperature 100, 150, 200 °C. For Cr sample with $T_{dep} = 150^{\circ}\text{C}$ a full width at half maximum (FWHM) of 3° was calculated, which indicates a good texture quality. The lattice constant a , of the Cr underlayer was estimated as 0.288 nm in agreement with the value of the bulk material. It is worth noting that using different values of sputtering parameters, e.g. power density and Ar pressure, larger values of FWHM were observed (data are not reported here). On top of the optimized Cr (200) underlayer ($T_{dep} = 150^{\circ}\text{C}$) an MgO seed layer with a nominal thickness of 2 nm was deposited at room temperature, followed by the deposition of FePt films with a nominal thickness of 10 nm.

The XRD spectra of three samples with 1, 2 and 3 nm thickness of intermediate MgO layer are shown in Fig. 3. All FePt layers showed (001) preferred orientation, and 2 nm MgO thickness is sufficient to maintain the $L1_0$ superstructure.

Figure 4 shows the evolution of XRD θ - 2θ scans for a series of FePt films deposited at different deposition pressures (3.5 - 15 mTorr) and at a constant substrate temperature (350 °C). The (001) superstructure peak is present in all the samples together with a weak (111) reflection. The lowest intensity of the (001) peak was observed for the sample deposited at 15 mTorr. Indeed, even at such a low temperature, the *fcc* phase undergoes progressive modifications of the local environment before reaching the complete transformation to the ordered *fct* phase. Furthermore, the transition to the *fct* phase is highly dependent on microstrains and the degree of short-range order in the film.

The long range order parameter S , which is a measure of the degree of chemical ordering of the FePt phase, was obtained by the square root of the ratio of the integrated intensities of the superstructure peak (001) and the fundamental (002) peak, $[I_{(001)}/I_{(002)}]^{1/2}$, as Cebollada et. al, [31] and Warren [36] have suggested. The highest value of the order parameter ($S = 0.72$) was observed for the sample deposited at 5 mTorr.

A second series of FePt films was prepared at different substrate temperatures (300 - 375 °C) and the deposition pressure P_{Ar} during sputtering was maintained at 5 mTorr (Fig. 5), which corresponds to the lowest intensity of the (111) peak in the previous deposition run (see Fig. 4). In all the samples, superstructure diffraction peaks were observed. At $T_{dep} = 350$ °C the intensity of the (111) reflection reaches the minimum value, indicating that adjusting the Ar gas pressure and the deposition temperature allows to effectively enhance the $L1_0$ chemical ordering even on a glass substrate. For the sample deposited [under optimal](#) conditions ($P_{Ar} = 5$ mTorr, $T_{dep} = 350$ °C) we can consider the growth of FePt(001) on the system {Hoya glass/Cr(200)/MgO(200)} as highly textured, [with the](#) epitaxial relationship FePt(001)[100] $\parallel$ MgO(100)[001] $\parallel$ Cr(100)[110]. The lattice constant c calculated from FePt (001) peak was 0.373 nm.

[As the *fcc-fct* transformation involves a distortion of the *fcc* unit cell, significant stress is created in the material: if the film is subjected to compression \(or tension\) the *c*-axis will preferentially lie in \(or perpendicular to\) the film plane. To investigate the role of stress during the *fcc-fct* ordering, the residual strain \(\$\epsilon\$ \) was determined at variable \$T_{dep}\$ and \$P_{Ar}\$, being the reason for taking \$\epsilon\$ instead of the stress the lack of published data of Poisson's ratio and Young's modulus for FePt films in the intermediate stage of ordering. In our case, the XRD equipment did not allow us to apply the so called \$\sin^2\Psi\$ method \[32\], where \$\Psi\$ is the tilt angle with respect to the normal to the film plane. Therefore, the residual strain was determined from the \$d\(002\)\$ spacing of the \(002\) superstructure peak in XRD spectra, corrected for the reversible thermal strain \[26\], of the individual FePt, MgO \[33\] and Cr layers \[34\] as well as of the glass substrate \[35\]. The residual strain \$\epsilon\$ is shown in Fig. 6 as a function of \$P_{Ar}\$ \(at \$T_{dep} = 350\$ °C\) or \$T_{dep}\$ \(at \$P_{Ar} = 5\$ mTorr\). A tensile strain was observed in the low \$P_{Ar}\$ \(low \$T_{dep}\$ \) regions, hence the formation of the *fct* phase with the *c*-axis perpendicular to the film plane is](#)

avored and the consequent development of a perpendicular magnetic anisotropy was confirmed by the magnetic measurements (see the discussion of magnetic properties).

The FE-SEM images for FePt films deposited at different deposition pressure ($T_{dep} = 350$ °C) and different deposition temperature ($P_{Ar} = 5$ mTorr) are reported in Fig. 7 and 8, respectively. In both case, the images evidences an island-like growth. With increasing Ar pressure, the film morphology changes from a maze-like structure with many interconnected grains at low P_{Ar} values, to a more uniform granular morphology with better isolated FePt grains, in accordance with *Thornton growth* model [37]. Similar effect of size reduction with increasing deposition pressure was also reported in FePt:X films [38] where the growth of columnar grains with enhanced granular microstructure and better inter-grain separation was observed. A phenomenological structural zone model of composite polycrystalline films (based on the supplement to the Thornton's structural zone model) was invoked to explain the microstructural variation for composite thin films fabricated under high temperature deposition conditions. With increasing the deposition temperature a grain growth is also observed as usually expected in a Volmer-Weber island growth mechanism in presence of a large lattice misfit.

The TEM cross sectional image of the sample with optimized FePt deposition conditions e.g. $T_{dep} = 350$ °C, $P_{Ar} = 5$ mTorr (Fig. 9a) shows a clear three-layer structure and confirms the grain isolation of the FePt top layer. HR-TEM is also presented in Fig. 9b. Even if the MgO layer was as thin as 2 nm, a continuous oxide layer was formed. The atomic planes from the $L1_0$ superstructure are well visible in the FePt grains. Fig. 9c, shows the electron diffraction pattern (EDP), which presents a (001) ring indicating the c -axis texture of the $L1_0$ FePt in agreement with the X-Ray results from Fig. 5c.

Magnetic Properties

The values of out-of-plane ($H_{c\perp}$) and in-plane ($H_{c\parallel}$) coercive fields of FePt films deposited at different Argon pressure (constant $T_{dep} = 350$ °C) and deposition temperature (constant $P_{Ar} = 5$ mTorr) are plotted in Fig. 10 (a) and (b), respectively, together with the values of the order parameter S . All the films showed a preferential perpendicular magnetic anisotropy. The best properties in terms of perpendicular squareness ratio ($M_r/M_s \sim 0.95$) and coercive field ($H_{c\perp} \sim 14$ kOe) were found for films deposited at $T_{dep} = 350$ °C and $P_{Ar} = 5$ mTorr, where the predominant (001) texture was observed. The effective uniaxial perpendicular magnetic anisotropy of the optimized sample, determined from the difference in the area enclosed between the in plane and perpendicular magnetization curves, is $(1.0 \pm 0.5) 10^7$ erg/cc, where the uncertainty is mainly due to the fact that the full saturation along the hard axis direction (i.e. in plane magnetization loop) was not reached at the maximum available applied field.

The zero field kink observed in the easy axis loop (Fig.10c, full symbols) indicates that a fraction of grains in the film does not have a uniaxial anisotropy and readily follows the external field. The narrow hysteresis loop and the finite remanence observed along the in-plane (hard axis) direction (Fig. 10c, open symbols) should be due to the presence of a small fraction of grains with an in-plane easy axis, probably those with [111] orientation, which were evidenced by the low intensity $L1_0$ (111) reflection in the XRD spectra. Furthermore, as the degree of chemical order may vary from grain to grain, producing a distribution of uniaxial anisotropy, a broad nucleation of magnetic reversal along the hard magnetic axis is what we actually observed in the in-plane loop.

The magnetization reversal of the optimized sample was investigated by measuring the angular dependence of the switching field, H_{sw} vs. ϕ , where ϕ is the angle between the applied field H and the easy axis EA (see inset in Fig. 11), and comparing the results with the theoretical Kondorsky (*incoherent switching* by domain wall motion in highly exchange

coupled systems) [39] and Stoner-Wohlfarth (S-W, coherent rotation for isolated particles) models [40]. The angular dependence of the switching field was determined by measuring, at different ϕ values (ranging between 0° and 90°), a series of easy axis *Direct Current Demagnetization* (DCD) curves using a non-conventional procedure that allows a meaningful comparison of experiments with simple models [41, 42]. After full saturation of the sample under a perpendicular field of 50 kOe in the SQUID magnetometer, the easy axis DCD curves were collected by using the vVSM, which allows vector angular measurements, by measuring at each angle ϕ the component of the remanent magnetization along the easy-axis direction M_{re} . These curves were used to determine the remanent coercivity H_{cr} (defined as the field where the remanence is equal to zero), which well represents the switching field H_{sw} [41, 42].

The angular dependence of the normalized switching field $H_{cr}(\phi)/H_{cr}(0^\circ)$ is reported in Fig. 11. The switching field gradually decreases with the angle, with a shallow minimum between 30° and 45° ($H_{cr}(45^\circ) = 0.9 H_{cr}(0^\circ)$, as compared to $0.5H_{sw}(0^\circ)$ of the S-W model) and an upturn at larger angles. Such behavior indicates a highly incoherent magnetization reversal process in presence of thermal activation, which should affect only the depth of the minimum, but not the angle at which it occurs [43,44]. In other words, the incoherent character of the reversal, due to inter-island coupling (magnetostatic and/or exchange) justifies the large shift of the switching minimum angle from the 45° angle of S-W model valid for coherent switching of isolated grains, as well as the observed upturn at larger angles [43,44].

CONCLUSIONS

We developed a thin film fabrication process at low temperature (below 400°C) for depositing highly textured $L1_0$ FePt films on a glass substrate, using a 30 nm thick Cr underlayer and MgO as a seed layer to induce an oriented texture and to lower the *fcc-fct* ordering temperature. The structural, microstructural and magnetic properties of FePt thin

films were investigated by varying the Ar sputter pressure P_{Ar} and the deposition temperature T_{dep} . From the analysis of the residual strain as a function of such parameters, we were able to optimize the growth process in such a way that a tensile strain occurred in the low P_{Ar} and T_{dep} region, favoring the desired out-of-plane c-axis growth of the *fct* FePt magnetically hard phase. Films deposited at $P_{Ar} = 5$ mTorr and $T_{dep} = 350$ °C showed the best properties in terms of chemical order parameter ($S = 0.72$), perpendicular squareness ratio ($M_r/M_s \sim 0.95$) and coercive field ($H_c \sim 14$ kOe). The reversal mechanism of such optimized sample was also investigated and the analysis indicated that the magnetization reversal is highly incoherent due to inter-island coupling (magnetostatic and/or exchange). Our results provide useful information on the low temperature growth of perpendicular anisotropy FePt films onto glass substrates, which are relevant for a variety of technological applications, such as magnetic recording and spintronic devices.

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Figure Captions

Figure 1. X-ray diffraction patterns of Cr (30 nm) deposited on glass substrate, at different substrate temperatures.

Figure 2. Rocking curve of the (200) peak in Cr films deposited at 300 °C.

Figure 3. X-ray diffraction patterns of FePt 10 nm film on $\text{MgO}_x/\text{Cr}_{30\text{nm}}/\text{Hoya}$ were $x = 1, 2, 3$ nm. The Cr (002) reflection is also indicated.

Figure 4. X-ray diffraction patterns of FePt(10 nm)/MgO(2 nm)/Cr(30 nm) films as a function of deposition pressure (3.5 - 15 mTorr) at constant deposition temperature ($T_{\text{dep}} = 350^\circ\text{C}$).

Figure 5. X-ray diffraction patterns of FePt(10nm)/MgO(2nm)/Cr(30nm) films as a function of deposition temperature (300 - 375 °C) at constant Argon pressure ($P_{\text{Ar}} = 5$ mTorr).

Figure 6. Residual strain ϵ as a function of P_{Ar} (panel a: $T_{\text{dep}} = 350^\circ\text{C}$) and T_{dep} (panel b: $P_{\text{Ar}} = 5$ mTorr)

Figure 7. FE-SEM image showing the surface of 10 nm $L1_0$ FePt island-like films, deposited at different deposition pressures (3.5 – 15 mTorr) and constant deposition temperature (350 °C). Bar scale: 100 nm.

Figure 8. FE-SEM image showing the surface of 10 nm $L1_0$ FePt island-like films, deposited at different deposition temperatures (300 – 375 °C) and constant Ar deposition pressure (5 mTorr). Bar scale: 100 nm.

Figure 9. (a) Cross-sectional TEM image of FePt(10 nm)/MgO(2 nm)/Cr(30 nm) films deposited at $P_{\text{Ar}} = 5$ mTorr and $T_{\text{dep}} = 350^\circ\text{C}$. (b) High resolution cross sectional TEM image showing the interface between the FePt, MgO and Cr layers. (c) Electron diffraction pattern.

Figure 10. Dependence of long range order parameter S (– $\blacktriangle$ –), perpendicular coercivity $H_{c\perp}$ (– $\bullet$ –) and parallel coercivity $H_{c\parallel}$ (– $\circ$ –) of Cr/MgO/FePt films on Argon pressure ($T_{\text{dep}} = 350^\circ\text{C}$) (a) and deposition temperature ($P_{\text{Ar}} = 5$ mTorr) (b). Normalized (M/M_s) in-plane (– $\circ$ –) and out-of-plane (– $\bullet$ –) hysteresis loops of the sample with optimized order parameter – $S = 0.72$ – and magnetic properties – $M_r/M_s \sim 0.95$ and $H_{c\perp} \sim 14$ kOe – ($T_{\text{dep}} = 350^\circ\text{C}$, $P_{\text{Ar}} = 5$ mTorr) (c).

Figure 11. Angular dependence of the experimental normalized switching field $H_{\text{sw}}(\phi)/H_{\text{sw}}(0^\circ)$ (– $\circ$ –) for the optimized sample ($T_{\text{dep}} = 350^\circ\text{C}$, $P_{\text{Ar}} = 5$ mTorr). The Kondorsky (dashed line) and Stoner-Wohlfarth (continuous line) models are reported for comparison.

Figures

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11