STRUCTURAL, MAGNETIC, AND MAGNETO-OPTIC PROPERTIES OF MnBi(Al, Pt) THIN FILMS: THERE IS STILL MORE TO LEARN

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Abstract - MnBi thin films with Al and Pt interlayers have been prepared by a sequential technique. Structural, magnetic, and magneto-optic properties at room temperature are discussed. We find that surface roughness intensifies with increasing film thickness due to the growth condition of Mn on Bi. Surface roughness is efficiently reduced by adding a thin Al or Pt interlayer. The coercivity strongly depends on adding interlayers or a protective overlayer. If the additional layer forms a rigid barrier, the MnBi formation is restricted perpendicular to the film surface, leading to a pronounced lateral growth and lower coercive fields. This could be related to an increase of 2.3% in volume between a stoichiometric Bi/Mn bilayer and the MnBi compound. The addition of Al interlayers does not reduce the Kerr effect considerably while a Pt interlayer reduces magnetization and, consequently, the Kerr effect.

KEYWORDS: MnBi, MAGNETO-OPTICS, KERR EFFECT, COERCIVITY, SURFACE ROUGHNESS, INTERLAYERS

INTRODUCTION

MnBi is one of the most intensively studied ferromagnetic materials and, yet, there are still open questions regarding its suitability as a magneto-optic recording media. MnBi shows an extraordinary large magneto-optic Kerr rotation of almost 2° at room temperature (as measured through a glass substrate) over a wide photon energy range [1]. Therefore, MnBi is an interesting candidate for magneto-optic storage applications in the blue spectral range. The hexagonal NiAs structure provides the necessary perpendicular magnetic anisotropy. However, there are some major complications which made an application impossible up to now. (i) A structural phase transition takes place at 340-355° C from a ferromagnetic low-temperature phase to a paramagnetic high-temperature phase [2]. (ii) The coercive field, H_c , increases with temperature showing a maximum at 280° C which is unfavorable for thermomagnetic writing [3]. (iii) Thin films deposited by the standard Bi/Mn-layering technique show a large grain size which increases noise in magneto-optic recording. To our knowledge, nobody has yet succeeded to grow MnBi by co-evaporation which would yield a better control over structural properties.

We started our investigation by reviewing the standard deposition technique of MnBi films and by verifying the magnetic and magneto-optic properties [4]. In contrast to previous work, the MnBi films are not covered with a protective SiO_x layer in order to be able to use surface-analyzing methods like scanning electron microscopy (SEM) and atomic force microscopy (AFM). The influence of Al and Pt additions on structural, magnetic, and magneto-optic properties of MnBi thin films is discussed in this paper. First results on Al additions have been published before [5].

EXPERIMENTAL

The MnBi thin films are deposited as subsequent Bi/Mn bilayers onto fused SiO_2 and glass substrates at ambient temperatures in a vacuum of 10^{-6} mbar. Al or Pt is added by placing an interlayer between subsequent Bi/Mn bilayers. A typical layering sequence is shown in Fig. 1. The thickness of

the Bi and Mn layers is fixed at 18 and 12 nm, respectively, to obtain after annealing the optimum stoichiometry. The deposition rate of 0.04 nm/s for Bi is such that a *c*-axis texture of the Bi layers is achieved which acts as a template for the MnBi formation. The deposition rate for Mn and Al is 0.2 nm/s and for Pt 0.01 nm/s. All films containing Al and Pt layers were annealed for one hour at temperatures between 300 - 380° C yielding films with *c*-axis orientation [5].

Magnetization measurements were performed on a commercial SQUID magnetometer. The polar Kerr spectra of all samples were measured through the substrate because of surface oxidation and poor reflectivity due to surface roughness while the Kerr hysteresis loops were obtained from both, the film and substrate side. The complex polar Kerr effect was measured in a fully-automated, high-resolution Kerr spectrometer over a wide photon energy range of 1.1-5.2 eV in magnetic fields up to 2.77 T at room temperature. For each data point, the difference of the rotations of the sample and a magneto-optically inactive reference, usually an aluminum mirror, is calculated in order to account for any rotation of the substrate and optical components in the stray field of the magnet. In addition, all measurements are taken in both, positive and negative magnetic fields in order to eliminate any rotations of nonmagnetic origin, as e. g., stress-induced birefringence of the films. The reflectivity was derived directly from the Kerr measurements relative to the Al reference mirror and is not corrected for the substrate.

FIG. 1. Typical layering sequence of the MnBi(Al, Pt) thin films. The thickness of the Bi and Mn layers is chosen to yield the optimum MnBi stoichiometry after annealing.

STRUCTURAL PROPERTIES

First, the influence of Al and Pt addition on the structural properties of MnBi thin films is discussed. The addition of Al and Pt does not have much effect on the preferred c -axis orientation of the MnBi(Al, Pt) thin films as is evident from x-ray diffraction analysis shown in Fig. 2. For the as-grown films (Fig. 2(a)), Bi(00 l) reflections are present indicating strong c -axis orientation. A few spurious features due to the sample holder are labeled with an asterisk. After annealing, the MnBi (Fig. 2(b)) as well as the films containing Al and Pt layers (Figs. 2(c) and (d), respectively) show strong MnBi(00 l) reflections indicating the hexagonal NiAs-type structure of MnBi. The small shifts observed in 2θ are due to a misalignment of the samples when mounting onto the x-ray sample holder. Note that there is no evidence for a second phase, like MnAl, MnPt or the high-temperature Mn_{1.08}Bi phase, as has been found by other groups [6, 7]. There is, however, a small amount of residual Bi in the annealed MnBi (Fig. 2(b)) and the MnBiPt film (Fig. 2(d)).

As the addition of Al or Pt neither reduces c -axis orientation nor changes the MnBi crystal lattice constant, the question arises what happens to the interlayers at all. In order to elucidate this question, Rutherford backscattering (RBS) experiments were performed using 1.4 MeV He⁺ ions and a scattering angle of 170° [8]. A comparison between the RBS spectra before (dashed line) and after (solid line) annealing of a Bi/Mn/Al(2 nm)/Bi/Mn sample is displayed in Fig. 3. The surface-backscattering energies of the components are marked by vertical lines. The

FIG. 2. X-ray diffraction spectra of MnBi(Al, Pt) thin films before and after annealing.

FIG. 3. Rutherford-backscattering spectra before (dotted line) and after (solid line) annealing of (a) a Bi/Mn/Al(2 nm)/Bi/Mn and (b) a Bi/Mn/Pt(2 nm)/Bi/Mn sequence. The vertical lines mark the surface-backscattering energy of the constituents.

offset to lower energies is indicative of the location of a component inside the film relative to the surface. Typically, heavy elements, like Bi, have less energy loss than light elements, like Mn or O. Focusing on the dominant Mn and Bi features, a distinct separation into two layers, represented by a double peak in the RBS spectra, is recognized in both systems in the as-grown state. This simply represents the layering sequence of the deposition (see Fig. 1).

After annealing, the separation is preserved to a great extent in the Al-doped system while the Pt-doped system reveals a complete interdiffusion of the two Bi/Mn bilayers. A small amount of oxygen is present directly at the surface increasing after annealing by a factor of about 2.5 [8]. It most certainly forms with Mn a thin Mn₃O₄ surface layer. This is corroborated by the asymmetry of the Mn double peaks, especially in the Bi/Mn/Pt/Bi/Mn sample, implying a higher Mn density at the surface. No anisotropy is observed, however, in the Bi/Mn/Al/Bi/Mn sample before annealing. This could indicate that the Al interlayer getters some of the oxygen during deposition thus forming Al₂O₃, which is a very firm oxide. In addition, this would explain why the two Bi/Mn bilayers are still separated after annealing. Pt, being a noble metal, will not form an oxide and will hence be a minor diffusion barrier to the MnBi formation across the interlayer. As will be discussed in the next sections, the different structural characteristics of Al and Pt interlayers have a strong effect on the magnetic and magneto-optic properties.

MAGNETIC PROPERTIES

We will briefly review the magnetic properties of pure MnBi films as a function of thickness [4]. The SEM pictures of a single Bi/Mn bilayer in comparison to a Bi/Mn/Bi/Mn double bilayer is shown in Fig. 4. By enlarging the film thickness d_{MnBi} , i.e. the number of Bi/Mn bilayers, surface roughness increases strongly during deposition and does not change after annealing. An AFM [9] study indicates that the surface roughness is generated during growth of Mn onto Bi.

The magnetic properties of annealed films are changing as well. The polar Kerr hysteresis loops, measured from the film side, show a strong decrease of the coercive field, H_c , from 1.25 T in a single Bi/Mn bilayer ($d_{\text{MnBi}} = 41$ nm) to 0.4 T in a (Bi/Mn)₃ sequence ($d_{\text{MnBi}} = 90$ nm), as plotted in Fig. 5. The reduction in H_c with increasing thickness can be understood qualitatively as an increase in MnBi grain size on the basis of a simple model which assumes spherical, single-domain particles [10]. From the SEM pictures, we can estimate a grain size of 200-300 nm in thick MnBi films. The high coercive fields are in contrast to earlier work [3, 11], where for a similar film thickness values of $H_c \leq 0.5$ T were measured. As will be discussed later, this is due to the altered growth condition when depositing a protective SiO_x layer.

Besides the decrease of H_c , an additional change in the characteristics of the magnetization reversal is observed. While a single Mn/Bi bilayer is reversing smoothly, as revealed in a symmetric 'S'-shaped slope of the inner loop (see Fig. 5(a)), a thick MnBi film exhibits a sudden onset of magnetization reversal before leveling off very slowly (see Fig. 5(b)).

In order to understand the magnetization reversal from a theoretical point of view, hysteresis loops were simulated by a Monte-Carlo method using a micromagnetic model which

FIG. 4. SEM picture of (a) a Bi(22 nm)/Mn(19 nm) bilayer and (b) a Bi/Mn/Bi/Mn double bilayer with a Bi and Mn layer thickness of 18 and 12 nm, respectively. Both pictures are taken before annealing. The characteristics do not change after annealing.

FIG. 5. Polar Kerr-hysteresis loops at a photon energy of 1.8 eV of (a) a Bi(22 nm)/Mn(19 nm) bilayer and (b) a (Bi/Mn)₃ sequence with a Bi and Mn layer thickness of 18 and 12 nm, respectively. The loops are taken from the film side after annealing.

includes dipole interaction, wall energy, Zeeman energy, and an energy barrier describing the reversal of a single grain [12]. The film is assumed to consist of cells with height, h , and lateral dimension, L , on a square lattice. Using experimental values for h and L , the model accurately accounts for the thickness dependence of the coercivity as demonstrated in Fig. 6. The value of h corresponds to the experimental film thickness while L is estimated from the SEM pictures (Fig. 4). However, the coercivity is a factor of

FIG. 6. Simulated hysteresis loops for a square lattice cell size of (a) height $h = 40$ nm and lateral dimension $L = 50$ nm and (b) $h = 90$ nm and $L = 300$ corresponding to a MnBi single grain.

FIG. 7. Disorder dependence of the hysteresis loops for $h = 90$ nm and $L = 300$ nm for (a) $\Delta_G = 0$ (no disorder) and (b) $\Delta_G = 0.4$.

two smaller than the experimental value indicating that the model is too simple to predict correct values of H_c .

The model can in addition explain the change in the magnetization reversal. As is evident from the SEM pictures, the MnBi grain size is randomly distributed and therefore, disorder should be incorporated into the model. This would yield a random distribution of L . As this is very difficult to simulate, due to the complicated evaluation of the dipole term, disorder is added to the model by assuming a random fluctuation of L only in the Zeeman term, since this is the leading term being quadratic in L [12]. The resulting hysteresis loop is plotted in Fig. 7 for a Gaussian distribution of L of width of $\Delta_G = 0.4$ and compared to the case without disorder.

Adding a thin Al or Pt interlayer between two Bi/Mn bilayers strongly reduces surface roughness as evidenced in the SEM pictures in Fig. 8. Comparing with single and double Bi/Mn bilayers (see Fig. 4), surface roughness decreases to the value of a single Bi/Mn bilayer. As demonstrated in an AFM study [9], the Pt produces a plateau-like structure when growing on a Bi/Mn bilayer, explaining the smoothing of the surface.

As shown in Fig. 9, the corresponding polar Kerr loops, measured from the film side, display a different behavior for Al and Pt interlayers. When adding an Al interlayer of $d_{Al} \geq 1.5$ nm, the Kerr loops develop a peculiar form. Starting at the highest positive magnetic field, magnetization reversal takes place in two steps. Shortly after reversing the field, at $H \approx -0.2$ T, the Kerr rotation, θ_K , shows a sharp decline before increasing again to a positive saturation value at $H \approx -1$ T. This behavior can be explained by assuming two decoupled magnetic layers with different values of H_c and opposite sign of θ_K . The inset in Fig. 9(a) shows a superposition of two model loops with the proposed characteristics correctly describing the experimental data. An antiferromagnetic coupling between the two layers can be

FIG. 8. SEM picture of (a) Bi/Mn/Al(2 nm)/Bi/Mn and (b) Bi/Mn/Pt(0.3 nm)/Bi/Mn with a Bi and Mn layer thickness of 18 and 12 nm, respectively. Both pictures are taken before annealing. The characteristics do not change after annealing.

excluded as in that case the switching should take place *before* reversing the external field [8]. Measuring the Kerr hysteresis loops through the substrate [8], reveals a common square loop with negative θ_K and a coercive field $H_c \approx 0.4$ T. In conclusion, by adding an Al interlayer with $d_{Al} \geq 1.5$ nm the MnBi film is divided into two decoupled magnetic layers, where the top one has a large H_c of ≈ 1 T, similar to a single Bi/Mn bilayer, and the bottom one has a very small H_c of ≈ 0.4 T, as in a thick MnBi film. Note that the bottom layer now manifests the same value of H_c as reported in samples

FIG. 9. Polar Kerr-hysteresis loops at a photon energy of 2.0 eV of (a) Bi/Mn/Al(2 nm)/Bi/Mn and (b) Bi/Mn/Pt(2 nm)/Bi/Mn. The loops are taken after annealing from the film side.

with a protective SiO_x layer [3, 11].

Adding a Pt interlayer (see Fig. 9(b)), yields a square loop with H_c slightly reduced as compared to a single Bi/Mn bilayer. By increasing the Pt-interlayer thickness gradually from 0.3 to 2 nm, H_c increases progressively from the value of a double Bi/Mn bilayer to the value of a single Bi/Mn bilayer [9]. This is surprising as the RBS data (see Fig. 3) show that the Pt interlayer merges completely with the Bi/Mn bilayers. Hence, one would expect that the coercivity remains constant at the value of a double Bi/Mn bilayer.

In order to understand the effect of adding Al and Pt interlayers or a protective SiO_x layer, let us discuss some consequences of the layering-deposition method. Deposition of a Bi/Mn bilayer results in an average Mn_xBi_{1-x} composition where the Mn concentration x_{Mn} is obtained by

$$x_{Mn} = \frac{\rho_{Mn} d_{Mn}}{M_{Mn}} \left(\frac{\rho_{Mn} d_{Mn}}{M_{Mn}} + \frac{\rho_{Bi} d_{Bi}}{M_{Bi}} \right)^{-1}. \quad (1)$$

Inserting $\rho_{Bi} = 9.8 \text{ g/cm}^3$, $\rho_{Mn} = 7.43 \text{ g/cm}^3$, $d_{Bi} = 18 \text{ nm}$, $d_{Mn} = 12 \text{ nm}$, $M_{Bi} = 209 \text{ g/mol}$ and $M_{Mn} = 55 \text{ g/mol}$, we get $x_{Mn} = 0.66$. This is far away from the ideal stoichiometry of $x_{Mn} = 0.5$. As evidenced by energy-dispersive x-ray analysis, about 10% of the Mn evaporates during annealing. Furthermore, a small amount of Mn is bound at the surface by oxygen (see Fig. 3). As a result, the complete Bi layer but only part of the Mn layer is engaged in the MnBi formation process. We can calculate the average volume, $v_{Bi/Mn}$, per formula unit MnBi assuming a Bi and Mn layer thickness corresponding to the ideal stoichiometry

$$v_{Bi/Mn} = v_{Bi} + v_{Mn} = \frac{1}{N_A} \left(\frac{M_{Bi}}{\rho_{Bi}} + \frac{M_{Mn}}{\rho_{Mn}} \right), \quad (2)$$

where N_A is Avogadro's number. Inserting the values, we calculate $v_{Bi/Mn} = 47.7 \text{ Å}^3$. This has to be compared with the cell volume per formula unit of NiAs-type MnBi, $v_{MnBi} = \sqrt{3}/4 a_{MnBi}^2 c_{MnBi} = 48.8 \text{ Å}^3$ using $a_{MnBi} = 4.29 \text{ Å}$ and $c_{MnBi} = 6.126 \text{ Å}$ [13, 14]. This is 2.3% larger than $v_{Bi/Mn}$. As a consequence, the film has to expand during crystallization generating internal pressure. This would lead to an increasing surface roughness when enlarging film thickness. The thicker the films the larger the disorder which is induced by the nucleation of individual crystallites. However, as has been discussed before, surface roughness develops already during deposition creating void space. Therefore, part of the internal stress induced by the volume increase during crystallization will be relaxed through the void space.

The change in coercivity behavior of MnBi films can also be explained by a volume increase. Depositing a hard interlayer, like Al₂O₃, or a protective layer, like SiO_x, will restrict expansion of the crystallites perpendicular to the surface, leading to a pronounced lateral growth of the MnBi crystallites. This will lead to larger lateral dimensions and a better interconnection of individual grains reducing the wall energy at the grain boundary. Both effects reduce coercivity which explains the small values observed.

MAGNETO-OPTIC PROPERTIES

In the last section we will discuss the magneto-optic properties of Al and Pt doped MnBi thin films. The complex polar Kerr spectra in the remanence state and the reflectivity relative to an Al reference mirror of a MnBiAl(0.3 nm) and two MnBiPt(d_{Pt}) films, with $d_{Pt} = 0.3$ and 1.5 nm, are shown in Fig. 10, as measured through the substrate. The remanence state is chosen because in that case no spurious effects of the Faraday rotation of the substrate are present that could falsify the spectra, especially in the UV part of the photon-energy range. Each spectra is divided by a factor of 1.5 to account for the interface-enhancement effect when measuring through the quartz substrate [15].

For the MnBiAl multilayer, two major peaks are observed in θ_K at 1.9 and 3.4 eV with an absorptive line shape. The maximum Kerr rotation amounts for both structures to -0.9° , the latter being very advantageous for blue recording. The corresponding features in the Kerr ellipticity, η_K , exhibit dispersive line shapes. The maximum value, $\eta_K = -0.9^\circ$, is found at 4.5 eV. The spectra are similar in size and shape for pure MnBi (not shown). Our measurements are in good agreement with recently published spectra [1, 16]. However, these studies were made on SiO_x-protected MnBi films. There, the peaks positions are also found at 1.9 and 3.4 eV but, unlike our results, the second peak ($\theta_K = 1.5^\circ$) is smaller by 15% than the one at lower energy ($\theta_K = 1.75^\circ$). To compare with our findings, the rotations have to be corrected for the interface effect. This yields $\theta_K = 1.16^\circ$ and 1° in fair agreement with our data.

FIG. 10. (a) Complex polar Kerr spectra in the remanence state and (b) reflectivity of MnBiAl(0.3 nm) (■), MnBiPt(0.3 nm) (●), and MnBiPt(1.5 nm) (▲). Open symbols represent the Kerr ellipticity. Measurements were obtained through the substrate at room temperature.

Adding Pt to MnBi reduces the Kerr rotation evenly across the photon-energy range except for the sample with a 2 nm thick Pt interlayer. Here, the first peak at 1.9 eV is more reduced than the peak at 3.4 eV. In the same way, the magnetization, μ , decreases as a function of Pt content from $\mu = 536$ kA/m to 304 kA/m for $d_{\text{Pt}} = 0.3$ and 1.5 nm. Normalizing the maximum Kerr rotation, θ_K^{max} at 3.4 eV to the magnetization, we get a ratio $\theta_K^{\text{max}}/\mu = 1.31 \times 10^{-3}$ and 1.20×10^{-3} deg m/kA, respectively, which is equal to within 10%. This indicates that the addition of Pt reduces primarily the magnetization of the MnBi films. As a consequence, θ_K is decreasing in the same way being in first order proportional to μ . At present, we do not know what causes the magnetization reduction.

The reflectivity, R , as plotted in Fig. 10(b), decreases continuously with photon energy in all samples. It is the largest for the Al doped film where R declines from 50% at 1.1 eV to 30% at 4 eV. This is in agreement with published data [16]. The Pt-doped films have lower values of R starting at 45% at 1.1 eV and decreasing to 20% at 4 eV. Note that this is not an absolute measurement. Therefore, the reflectivity might have an absolute error of several percent but the relative changes should be real. If we calculate the shot-noise limited figure of merit, $FOM = \sqrt{R}(\theta_K^2 + \eta_K^2)^{1/2}$, a value of $FOM = 0.49^\circ$ is derived at 3 eV. This is considerably more than in Co/Pt multilayers [17] explaining the high interest in this compound as a potential magneto-optic storage material.

As *ab-initio* calculations of the Kerr effect have made an immense progress in the last few years, we would like to compare our data with recent first-principles studies on the Kerr effect of MnBi [18, 19]. The theoretical studies show that the high intrinsic Kerr effect in MnBi is due to the coincidence of three favorable issues: (i) a large magnetic moment of $3.7 \mu_B$, (ii) a large spin-orbit coupling of Bi, and (iii) a large hybridization between the d and p states of Mn and Bi. This leads to strong, dipole-allowed $\text{Mn-}3d \rightarrow \text{Bi-}5p$ as well as $\text{Bi-}5p \rightarrow \text{Mn-}4d$ transitions which are magneto-optically active because of (i) and (ii). Although the first peak at 1.9 eV is computed in good agreement with experiment [18] the second peak at 3.4 eV does not appear in the primary calculations. At present, there are two approaches to account for the high-energy peak. One is by assuming a change in stoichiometry from MnBi to a Heusler-like Mn_2Bi [18], the other one is by assuming oxygen filling in the interstitial sites of the MnBi unit cell [19]. Future measurements have to clarify if any of these mechanisms is responsible for the peak at 3.4 eV or if the primary calculations need to be modified.

CONCLUSIONS

Regarding the suitability of MnBi films as a magneto-optic recording material the following conclusions can be made. MnBi has a very high room-temperature Kerr rotation θ_K in the blue spectral range reaching 1° . By using a sequential deposition technique, Al and Pt interlayers can be added without degrading θ_K considerably. The interlayers

improve surface quality, reduce grain size and can be used to optimize the coercivity H_c . The large grain size and surface roughness is found to be due to the growth behavior of Mn on Bi during deposition. Consequently, co-evaporation of Mn and Bi might be the key to an efficient reduction of grain size and surface roughness. Concerning the major hindrances for an application, further magneto-optic measurements at elevated temperatures will be performed in near future to investigate the influence of the Al and Pt interlayers on the structural phase transition and the H_c anomaly.

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