Influence of Ru/Ru–SiO₂ underlayers on the microstructure and magnetic properties of CoPt–SiO₂ perpendicular recording media

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Abstract

SiO₂ was doped into Ru underlayer to reduce the grain size of CoPt–SiO₂ perpendicular media. The effects of SiO₂ volume fraction and sputtering deposition pressure of Ru–SiO₂ underlayer on the microstructure and magnetic properties of CoPt–SiO₂ media were studied. Increasing SiO₂ volume fraction in Ru–SiO₂ layer decreased the grain sizes of Ru and CoPt. Adding 5% SiO₂ to Ru–SiO₂ layer increased the coercivity and enhanced the exchange decoupling and thermal stability of CoPt–SiO₂ layer. A further increase in SiO₂ volume fraction caused the deterioration of magnetic properties of CoPt–SiO₂ layer. Deposition of Ru–SiO₂ layer at 1.3 Pa resulted in a smaller activation volume and higher thermal stability of the CoPt–SiO₂ media than that deposited at 0.4 Pa.

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1. Introduction

One of the major challenges in further increasing the areal density of perpendicular magnetic recording media is in reducing the grain size and improving the signal to noise ratio by minimizing transition noise [1]. Efforts have been made to reduce grain size by adding oxide-based materials into the magnetic layer [2] or by depositing the magnetic layer in an argon and oxygen environment [3]. However, too much oxide in the magnetic layer would reduce its magnetic anisotropy, leading to increased thermal instability due to the superparamagnetic effect. An alternative approach is to reduce the grain size of the intermediate layer and hence, the grain size of the magnetic layer. The addition of synthetic nucleation layers [4], RuCr–oxide underlayer [5], NiAl underlayer [6] and Ar-etched Ru underlayer [7,8] has been reported to be able to decrease the grain size of the magnetic layer to below 6 nm. However, a deteriorated texture or reduction of the nucleation field of the magnetic layer was also observed. Recently, Yuan et al. reported that doping oxide into the Ru underlayer could reduce the grain size of the magnetic layer [9]. The reduction of grain size was based on a large volume fraction of oxide addition into the Ru underlayer, which deteriorated the texture of the latter. However, their report on the magnetic properties of the media with reduced grain size was insufficient for the evaluation of the effects of Ru-oxide underlayer on the recording media. A systematic understanding of the magnetic properties of the recording media with reduced grain size was important because it would be desirable to reduce grain size without deteriorating the magnetic properties for practical applications. In this paper, SiO₂ was doped into the Ru intermediate layer to reduce the grain size of the CoPt–SiO₂ magnetic layer. The effects of SiO₂ volume fraction and sputtering pressure of Ru–SiO₂ intermediate layer on the microstructure and magnetic properties of CoPt–SiO₂ magnetic recording media were studied.

2. Experiments

Granular media of CoPt–SiO₂(15 nm)/Ru–SiO₂(10 nm)/Ru₈₈ (bottom Ru, 15 nm)/Ta (4 nm)/glass were deposited at room temperature by magnetron sputtering. The base pressure of sputtering was lower than 5 × 10⁻⁶ Pa. The Ta layer and Ru₈₈ layer were deposited at 0.4 Pa. The Ru–SiO₂ (hereafter referred to as Ru₈₈) layer was deposited at 0.4 Pa and 1.3 Pa argon pressures respectively. The Co₇₂Pt₂₈, Ru, Ta and SiO₂ targets (made by Toshima MFG and Williams Advanced Materials with purities of 99.9%) were used for the sputtering. DC and AC sputtering were used for the metal and SiO₂ targets respectively. The volume fraction of SiO₂, adjusted by changing the sputtering time of SiO₂ relative to that of Ru₈₈, was varied from 0 to 20% in the Ru₈₈ underlayer. The composition and thickness of the CoPt–SiO₂ layer were kept constant in all samples. Coercivities of the media were measured using
the vibrating sample magnetometer (VSM, Lakeshore 7407) under maximum applied field of 1.2 T. Time-dependent remanent coercivities of the media were measured using the alternating gradient force magnetometer (AGFM, Princeton MicroMag 2900) under maximum applied field of 2 T. The textures of the films were investigated using Philips XPert X-ray diffractometer (XRD) with CuKα1 radiation (source wavelength is 1.5406 Å). The microstructures of the films were investigated using JEOL-2010 transmission electron microscope (TEM). The TEM samples of Ru underlayer were prepared on carbon coated Cu grids and those of CoPt–SiO2 layers were prepared by grinding and polishing the films sputtered on glass substrates.

3. Results and discussions

Fig. 1 shows the full width at half maximum (FWHM) values of Ru (00.2) rocking curves as a function of SiO2 volume fraction at the deposition pressures of 0.4 Pa and 1.3 Pa, respectively. For the samples deposited at 0.4 Pa, the FWHM increased from 3.4° to 3.9°. For those deposited at 1.3 Pa, the FWHM increased from 3.8° to 4.1°. The FWHM of Ru, deposited at 0.4 Pa was smaller than those deposited at 1.3 Pa, which was attributed to the larger kinetic energy of Ru adatoms at 0.4 Pa deposition than that at 1.3 Pa deposition [10]. The texture information of the CoPt layer could not be unequivocally determined from the XRD results due to the very close lattice constant-c between CoPt (00.2) and Ru (00.2).

Fig. 2 shows the bright field TEM images of Ru layer deposited with different SiO2 volume fractions under different pressures. Fig. 2(a) showed that for film deposited at 0.4 Pa without SiO2 addition, the Ru grain size was very large and grain boundaries were ill defined. This was due to the high mobility of Ru adatoms at 0.4 Pa deposition. With addition of SiO2, isolated Ru grains were observed. The grain sizes (center to center distance, averaged over randomly selected 100 pairs of grains) were 6.7 ± 0.9 nm and 5.3 ± 1.3 nm for 10% and 20% addition of SiO2, respectively. The grain boundaries were deduced to be SiO2 due to the phase separation between Ru and SiO2. For films deposited at 1.3 Pa, isolated grains were observed even without SiO2 addition as shown in Fig. 2(d). This was caused by the low mobility of Ru atoms and the “shadowing effect” [11] of Ru grains at 1.3 Pa deposition. The grain boundaries were deduced to be voids. With the addition of SiO2, these grain boundaries became a hybrid of voids and oxides. The grain sizes were 6.6 ± 0.7 nm, 6.4 ± 0.9 nm and 4.5 ± 1.0 nm for 0%, 10% and 20% of SiO2, respectively.

Fig. 3 shows the bright field TEM images and grain size distributions of CoPt–SiO2 media, with Ru, deposited at 0.4 Pa (Fig. 3(a)–(d)) and 1.3 Pa (Fig. 3(e)–(h)). The grain sizes in Fig. 3 (d) and (h) were center to center distance of two neighboring grains and counted over randomly selected 100 grain pairs. The average and standard deviations of the grain sizes, which were obtained by log-normal fitting to the data, were indicated in the figures. Addition of SiO2 into the Ru layer decreased both the grain size and size distribution of CoPt grains. The reduction of grain size was attributed to the smaller Ru grains and the enhanced oxide segregation in the initial growth region of the CoPt layer [12]. The average grain size of CoPt layer with Ru layer deposited at 1.3 Pa was larger than that at 0.4 Pa when SiO2 content was smaller than 20 vol.%. This is due to the better isolated Ru grains in the Ru layer deposited at 1.3 Pa than at 0.4 Pa, as shown in Figs. 2 and 3(c) and (g). However, although addition of SiO2 into Ru layer led to smaller magnetic grain size, some of the magnetic grains became interconnected, thus increasing the exchange coupling of magnetic grains, as shown in Fig. 3(b) and (f).

Fig. 4 shows the dependence of out-of-plane hysteresis loops and coercivities of CoPt–SiO2 media on the SiO2 volume fraction and deposition pressure of Ru layer. For media with Ru deposited at 1.3 Pa, adding 5 vol.% SiO2 into the Ru did not lead to noticeable changes of the coercivity. With further increase in SiO2 content, the coercivity decreased. For media deposited at 0.4 Pa, adding SiO2 into the Ru layer did not lead to appreciable changes of the coercivity. However, the media with Ru deposited at 1.3 Pa displayed higher coercivities than those with Ru deposited at 0.4 Pa. The coercivity of the media was determined by both the microstructure of the CoPt grains and magnetization reversal mechanism as described below.

In order to understand the magnetization reversal mechanism of the CoPt–SiO2 media, angular dependent coercivities were measured. Fig. 5 shows the angular dependent coercivity curves of CoPt–SiO2 media with Ru layer deposited at different pressures and with different SiO2 volume fractions. The Stoner–Wohlfarth (S–W) model represents fully exchange-decoupaged magnetization reversal and domain wall motion (DWM) (Kondorsky model [13]) represents strongly exchange-coupled reversal. For films with Ru, deposited at 0.4 Pa, adding 5 vol.% SiO2 into the Ru resulted in the magnetization reversal mechanism becoming closer to the S–W mode. This is attributed to improved isolation of Ru grains and hence, better isolated CoPt grains. With a SiO2 content of 10 vol.%, the curve did not change appreciably. With a further increase in SiO2 content beyond 15 vol.%, the curves approached the DWM mode. When the Ru was deposited at 1.3 Pa, no significant changes were observed when SiO2 content was smaller than 10 vol.%. However, the curves approached the DWM mode when SiO2 content was larger than 10 vol.%. As seen from Fig. 1, addition of SiO2 into the Ru layer deteriorated the texture of the magnetic grains, which might account for the observed changes in the curves of the angular dependent coercivities. When the SiO2 content exceeded 10 vol.%, the sizes of Ru and CoPt grains decreased. The volume of non-magnetic grain boundary in the CoPt layer increased. As a result, the non-magnetic grain boundary in the CoPt layer would possibly be thinner if one-to-one grain growth was not realized [8]. Thinner non-magnetic grain boundary would lead to stronger exchange coupling between magnetic grains [14]. The reversal mechanisms of media with Ru, deposited at 1.3 Pa were closer to S–W mode than those of media deposited at 0.4 Pa. This result could be attributed to the existence of void grain boundaries in the Ru layer deposited at 1.3 Pa which improved the grain isolation of CoPt layer as shown in Fig. 2.

Fig. 6(a) shows the activation volume (Vact) of the CoPt recording layer as a function of SiO2 volume fraction in Ru, under deposition pressures of 0.4 and 1.3 Pa. The values of Vact were obtained by fitting time-dependent remanent coercivities [15,16], using the least-squares method. The fitting quality was evaluated by the square of the multiple correlation coefficients R2 where 0 ≤ R2 ≤ 1 with R2 = 1 corresponding to perfect data fitting. R2 of all the fittings were larger than 0.97, except for 0.4 Pa deposition with 5 vol.% SiO2 where
$R^2 = 0.94$ and with 20 vol.% SiO$_2$ where $R^2 = 0.78$. The $V_{\text{act}}$ was an indirect parameter of media representing the areal density [17]. The smaller the $V_{\text{act}}$, the higher the areal density that the media could support. For media with Ru$_4$ deposited at 0.4 Pa, adding 5 vol.% SiO$_2$ into the Ru$_4$ layer reduced $V_{\text{act}}$ slightly. With further increase in SiO$_2$ beyond 5 vol.%, $V_{\text{act}}$ increased. For media with Ru$_4$ deposited at 1.3 Pa, adding 5 vol.% SiO$_2$ into the Ru$_4$ did not result in an appreciable change in $V_{\text{act}}$. However, the $V_{\text{act}}$ increased slightly with further increase in SiO$_2$ content. The $V_{\text{act}}$ was determined by the size of magnetic grains and the strength of exchange coupling between these grains. Smaller grain size and weaker exchange coupling decrease the $V_{\text{act}}$ and vice versa. For media with Ru$_4$ deposited at 0.4 Pa, adding 5 vol.% SiO$_2$ into

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Fig. 2. Planar-view TEM images of Ru$_4$ layer deposited with the SiO$_2$ volume fraction and pressure of (a) 0% and 0.4 Pa, (b) 10% and 0.4 Pa, (c) 20% and 0.4 Pa, (d) 0% and 1.3 Pa, (e) 10% and 1.3 Pa and (f) 20% and 1.3 Pa. TEM images in this figure were obtained from samples deposited on the carbon coated Cu grids.
the Ru, led to the decrease in grain size and strength of exchange coupling between grains, since Ru was nearly a continuous layer without SiO₂ addition. As a result, the $V_{act}$ decreased. However, the $V_{act}$ of the media with Ru deposited at 1.3 Pa was higher than those of the media with Ru deposited 0.4 Pa. This was caused by the smaller and more isolated CoPt grains at 1.3 Pa compared to that at 0.4 Pa.

**Fig. 3.** A comparison of TEM images of CoPt-SiO₂ media with Ru deposited under different conditions. Images (a) and (b) are planar-view images deposited at 0.4 Pa without and with 10 vol.% SiO₂ in Ru. Images (e) and (f) are planar-view images deposited at 1.3 Pa without and with 10 vol.% SiO₂ in Ru. Images (c) and (g) are cross-section images of the samples in (b) and (f). Plots (d) and (h) are the grain size distributions of the CoPt media with Ru deposited at 0.4 Pa and 1.3 Pa and various SiO₂ additions. The TEM images were obtained from samples deposited on the glass substrates.
Fig. 6(b) and (c) shows the effective anisotropy constant ($K_{\text{ueff}}$) and the thermal stability factor (TSF, defined as $K_{\text{ueff}}V_{\text{act}}/k_B T$) of CoPt–SiO$_2$ media as a function of the SiO$_2$ volume fraction in Ru deposited at 0.4 Pa and 1.3 Pa. The TSF was obtained by fitting the time-dependent remanent coercivities into Sharrock’s equation as follows [18]

$$H_c(t) = H_0 \left\{ 1 - \left[ \frac{k_B T}{K_{\text{ueff}} V_{\text{act}} \ln f_0 t} \ln 2 \right]^n \right\}$$

where $H_c(t)$ is the time-dependent remanent coercivity, $k_B$ is the Boltzmann constant, $T$ is the temperature taken as 298 K, $f_0$ is the attempt frequency taken as $10^9$, and parameter $n$ is taken as 2. $R^2$ of all the fittings were larger than 0.97, except for 0.4 Pa deposition with 5 vol.% SiO$_2$ where $R^2 = 0.93$ and with 20 vol.% SiO$_2$ where $R^2 = 0.8$. $K_{\text{ueff}}$ was then obtained by dividing TSF by $V_{\text{act}}/k_B T$. For media with Ru deposited at 0.4 Pa, adding SiO$_2$ into Ru layer did not cause noticeable changes in $K_{\text{ueff}}$. However, for media with Ru deposited at 1.3 Pa, adding 5 vol.% SiO$_2$ into Ru layer resulted in a slight increase of $K_{\text{ueff}}$. 

Fig. 3 (continued).
The $K_{\text{eff}}$ decreased when SiO$_2$ content was 10 vol.%. Further increasing SiO$_2$ content did not change $K_{\text{eff}}$ appreciably. From Fig. 6(c), for media with Rut deposited at 0.4 Pa, adding 5 vol.% SiO$_2$ into Rut layer decreased the TSF slightly. Further increasing SiO$_2$ content beyond 5 vol.% resulted in a slight increase in TSF. When Rut was deposited at 1.3 Pa, adding 5 vol.% SiO$_2$ into the Rut layer increased the TSF. When SiO$_2$ was increased to 10 vol.%, the TSF decreased. Further increasing SiO$_2$ content beyond 10 vol.% did not change TSF appreciably. The TSF is proportional to the product of $K_{\text{eff}}$, which is determined by the microstructure of the film [19], and $V_{\text{act}}$ (which is determined by the grain size and exchange decoupling between the grains). Therefore, the changes of microstructure, grain size and exchange coupling affected the TSF.

4. Summary

In this paper, the effects of SiO$_2$ volume fraction and sputtering pressure of Rut layers on the microstructure of the layers and the magnetic properties of CoPt–SiO$_2$ recording media were studied. When Rut layer was deposited at 0.4 Pa, the sizes of the Ru and CoPt grains decreased from 6.7±0.9 to 5.3±1.3 and from 8.2±1.9 to 5.1±1.1 with the addition of SiO$_2$ up to 20 vol.% respectively, but their c-axis orientations slightly deteriorated. Increasing the deposition pressure of the Rut layer from 0.4 Pa to 1.3 Pa further reduced the grain size due to the porosity in the grain boundary regions of the Ru$_x$ layer. Addition of 5 vol.% SiO$_2$ into the Ru$_x$ layer increased the coercivity, the exchange decoupling and the thermal stability of the CoPt layer. However, further increase of SiO$_2$ volume fraction in the Ru$_x$ layer decreased the grain size, the coercivity and the exchange decoupling, degrading the thermal stability of the CoPt layer. On the other hand, CoPt media with Ru$_x$ layer deposited at 1.3 Pa showed a much higher coercivity, better exchange decoupling and better thermal stability than those of the media with Ru$_x$ layer deposited at 0.4 Pa. The above results suggest that small amounts of SiO$_2$ addition and deposition pressure of 1.3 Pa can reduce the grain size of the Ru$_x$ layer and hence, the recording layer without adversely affecting the magnetic properties of the recording layer.

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Fig. 6. Plot (a) activation volume ($V_{\text{act}}$), (b) effective anisotropy constant ($K_{\text{ueff}}$) and (c) thermal stability factor of the CoPt–SiO$_2$ recording media as a function of the SiO$_2$ volume fraction in Ru, deposited at 0.4 Pa and 1.3 Pa. The error bars represented the 95% confidence interval on the estimates of the parameters obtained by fit. The lines were only used to guide the readers’ eyes.

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