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Sputtering pressure effect on microstructure of surface and interface, and on coercivity of Co/Pt multilayers

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Thin Co/Pt multilayers were prepared on Si and glass substrates by sputtering with Ar pressures ranging from 2.5 to 15 mTorr. The bilayer structure of the samples was $\text{Co}(3 \text{ \AA})/\text{Pt}(15 \text{ \AA}) \times 17$, and all samples had the easy axis of magnetization perpendicular to the sample surface as determined with a SQUID magnetometer. All samples retained the layered structure, as revealed by low-angle x-ray diffraction. In addition, diffraction peaks due to the formation of Co-Pt compounds (presumably at the interfaces between Co and Pt) were identified. The coercivity of samples changed from about 400 Oe for films deposited at low Ar sputtering pressure (2.5 mTorr) to as high as 2300 Oe for films deposited at high Ar pressure (15 mTorr). Ellipsometry and atomic force microscopy were used to study surface roughness and microstructure of samples prepared at different sputtering pressures.

I. INTRODUCTION

Co/Pt multilayer (ML) films made by different techniques, such as molecular beam epitaxy, electron-beam evaporation, and sputtering, have been intensively studied in the past few years because of their important potential application as optical data storage media.¹⁻⁴ It is well known that the magneto-optical, and magnetic properties (such as Kerr effect and coercivity) of Co/Pt ML films depend strongly on the deposition method.^{4,5} Experiments have shown that electron-beam evaporated Co/Pt ML films have higher coercivity than sputtered Co/Pt ML films. However, the Kerr rotation of evaporated Co/Pt ML films is smaller than that of sputtered films. Also, Co/Pt ML films deposited using different sputtering gases, including Ar, Xe, and Kr, were studied, and a systematic change of coercivity on sputtering gas species was found.⁵ But origins of the change of the coercivity in these films remained unclear because no differences in microstructure such as grain size were found in the films.

It was also unclear whether the crystalline alloy $\text{Co}_x\text{Pt}_{1-x}$ forms or not at interfaces between the Co and Pt layers, or if compounds form. Due to the fact that Co and Pt are miscible over the complete composition range⁶ and that sputter deposition does not result in atomically sharp interfaces, it is likely that the crystalline alloys $\text{Co}_x\text{Pt}_{1-x}$ form at interfaces between Co and Pt. Compound formation was indicated in our previous work,⁴ as well as by others.⁷ However, there is still some controversy concerning this formation. A major purpose of the paper is to present clear evidence in favor of compound formation.

II. EXPERIMENTS

Co/Pt ML films were sputter deposited on Si wafers, as well as on glass at different Ar gas pressures, at room temperature. The nominal structure of the samples is $\text{Co}(3 \text{ \AA})/\text{Pt}(15 \text{ \AA}) \times 17$ with a 300-Å Pt underlayer, as listed in

Table I. Both low, and high-angle x-ray diffraction spectra were taken to verify the layered structure and to investigate the interface crystallinity with a Rigaku x-ray diffractometer, using $\text{Cu } K_\alpha$ radiation ($\lambda = 1.542 \text{ \AA}$). Magneto-optical polar Kerr rotation θ_k and coercivity H_{cl} were measured on a magneto-optical polar Kerr effect spectrometer described elsewhere.⁹ The coercivity H_{cl} of the samples was determined from Kerr rotation hysteresis loops measured at a wavelength of 5000 Å, with applied magnetic fields over the range from 0 to ± 8 kG magnetic, fields oriented perpendicular to the sample surface. The microscopic surface roughness of the samples was examined by ellipsometry and by atomic force microscopy (AFM). The experimental results for films deposited on glass substrates are almost identical to the results from films on Si wafers. The results shown in the next section are from films on Si wafers except for the x-ray diffractograms which are of films on glass, since then there is no peak showing up from the substrate.

III. RESULTS AND DISCUSSION

A typical low angle x-ray diffractogram from sample 1 deposited in Ar gas of 2.5 mTorr in Fig. 1 demonstrates the integrity and periodicity of the layered structure of samples. The two diffractograms (a) and (b) in Fig. 2 are from samples 2(5.0 mTorr) and 3(8.5 mTorr), respectively. The peaks around $2\theta \cong 40^\circ$ in the x-ray diffractograms reveal that the films were at least partially crystalline, and the Pt and $\text{CoPt}_{3+\delta}$ (δ is a small number) phases are identified as shown in Figs. 2(a) and 2(b). The diffractograms of samples 1(2.5 mTorr) and 2(5.0 mTorr) are similar, as are the diffractograms of samples 3(8.5 mTorr) and 4(15.0 mTorr). Other parameters associated with these samples can be found in Table I.

The peak at $2\theta \cong 40^\circ$ in Fig. 2(a) is mainly due to the (111) planes of Pt layers and the Pt underlayer, which are parallel to the film surface. The second-order Pt(111) peak

TABLE I. Sample structures and other parameters.

| Sample No. | 1 | 2 | 3 | 4 |
|-----------------------------------|--|------|------|------|
| Co/Pt | 3 Å Co/15 Å Pt for all samples | | | |
| No. of bilayers | 17 bilayers on 300 Å Pt buffer for all samples | | | |
| λ_L (Å) | 17.8 | 20.3 | 20.7 | 17.7 |
| Substrate | Si wafer | | | |
| Ar P_s ($\times 10^{-3}$ Torr) | 2.5 | 5.0 | 8.5 | 15 |
| H_{cl} (Oe) | 416 | 833 | 1875 | 2291 |

Note: Ar P_s = Ar sputtering pressure. H_{cl} = coercivity. λ_L = bilayer thickness.

labeled Pt(222) is also visible at about $2\theta \cong 86.4^\circ$ as shown in Fig. 2(a). The existence of the satellite peak⁹ at about $2\theta = 35.5^\circ$ shows that the film possesses superlattice structure character. From the difference of angular positions of the satellite peak and of the main peak labeled Pt(111) at about 40° , the bilayer thickness in the multilayers can be calculated.

When the sputtering pressure is increased, the peak at $2\theta \cong 40^\circ$ splits into the two peaks shown in Fig. 2(b), which have been identified as being from Pt(111) and $\text{CoPt}_{3+\delta}(111)$, respectively. From the angular position of the satellite peak at $2\theta = 36.1^\circ$ and bilayer thickness of the film, one can estimate that the position of the main superlattice peak should be at about $2\theta = 40.5^\circ$, which is the position of the $\text{CoPt}_{3+\delta}(111)$ peak. Therefore, the crystalline structure in the layered region has changed. It is rather difficult to positively determine the phase structure in nanostructured multilayers. We address this question by looking for higher-order x-ray diffraction peaks.

It is clear that there exist two weak peaks at $2\theta = 85.7^\circ$ and 87.4° as shown in Fig. 2(b): These two peaks have been identified as the second-order peaks of Pt(111) and $\text{CoPt}_{3+\delta}(111)$ labeled Pt(222) and $\text{CoPt}_{3+\delta}(222)$, respectively. The existence of the second-order peak at $2\theta = 87.4^\circ$ is strong evidence that there exists another crystalline structure with a d spacing of 2.22 Å which happens to be the d spacing of $\text{CoPt}_3(111)$ planes.

The detailed mechanism of how the $\text{CoPt}_{3+\delta}$ compound forms is not clear. One explanation comes from examining the chemical interactions between Co and Pt.

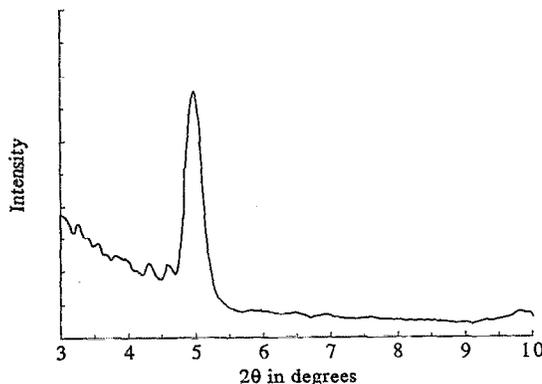


FIG. 1. Low-angle x-ray diffractogram of sample 1. The peak indicates the bilayer thickness 17.8 Å.

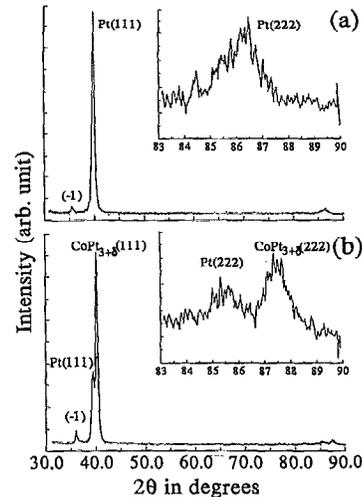


FIG. 2. High-angle x-ray diffractograms of samples 2(a) and 3(b).

According to the Miedema model,¹⁰ the heat of mixing for Co and Pt is negative (-11 kJ/g at. for CoPt). This thermodynamic condition may result in sufficient kinetic mobility for Co and Pt atoms. In studies of sputter deposited Ti/Ni¹¹ and Ni/Zr¹² multilayers, it was found that the stability against interdiffusion was higher in films deposited at low deposition pressure. Also, from our previous study⁴ of evaporated and sputtered Co/Pt ML films, there is another peak corresponding to a d spacing of 1.915 Å. This peak was present in diffractograms of three of our samples. The peak was identified as being from $\text{CoPt}_3(200)$.

Figure 3 shows the systematic changes of coercivity H_{cl} and Kerr rotation θ_k of the films with different sputtering gas pressures. A high coercivity (1800 Oe) is obtained when the sputtering gas pressure is more than 8 mTorr (in the case of samples 3 and 4). All samples show 100% remanence in the perpendicular direction. The Kerr rotation decreases with increasing Ar pressure, as shown in Fig. 3. The decrease in Kerr rotation may be related to the interfacial structure in Co/Pt ML films. But this point has not been fully studied.

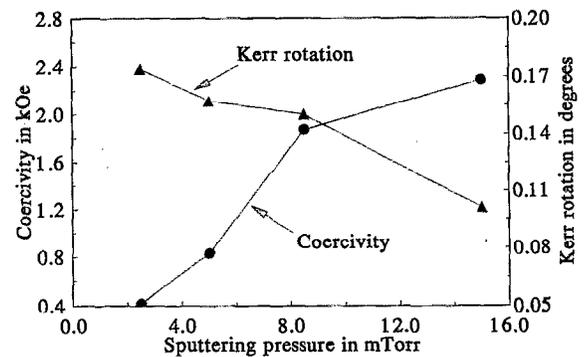


FIG. 3. Coercivity H_{cl} and Kerr rotation θ_k of samples 1, 2, 3, and 4. H_{cl} in kOe and θ_k in degrees were determined from Kerr rotation loops measured at $\lambda = 5000$ Å in magnetic field 0 to ± 8 kG.

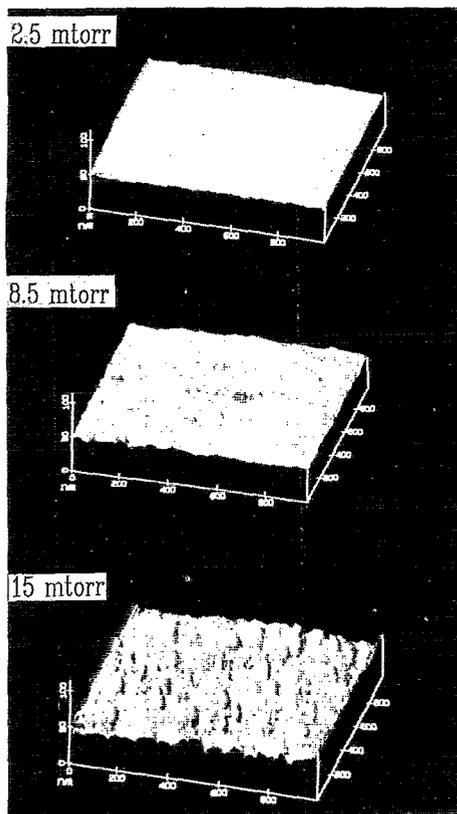


FIG. 4. Surface-views of AFM images of samples 1, 3, and 4. Units are nanometers.

The AFM images in Fig. 4 show the surface microstructures of samples 1(a), 3(b), and 4(c). Figure 4 reveals that at low sputtering pressures [e.g., 2.5 mTorr, Fig. 4(a)], the surface is very smooth and the grain size is very small. The average grain size for the low sputtering pressure is about 200 Å. When the sputtering pressure is increased, the films are not as dense, and the average grain size is considerably increased (to more than 1000 Å), as shown in Fig. 4(c). The surface roughness of the samples determined ellipsometrically is shown in Fig 5. The ellipsometrically determined roughness layer thickness on the surface is consistent with AFM images. The surface roughness of thin films (a few hundred Angstroms thick) is qualitatively related to the grain size. The smoother the surface, the smaller the grain size.

All the experimental results shown above suggest that the sputtering pressure or the bombardment of energetic ions reflected from target while sputtering change the microstructure of films. Sufficient ion bombardment of the films will smooth the surfaces, reduce the size of grains, and make the films dense, as indicated by Hashimoto *et al.*¹³ While in the absence of ion bombardment, grains in the films will be large and surfaces rough. Since, in general, the coercivity, strongly depends upon the shape and size of grains in the materials, the increase in coercivity of the samples with increase in sputtering gas pressure can be at least partially attributed to the change in film microstructure.

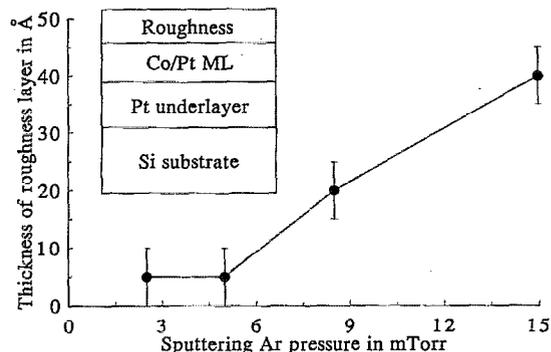


FIG. 5. Ellipsometrically analyzed thickness of surface roughness layer of samples 1, 2, 3, and 4, and the model on which the analysis was based.

ture. It should be pointed out that Co/Pt ML films are highly optically absorptive, and coercivity measurements using the Kerr rotation spectrometer are sensitive only to the first few hundred Angstroms of film. In other cases where films are very thick and the microstructure on the film surfaces are different from the interior of films, the coercivity as determined by the optical method may be different from that determined by the magnetic method which takes an average over the whole film. The coercivities determined by SQUID and by Kerr rotation loops of the samples in this study are about the same, since the film thickness of all the samples is only 300 Å.

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