Magneto-optical properties of obliquely evaporated Ni films

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Longitudinal magneto-optical Kerr effect (MOKE) configuration and scanning electron microscopy (SEM) were used to investigate the effects of deposition angle, α on magnetic properties and morphology of obliquely evaporated Ni films. The results show that the angle of deposition has a critical effect on the magnetic anisotropy of the films. These effects are due to the microstructure of the films, which is controlled by the angle of deposition with respect to the sample normal. The results show the presence of shape anisotropy governing the demagnetization of the magnetic fields. The anisotropy, coercivity and squareness of the hysteresis loops increased with an increase in α of the vapour flux. These properties and surface roughness became marked for off-normal deposition angles larger than 50°. At low α s, the easy axis of magnetization lay perpendicular to the incidence plane. At large α s the easy axis changed parallel to the incidence plane. The results may be quantitatively understood from the presence of an inclined columnar microstructure with shape anisotropy governing the demagnetization of the magnetic fields. C *2003 Kluwer Academic Publishers*

1. Introduction

As the demand for higher storage capacity increases because of the expanding computer industry, there has been growing interest and activities in developing thin film recording media. An important factor driving the interest in thin film magnetism is the capability to deliberately modify the structure of a material in order to influence the magnetic properties. Thin film media offer advantages of higher saturation moment, high squareness and high coercivity [1]. The successful application of ferromagnetic thin films in technological devices requires that the magnetic properties of the films be accurately controlled. The magnetic behaviour of such films can be tailored via regulation of the deposition condition. An example of such control of deposition conditions is the oblique deposition of a ferromagnetic material. Such growth conditions induce canted exchange-decoupled grains, which dominate the magnetic properties of the film due to the shape anisotropy of the individual grains.

Crystallites in obliquely deposited films grow along a direction inclined from the film normal towards the vapour beam direction [2–5] and align along the direction perpendicular to the incidence plane [6]. Fig. 1 gives a schematic representation of films with inclined columnar features. The growth and alignment induce magnetic anisotropy in the films through the anisotropy of demagnetizing field. In an ideal uniaxial anisotropy film, the coercive force in the hard direction is zero. For such a film the anisotropy field, H_k , is the minimum applied field required to magnetize the film to saturation in the hard direction. For films in which the hard direction coercive force is not zero, the anisotropy field is expressed by an equation of the form [7]

$$H_k \approx M_s \left(\frac{\partial H}{\partial M}\right)_{M=0} \tag{1}$$

where H and M_s the applied field and saturation magnetization respectively. This assumes linear M verses H dependence for fields below saturation. Uniaxial anisotropy in thin films is characterized by a directional dependence of energy of the form [8]

$$E_k = K_u \sin^2 \varphi_k \tag{2}$$

where K_u is the uniaxial anisotropy constant and E_k is the anisotropy energy for a magnetization vector making an angle φ_k with the anisotropy axis. The anisotropy field H_k (stress) due to stress is expressed in the form [9]

$$H_k(\text{stress}) = \frac{3\lambda_s \Delta \sigma}{M_s} \tag{3}$$

where $\Delta \sigma$ is the difference between the stresses along the directions perpendicular and parallel to the incidence plane and λ_s is the magnetostriction constant. The relationship between K_u , H_k and M_s is of the form [10]

$$K_{u} = \frac{1}{2}M_{s}H_{k} + 2\pi M_{s}^{2}$$
(4)

Magnetic anisotropy is one of the effective tools used to investigate the formation mechanism of the columnar



Figure 1 Definition of angles of deposition α and of inclination β of the columns, respectively.

grain structure in obliquely evaporated films, because the columnar grain structure induces the magnetic anisotropy through the shape anisotropy [11]. Magnetic anisotropies of evaporated tape media have been studied, and their origins have been attributed to crystalline anisotropy, shape anisotropy, and/or magnetostriction [12, 13]. The effects of oblique incidence during sputter deposition on magnetic anisotropy have been reported [14]. Because of the unique columnar microstructure in obliquely deposited films, many interesting and potentially useful phenomena have been observed in these type of films. However the correlation between the magnetic anisotropy and the film microstructure is not yet clear. We investigate the effects of oblique-incidence on magnetic anisotropy in Ni films. The physical origin of the magnetic anisotropy was studied by analyzing the surface and cross-sectional morphologies of the films using a scanning electron microscopy (SEM).

2. Experimental procedure

Films were prepared by evaporating 99.5% pure nickel wire on 1-mm-thick glass slides at a pressure less than 10^{-6} mbar using a conventional oil diffusion pumped system. Corning 7059 glass slides were cleaned and attached to a substrate holder placed 15 cm directly above the evaporation source. The substrate holder was rotatable so that the angle α between the direction of the incident evaporated flux and the substrate normal could be set at any value between 0 and 90°. Deposition rate, r was recorded in situ on a quartz-crystal monitor. Film thickness, d was obtained ex situ by a Tencor Alphastep 200 surface profilometer. The samples had d in the range of 55 to 250 nm and were deposited at rates between 0.8 and 1.2 nm s⁻¹. The deposition rate was monitored in situ by a quartz crystal microbalance. Films made by this technique have columnar features whose long axes are oriented towards the direction of the incident flux.

A longitudinal magneto-optical Kerr effect (MOKE) configuration was used to obtain hysteresis loops for the samples. The MOKE magnetometry provides a novel technique for detecting two in-plane magnetization components in ferromagnetic materials. This

method utilizes the magneto-optical Kerr effect to sense the magnetization as a function of the applied field. It is able to probe magnetization in small regions of a sample.

Polarization changes due to magneto-optic Kerr effect are very small. To detect these small changes, a lock-in amplifier was used to measure the MOKE signal. A lock-in amplifier uses phase sensitive detection technique to single out a signal at a specific reference frequency. Noise signals at frequencies different from the reference frequency are rejected and do not affect the measurement. A low-noise current preamplifier was used to convert the weak MOKE signal to an output voltage proportional to the input current. A photodiode was used to detect the intensity changes. A computer and LabVIEW program controlled the experimental setup and data acquisition.

The Kerr signals for samples prepared at different deposition angles were measured at room temperature (23°) . Polarized laser light of wavelength 632 nm from a diode laser source with a power output of 5 mW was used in the study. The sample was placed centrally between the poles of an electromagnet. The laser light was incidence at an angle of 60° to the sample normal. The reflected light was passed through an analyzer, which was rotated to obtain the extinction position. To accommodate the entire amplitude of the Kerr signal the analyzer was rotated through 4° on either side of the extinction position. The Kerr signal was detected, amplified and measured for varying field to obtain a hysteresis loop for different samples. Measurements were performed with the field applied in direction parallel and perpendicular to the incident deposition plane. A high resolution Leo 1550 SEM was used to obtain images of the top surface and cross-sectional area of the samples.

3. Results and discussion

To investigate the effects of vapour incidence angle on the magnetic properties, the longitudinal Kerr loops of Ni films were measured. Fig. 2 shows the magnetization for a 55 nm thick Ni films deposited at $\alpha = 0^{\circ}$. Measurements in the parallel and perpendicular directions



Figure 2 Kerr hystersis loop for film prepared at deposition angle $\alpha = 0^{\circ}$ and thickness d = 55 nm.

to the incidence plane gave identical results. This may be due to random orientation of the crystallites. A similar result was reported by Gau [1] for the in-plane coercivity measured both along the incidence plane and transverse plane for evaporated Co-Ni films. The film is isotropic in the film plane. Electron micrographs of films prepared by vapour beam impinging upon the substrate at normal incidence show that the crystallites are randomly oriented.

Fig. 3 show hysteresis loops for films prepared at deposition angles of 20°, 40°, 60° and 80° and having thicknesses *d* of 55, 85, 55 and 90 nm respectively. An increase in α yields higher coercivity and improved squareness of the hysteresis loops. There are differences between loops recorded parallel and perpendicular to the incidence plane. Judging from the difference in magnetization M_r at remanence, the easy direction of magnetization in the film plane is perpendicular to the incidence plane at low deposition angles, as seen from Fig. 3a. However, at large deposition angles the direction changes to lie parallel to the incidence plane. The transition seems to take place when α is between 40° and 60°.



Figure 3 Hysteresis loops for film evaporated at α being 20°, 40°, 60° and 80° onto glass substrates and measured parallel and perpendicular to the incidence plane. The films had thickness *d* as indicated in the diagrams.

Fig. 3c shows hysteresis loops for films that had thickness of 55 nm and deposited at $\alpha = 60^{\circ}$. The squareness of the hysteresis loop for measurement perpendicular to incidence plane of deposition is not pronounced. For comparison the hysteresis loops for films deposited at $\alpha = 80^{\circ}$ and having thicknesses *d* of 90 nm are presented in Fig. 3d.

The sharp rise in the coercivity for films deposited at oblique angles larger than 40° is attributed to the induced out-of-plane shape anisotropy associated with the columnar structure of the films. The coercivity, anisotropy field and the squareness increased with the increase in α .

Pure longitudinal Kerr effects can be observed when magnetization is in the plane of the sample. The loops display an in-plane magnetic easy-axis direction parallel to the incidence plane. For the magnetic field applied along the easy axis (along the columns), the hysteresis loops exhibit a square shape, while, for a magnetic field applied perpendicular to the easy axis, a quasi-linear field dependence of the magnetization is observed before reaching the saturation magnetization. Films deposited at different angles to the substrate normal have different microstructures, and therefore different magnetic anisotropy. Best squareness was obtained for films deposited at angles larger than 50° to the substrate normal. The magnetization and squareness of the loop measured along the incidence plane was much larger than in the transverse direction.

The variation in magnetic properties with increased angle of deposition can be explained by the residual anisotropy being overcome by the growth-induced anisotropy. The shape anisotropy of the Ni grains becomes apparent as the grains become more loosely packed. This poor packing also causes the total magnetization of the samples to be reduced having the effect of reducing the thin film shape anisotropy (i.e. $4\pi M_s$) of the samples.



Figure 4 SEM surface micrograps of 70 nm Ni films thickness: (a) $\alpha = 80^{\circ}$ and (b) $\alpha = 85^{\circ}$.





The observed anisotropy is believed to be due to a self-shadowing geometrical effect arising from the chaining of small nuclei to the incidence plane [15]. The sharp rise in the coercivity for films deposited at oblique angles larger than 50° is attributed to the induced out-of-plane shape anisotropy associated with the columnar structure of the films [16]. The coercivity, anisotropy field and the squareness increased with the increase in α . Films deposited such that the incident vapour species reach the substrate under a large off-normal have preferred direction of magnetization.

The surface topography and the cross-sectional area of the samples were observed using SEM. Fig. 4 show images of the surface for films deposited at 80° and 85° and had thickness of 70 nm. There is strong crystalline orientation in the film plane. The cross-sectional micrograph of the fracture in the deposition plane shows well-defined columnar microstructure with columns, which are uniform in width from the substrate to top of the films. The column boundaries are separated by voids. Fig. 5 shows a cross-sectional morphology of a film evaporated at incidence angle of 80° and had thickness of approximately 250 nm. The film comprises four layers: the first layer was deposited on the glass substrate and the second, third and fourth on top of the film layer. This was done to be able to obtain a thick film since the tungsten filament would break during evaporation of thick films. The vacuum was broken after deposition of each layer was made, new source material was placed in the tungsten filament, the chamber was pumped down to pressure less than 10^{-6} mbar, and a new deposition made. It took approximately three hours between deposition of any two consecutive layers. All layers were evaporated at the same angle. The columnar inclinations of the first layer on glass are larger than the subsequent layers, which illustrates the effect of the nature of substrates on the growth of the grains.

The alignment direction of the columns was nearly normal to the substrate for the films deposited at angles of incidence up to 50° . However, the films deposited at incident angles larger than 50° , revealed that columns inclined toward incident vapour direction with respect to the substrate normal. The porous cross-sectional morphologies are consistent with the surface features consisting of elongated grains in perpendicular directions to the incidence plane. For larger deposition angles, the columnar microstructure develops to a marked extent because the islands grow individually, elongating along the vapour beam direction in preference to their mutual coalescence.

4. Conclusion

Ni films prepared by oblique-incidence deposition have in-plane magnetic anisotropy. The anisotropy increases with the increase in deposition angle and became pronounced for α larger than 50°. This is due to the presence of an inclined columnar microstructure with shape anisotropy of the inherent structural units affecting the demagnetization of the magnetic fields, thereby invoking field dependent magnetization. In the film plane, the direction of easy axis of magnetization lies perpendicular to the incidence plane at low deposition angles. But at high deposition angles, the easy axis of magnetization changes direction parallel to the incidence plane. Our results highlight the extreme sensitivity of magnetic anisotropy in obliquely deposited films to the microstructural morphology.

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