Optical constant of CoFeB thin film measured with interference enhancement method

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Optical constants (n and k) of $Co_{20}Fe_{60}B_{20}$ (CoFeB) thin films (2~40nm) are measured by using interference enhancement method with spectroscopic ellipsometry in the wavelength range of 270~1600nm. The effects of film thickness, protection layer and annealing process on the optical constant of CoFeB film are investigated. In the range of 40~10nm, both n and k decrease with the decrease of thickness. The protection layer of SiO_2 on the CoFeB film is helpful for the precise measurement of the n and k values. The annealing process has less effect on the optical constant of the film with the protection layer as compared to the one without it.

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1.Introduction

Recently a novel magnetooptical spatial light modulator (MOSLM) based on spin transfer torque (STT) technology was proposed to address the limited viewing angle and bandwidth issues of holographic 3D display system [1]. Its submicron scale pixel size enables a viewing angle larger than 60 degree; The STT technology used makes the pixel to be switched in ns scale (10 ~15ns), which subsequently increases the bandwidth of the display system significantly. The pixel of this type of SLM is composed of thin film stack, the thickness and optical constant of each individual film will affect the final magnetooptical (MO) response of the whole film stack. With the film thickness approaching to several tens of or several nm, the optical constant becomes different from its bulk counterpart [2]. In order to accurately predict the MO response of the film stack, precise measurement of the optical constant in thin film status is important.

As a main free layer material for STT-based devices, such as STT magnetic random access memory (STT-MRAM), CoFeB with various compositions has been studied extensively [3,4]. But its optical properties, especially for thin film are still less investigated. Kalashnikova et al. reported the optical properties of CoFeB/SiO_2 granular nanostructure [5]. As the CoFeB material was embedded in a SiO_2 matrix, the optical properties reported may not represent the optical properties of CoFeB thin film.

Spectroscopic ellipsometry (SE) measures the polarization state change ρ in the light reflected or transmitted from a sample. The polarization state change is described by an amplitude ratio $\tan(\psi)$ and phase difference Δ between the p- and s- components of the light.

$$\rho = \tan(\psi)e^{i\Delta} = \frac{R_p}{R_s} \tag{1}$$

where R_p and R_s are the complex Fresnel coefficients for p- and s-light respectively. By modelling and fitting the SE data, one can obtain the optical constant and thickness of the film. This technology has been widely applied to measure the thickness and corresponding optical constant of dielectric and semiconductor thin film [6,7]. For an absorbing metal material, there is a strong correlation between the film thickness and optical constant which makes it difficult to measure both the thickness and optical constant simultaneously [8]. In order to address this issue, several methodologies based on SE have been developed [2,8], which include methods such as opaque coating, transparent wavelength region, simultaneous SE and intensity, multiple sample analysis, multiple ambient method, optical constant parameterization and interference enhancement (IE). IE is believed to work best for strong absorbing material among methods mentioned above. It is first reported by McGahan for the characterization of carbon films [8].

When the light beam is incident on a thick transparent layer (>100nm) covered silicon wafer at different angles, the SE data obtained features in different interference oscillation patterns. If an absorbing layer is deposited on the top of the transparent layer, a damped interference oscillation pattern will be obtained. For different incident angles, the modulation of the interference oscillation pattern by the absorbing layer will be different. This will help to reduce the correlation between the thickness and optical constant of the absorbing film and make simultaneous measurement of these two parameters unique. This is the basic working principle of IE. One requirement for this method is that the film should be thin enough to allow certain fraction of the light to be reflected back to generate the interference oscillation pattern.

In this paper, we use variable angle incidence spectroscopic ellipsometry (VASE) to determine the optical constants of CoFeB thin films with IE method. The effects of the film thickness, protection layer and annealing process on the optical constants are investigated.

2. Experiments and procedures

The $Co_{20}Fe_{60}B_{20}$ (CoFeB) thin films with different thicknesses are deposited on a 1"x1" Si substrate covered by a 1000nm-thick thermal SiO_2 with an AJA ATC 2200-V sputtering system. No intentional heating is applied to the substrate during the film deposition process. Two sets of samples of different thickness are deposited. For one set of samples, a 2nm SiO_2 film is deposited on top of the CoFeB film as a protection layer to prevent it from oxidation in the air. For the other set of samples, the CoFeB films deposited are unprotected. The depositions are performed at 1mTorr for CoFeB and 3mTorr for SiO_2 respectively. The deposition rates for CoFeB and SiO_2 are 0.01124nm/s and 0.02324nm/s respectively, which are calibrated with a thicker sample under the same deposition conditions. All the samples are annealed at 300 °C for 2 hours under a pressure of $5x10^{-6}$Torr in a customized vacuum annealing furnace.

The optical properties of all the substrates, as-deposited and annealed films are measured with a commercial J. A. Woollam V-VASE variable angle of incident spectroscopic ellipometry, which consists of a continuous rotating analyzer with an adjustable Berek waveplate [9]. The wavelength range is from 270nm to 1600nm which is slightly narrower than the equipment's available wavelength range of 250nm~1700nm. The SE data are obtained for three different incident angles (65°, 70°, 75°) and then processed with the equipment's data acquisition and analysis software WVASE.

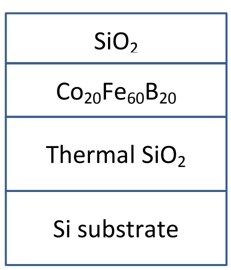


Figure 1. Layered model for analysis of the SE data

To minimize the effect imposed by the substrate on the final result of absorbing film, SE data of bare substrates are measured before the CoFeB film deposition on the substrate. The thickness of the thermal oxidized SiO_2 is fitted and kept as a constant in the future analysis. After the CoFeB and SiO_2 films were deposited on the substrate, new set of SE data are measured. A film stack model as shown in Fig.1 is built to simulate the response of film to the incident light. For the film with 2nm SiO_2 protection layer, we first fix the top SiO_2 thickness at 2nm and fit both the n and k values and thickness of the CoFeB film. After that the SiO_2 thickness is allowed to vary, this is to compensate the imperfection of the model in which the interface is assumed to be perfectly plat and there is no transition layer at the interface region. By doing so, the mean square error (MSE) of the fitting will be reduced. It makes final n and k curves smoother. For films without SiO_2 protection layer, we fit both the optical constant and thickness of the CoFeB film simultaneously. A virtual oxide layer is normally coupled on top of the CoFeB film to further reduce the MSE.

The Kramers-Kronig consistence is ensured by using a dispersion equation to model the optical constant of CoFeB film. The dispersion equation is composed of one Drude, two Lorentz and one Gaussian oscillator terms. The initial value for the fitting is set as the optical constant for Fe in the software's material library.

The atomic force microscope (AFM) measurement of the morphology and roughness of CoFeB films are carried out with a Veeco Dimension 3100 scanning probe microscopy.

3. Results and Discussions

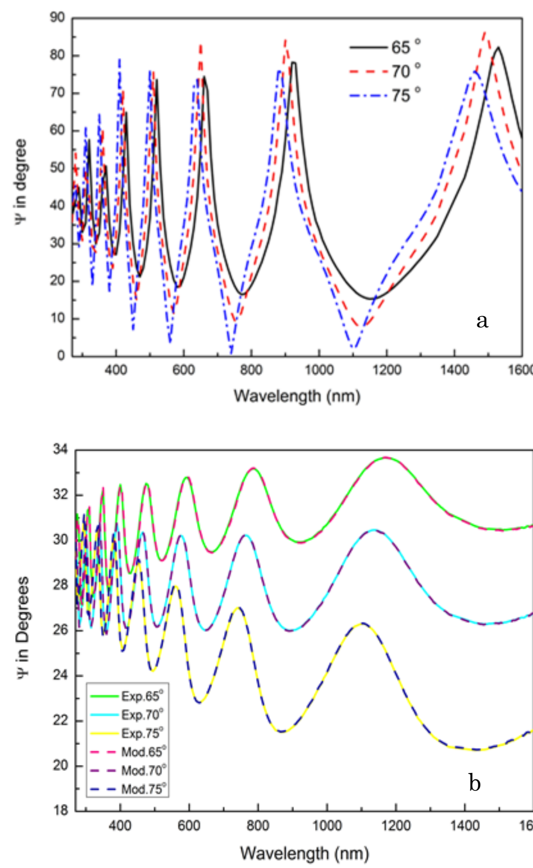
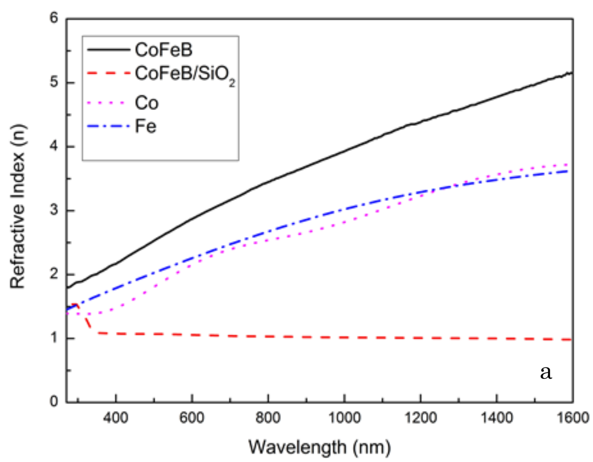


Figure 2. SE data at three incident angles (65°, 70° and 75°) for the thermal oxidized substrate (a) without and (b) with the CoFeB film on top of it.

The typical raw SE data for the thermal oxidized silicon wafer and with CoFeB thin film on top of it are shown in Fig.2. For bare wafer, as shown in Fig.2a, the ψ value oscillating amplitudes fall in ~80° for all the three incident angles. When a CoFeB film is deposited on it, both the oscillation amplitude and the peak position are changed compared to that of the bare wafer. For the data shown in Fig.2b, the nominal film thickness of CoFeB is 30nm; the amplitude of the oscillation is modulated to several degrees. And for different incident angles, the ranges of oscillation are also different. With the increase of incident angle increase from 65° to 75°, the oscillation range of ψ changed from 27.1°~33.6° to 20.7°~31.1° respectively. The difference of the SE data obtained for different angles allows the thickness and optical constant of the CoFeB film to be measured simultaneously and uniquely. The solid lines in Fig.2b are experimentally measured SE data, and the dashed lines over the solid ones represent the model fitted curves. One can see that the model fitted curves match very well with the experimental data.

3.1 Optical constant of optical thick CoFeB films

The metal film with a thickness ≥ 80nm is considered as optical thick film [10], which means its optical properties are close to that of its bulk material. For reflection mode, the thickness can be reduced to ≥40nm due to the light passing through the film twice.



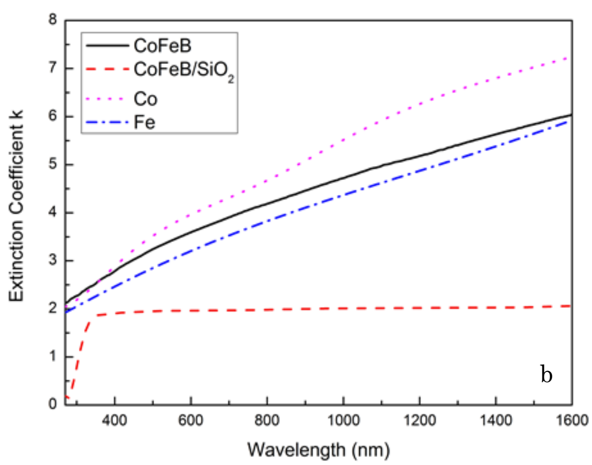


Figure 3. Comparison of (a) refractive index n and (b) extinction coefficient k among CoFeB, Fe[11], Co[11] and CoFeB/SiO_2 nanocomposite[5].

In order to compare the optical constant of CoFeB film with other bulk materials like Fe, Co and a reported CoFeB/SiO_2 nanocomposite, the optical constant of a 40nm-thick CoFeB film is measured. Fig. 3a and Fig. 3b show the comparison of n and k values between CoFeB film deposited on the thermal oxidized silicon wafer and CoFeB/SiO_2 nanocomposite [5], Fe and Co reported in the literature [11]. The general trend of the spectral responses of real and imaginary parts of the dielectric functions ε_1 and ε_2 of Fe and Co follow the typical trend which can be described by Drude model. There are oscillations on the curve for Co and for Fe, the curve is much smoother. While for CoFeB/SiO_2 nanocomposite, the refractive index dropped abruptly from 1.58 to 1.08 in the range from 270nm to 350nm and kept almost constant over the other wavelength range investigated; while the extinction coefficient of it increased from 0.20 to 1.95 in the range from 270nm to 530nm, and keep constant for remaining wavelength range. From Fig. 3a and Fig.3b, one can see that the trend of the n and k data obtained for the 40nm-thick CoFeB film are more similar as that of Fe and Co rather than that of CoFeB/SiO_2 nanocomposite. Its value for refractive index, are higher than that of both Fe and Co, while for extinction coefficient, it falls in between that of Fe and Co in most of the wavelength range.

The difference of the spectra for our CoFeB film and CoFeB/SiO_2 nanocomposite may be due to the difference in material structure. For nanocomposite, the CoFeB material is embedded in the SiO_2 matrix, our film is continuous film. The light-material interaction should be much different from each other. Therefore, the optical property of our CoFeB film is more similar to that of Fe and Co, rather than the CoFeB/SiO_2 nanocomposite.

3.2 Optical constants of CoFeB films with different thicknesses

The optical constants n and k are measured for a thickness range from 2nm to 40nm. The n and k values for different thickness are plotted in Fig. 4. Fig. 4a, 4b are for the film with thickness from 10 to 40nm and Fig. 4c, 4d are for the range of 2~5nm. Further detailed for three typical wavelengths in visible range (450nm, 535nm and 650nm) are provided in Fig. 6. From Fig. 4a and 4b, one can see that all the curves follow the same wavelength dependence trend as that of 40nm-thick CoFeB and the values decreased with the decrease of thickness in the range from 40nm to 10nm for both n and k. And the amplitude of change increases with the increase of wavelength. As shown in Fig.4c and 4d, when the thickness is reduced to 5nm, although the n value is still increasing with the increase of wavelength, its wavelength dependence trend is slightly different from that in the range of 40~10nm. And its value is even higher than that of 10nm-thick CoFeB film. By further reducing the film thickness to 2nm, both the shape and value in the spectrum are different from the spectra for CoFeB films with thickness >10nm. These abnormal phenomena in 5nm and 2nm thick films may indicate that the CoFeB films are transiting from optical isotropic range to optical anisotropic range with the decrease of the film thickness [7, 12]. In the anisotropic range, the traditional model we used to model the optical response will not be valid.

The morphologies and roughness of the CoFeB films are measured with AFM. With the film thickness decreases from 40nm to 2nm, the roughness Ra decreases from 0.295nm to 0.165nm. From the morphology for 2nm and 40nm as shown in Fig. 5, the morphology of 2nm-thick film is uniform, the grain size is smaller and there are fewer holes compared with that of 40nm thicker film. But as the film thickness is only 2nm, the ratio of the rough surface area to the film volume is much larger compared with that of thicker film, therefore its effect on the thinner film optical constant is larger.

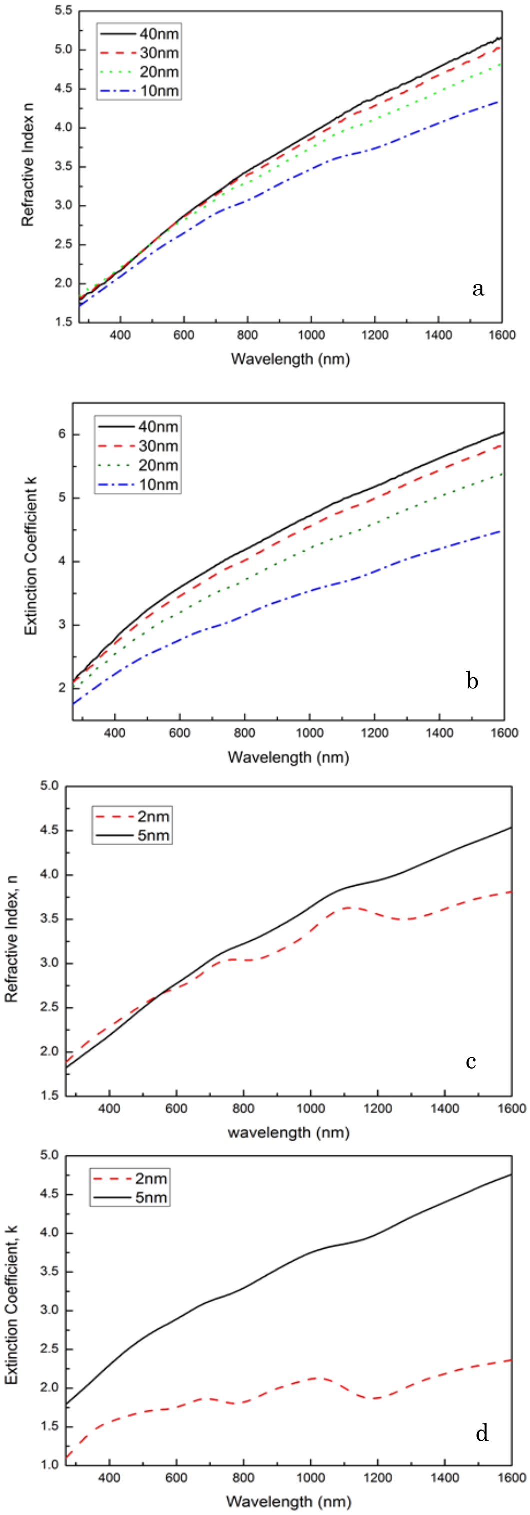


Figure 4. Optical constants of CoFeB films with different thickness in the range of 2~40nm: (a),(c) for refractive index n and (b), (d) extinction coefficient k

With the film thickness reduction, the electron mean free path is also reduced, this will affect the optical constant too [13]. All of these factors will contribute to the optical property changes.

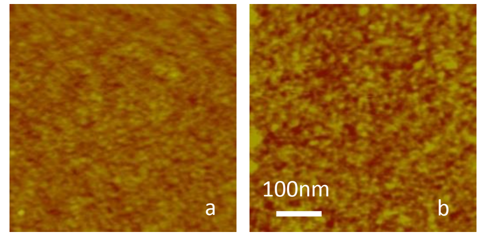


Figure 5. Morphologies of 2nm and 30nm-thick CoFeB films with 2nm SiO_2 protection layer on top of it.

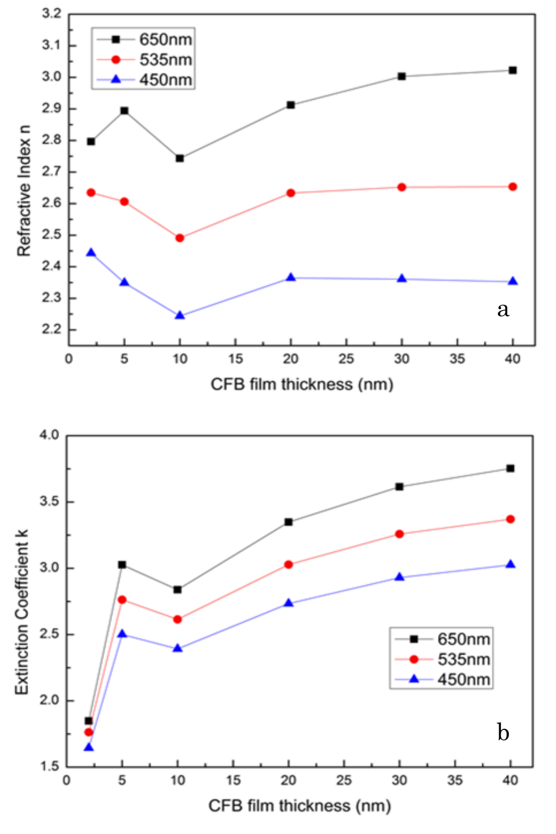


Figure 6. Optical constants (a) n and (b) k of the CoFeB film against thickness at different wavelength (450nm, 535nm and 650nm)

3.3 Effects of protection layer SiO_2 and annealing process

It is well known that the exposure of a fresh deposited metal surface to the air will cause the oxidation of the metal film surface. One method to prevent the surface from oxidation is to protect the film with a dielectric film such as SiO_2. In order to investigate the effect of the protection layer on the n and k values computed, we deposited two sets of samples, one is with 2nm SiO_2 film on top of the CoFeB film, and the other with CoFeB film unprotected and exposed to the environment at room temperature. In order to minimize the effect of the oxidation, the optical properties were measured with ellipsometer immediately after the deposition.

To study the effect of annealing process on the optical constants, the samples were annealed at 300°C for 2 hour in a vacuum furnace.

Fig. 7 shows the optical constants of 40nm-thick CoFeB with and without SiO_2 protection layer as well as with and without annealing process at 300°C for 2 hour.

From Fig. 7a, one can see that the CoFeB films with and without protection layer has a significant difference in refractive index in different wavelength range. For the as-deposited film (represented with ad in the graph), the n value of the film with protection layer is lower than that without it in the range from 270nm to around 850nm. In the range from 850nm to 1600nm, the film with protection layer has higher n value. For the film with a protection layer, both the shape and the value have minor changes before and after annealing over the whole measurement range. But for the film without a protection layer, the change in both the shape and value caused by annealing are much larger.

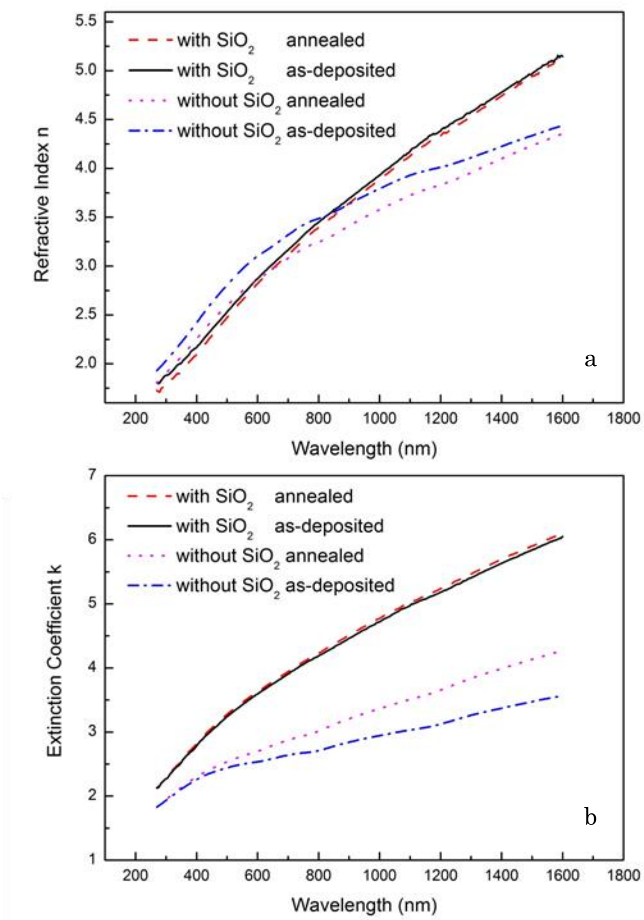
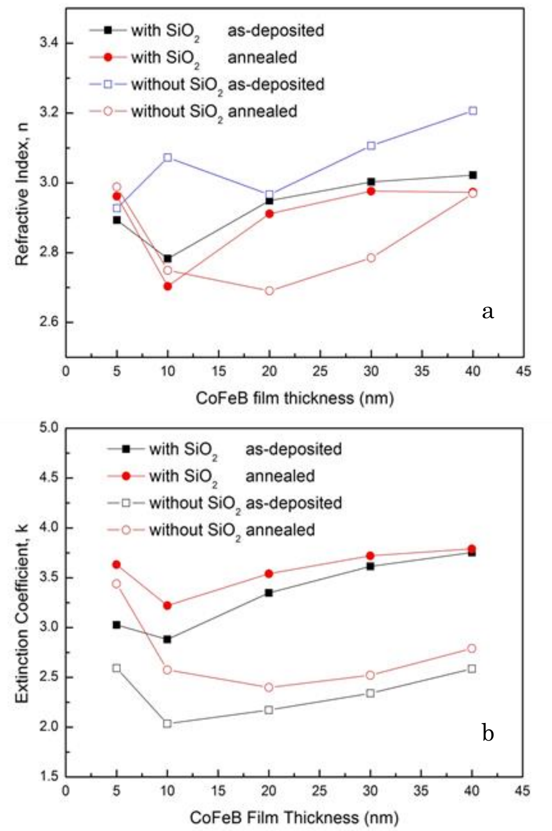


Figure 7. Optical constants (a) n and (b) k before and after the annealing process for 40nm-thick CoFeB with and without 2nm SiO_2 protection layer.

In the Fig. 7b, the extinction coefficient value for the film with protection layer is higher than that without it over the whole measurement range. As demonstrated in the refractive index curve (Fig. 7a), the annealing process has greater effect on the film without a protection layer.



Fgure. 8. Optical constants (a) n and (b) k at 650nm before and after the annealing process for CoFeB with and without 2nm SiO_2 protection layer.

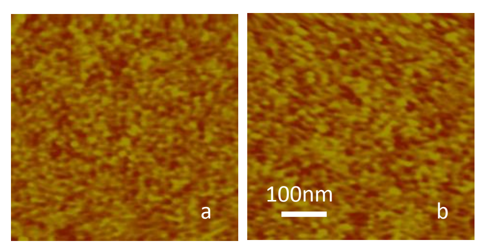


Figure. 9. AFM image of the morphology of annealed 40nm-thick CoFeB films without (a) and without (b) 2nm-thick SiO2 protection layer. (scanning range is 500nm×500nm, the scale size is 5nm

For the films with other thickness, the similar phenomena shown for the 40-thickness CoFeB films are observed. Figure 8 shows the n and k values for different film thickness at 650nm.

For the film with protection layer, the CoFeB is protected from oxidation all the time, and the environment have less effect on its optical constant. Therefore, its optical constant almost remains the same before and after annealing process. The slightly change may be due to the crystalline of the CoFeB film.

While for the film without protection layer, the CoFeB material is directly exposed to the environment. Even we measured the optical constant immediately after the deposition; the film may still be oxidized after it is taken out from the vacuum chamber.

The annealing process may cause further oxidation and crystalline of the CoFeB, which change the optical constant of it further.

The morphologies and roughness of the annealed 40nm-thick CoFeB films with and without protection layer are measured. The morphology of the film without protection layer shows that the grain size is even finer, and the roughness of it is 0.206nm which is lower than the 0.236nm achieved by the film with protection layer. The roughness difference may be due to the different deposition rates of SiO_2 and CoFeB. The deposition rate for CoFeB is slower; therefore its roughness is lower. The roughness definitely has some effect on the optical constant, but the oxidation of the CoFeB may be more prominent.

4. Summary

The optical constants of as-deposited and annealed CoFeB films with different thicknesses are measured with the interference enhancement method by a variable angle of incident spectroscopic ellipsometry (VASE) in the wavelength range from 270nm to 1600nm. The effects of film thickness, thin protection layer and annealing process on the optical constant of the CoFeB films have been studied. A thin protection layer is necessary to make the oxidation of the CoFeB film has less effect on its optical constant. Annealing process has greater effect on the properties of the unprotected CoFeB film than on that of the protected film. The optical constant of the film with thickness less than 10nm may experience the transition from optical isotropic range to optical anisotropic range. Further detailed study is needed to sort out the reasons which affect the optical properties of ultrathin films (<5nm).

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