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Influence of magnetic layer thickness on $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer thin films^{*}

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In the present work, a series of $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer thin films is fabricated using a reactive magnetron sputtering equipment. The thickness of SiO₂ interlayer is fixed at 3 nm, while the thickness values of $Fe_{80}Ni_{20}-O$ magnetic films range from 10 nm to 30 nm. All films present obvious in-plane uniaxial magnetic anisotropy. With increasing the $Fe_{80}Ni_{20}-O$ layer thickness, the saturation magnetization increases slightly and the coercivity becomes larger due to the enlarged grain size, which could weaken the soft magnetic property. The results of high frequency magnetic permeability characterization show that films with thin magnetic layer are more suitable for practical applications. When the thickness of $Fe_{80}Ni_{20}-O$ layer is 10 nm, the multilayer film exhibits the most comprehensive high-frequency magnetic property with a real permeability of 300 in gigahertz range.

Keywords: magnetron sputtering, multilayer films, soft magnetic property, high frequency permeability

PACS: 75.75.-c, 75.70.Cn, 75.30.Gw, 75.50.Gg

1. Introduction

With the rapid development of electromagnetic equipment such as magnetic sensors, planar inductors, and transformers, the development of magnetic films with excellent soft magnetic properties and high-frequency characteristics is urgently needed. For soft magnetic films, they should firstly keep low coercivity (H_c) and high saturation magnetization (M_s) . Meanwhile, large electrical resistivity (ρ) is required to effectively restrict the eddy current loss to a low value. When operating in GHz range, they should possess large permeability (μ) and high resonance frequency (f_r).^[1-4] According to the Snoek's limit,^[5] static permeability (u_s) is expressed as $\mu_{\rm s} = 1 + 4\pi M_{\rm s}/H_{\rm k}$, and the $f_{\rm r}$ is proportional to $(H_{\rm k} \times 4\pi M_{\rm s})^{1/2}$, where $H_{\rm k}$ is the magnetic anisotropy field. So the coordination between the M_s and H_k is important to adjust the μ_s and f_r in order to adapt to different requirements. In earlier years, mainly Fe-based and Co-based single-layered thin films have been studied and some important progress made.^[6–8] However, increasing the thickness values of these films will apparently influence their microstructures and inner stresses. When the film thickness value exceeds hundreds of nanometers, the high-frequency performance will deteriorate due to the fact that the coercive force becomes larger.^[9] Besides, any post fabrication treatment or induced condition may possibly make the films unstable and increase the difficulty in their preparation. In recent years, multilayer film with structure composed of magnetic and nonmagnetic layers, has received much attention. The nonmagnetic interlayer can break the grain growth in the magnetic layer, so a film with large thickness can still possess low coercivity and small grain size. On the other hand, the high resistivity of the nonmagnetic phase and layered structure can reduce eddy current loss in high frequency. Studies show that multilayer thin films are promising materials for high frequency applications.

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Usually Fe-based alloys with high M_s are used for providing magnetic moments, and different insulating phases are widely studied to improve the values of ρ and H_k in order to obtain good magnetic properties when operating in GHz range.^[10–12] In our previous work, we chose Fe₈₀Ni₂₀-based alloy as the magnetic layer and systematically investigated the effects of different insulating phases used as the interlayer on the magnetic properties of multilayer thin films, and achieved high-frequency soft magnetic multilayer thin films with excellent properties.^[13] In this paper, the [Fe₈₀Ni₂₀–O/SiO₂]_n multilayer thin films are prepared with the SiO₂ interlayer thickness fixed at 3 nm. Further study is focused on changing the Fe₈₀Ni₂₀–O magnetic layer thickness value (*t*) to investigate its influence on the magnetic properties and high-frequency characteristics of the films.

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2. Experiments

 $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer thin films were deposited on Si substrates and glass slides by reactive magnetron sputtering at ambient temperature. The background pressure of the chamber was below 5×10^{-4} Pa. Before deposition, the flow rates of Ar gas and O2 gas introduced into the chamber were 20 and 0.2 sccm respectively. During sputtering, the pressure was maintained at 0.4 Pa. The high-purity Fe₈₀Ni₂₀ (purity 99.99%) and Si (purity 99.99%) target were used to prepare the multilayer films. The discharge power was set at 75 W to sputter the Fe₈₀Ni₂₀ target using the equipment DC Advanced Energy, while the power for sputtering Si target was set at 100 W using the RF power supply. A substrate for depositing the films rotated at a rotation speed of 24 rpm, and the distance between the substrate and the target was 8 cm. In order to obtain well-laminated structure in the film, we set a baffle above each of the targets to perform the alternative sputtering. Each layer thickness was controlled by the opening time of the baffle. The thickness of SiO₂ interlayer was fixed at 3 nm, while the Fe₈₀Ni₂₀-O layer thickness was changed from 10 nm to 30 nm. The total thickness of the [Fe₈₀Ni₂₀- $O/SiO_2]_n$ multilayer thin film was measured by surface profiler (Dektak-III). X-ray diffraction (XRD) with Cu $K_{\alpha 1}$ radiation (40 kV, 30 mA) was used to analyze the crystal structures and phases of the films. The cross-section structures of the films were investigated by transmission electron microscopy (TEM, Tecnai F30). The vibrating sample magnetometer (Lake Shore VSM-7404) was used to measure the magnetic properties of the films at room temperature. The high frequency properties of the films from 100 MHz to 5 GHz were measured by a vector network analyzer.

3. Results and discussion

Figure 1 shows the XRD results of the $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer films with the values of magnetic layer thickness (*t*) ranging from 10 nm to 30 nm. Each sample shows a (110) texture of body-centered cubic (bcc) α -Fe₈₀Ni₂₀ phase. No diffraction peak of SiO₂ phase is found, indicating that SiO₂ exists as amorphous silicon oxide. As *t* increases from 10 nm to 30 nm, the intensity of the peak turns sharper and sharper, which means that the grain size becomes larger with the increase of film thickness *t*. This was caused by the more effective time in which the Fe₈₀Ni₂₀–O grains grew before the SiO₂ interlayer was interrupted.

The typical $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer film with $Fe_{80}Ni_{20}-O$ magnetic layer thickness (t = 10 nm) was used for TEM test, of which the cross-sectional image is shown in Fig. 2. As shown in the figure, the interface between the adjacent layers is clear and the magnetic grain growth is totally interrupted by the insulating SiO₂ interlayer, which reflects a well layered structure in the multilayer film.



Fig. 1. (color online) XRD patterns of the $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer thin films with magnetic layer thickness values varying from t = 10 nm to 30 nm.



Fig. 2. (color online) High-resolution cross-sectional image of the $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer film with magnetic layer thickness t = 10 nm.

The hysteresis loops of these multilayer films with different value of t are shown in Fig. 3. Each of all the films could remain obvious in-plane uniaxial magnetic anisotropy (IPUMA) with the residual magnetization close to the saturated magnetization. The easy axis is parallel to the tangential direction of the substrate and the hard axis is in the vertical direction. The origin of the anisotropy field H_k had been explained elsewhere.^[14–16] According to these points, there are several reasons for introducing the H_k . Firstly, introducing a very low dose of oxygen can apparently affect the orientation of the magnetic moment. Secondly, the oblique incidence with respect to the substrate rotation will induce a preferable orientation of the magnetic atoms. Thirdly, the exchange coupling between adjacent ferromagnetic layers will result in stronger H_k . After comparison, we find that the change in t does not have an obvious effect on the H_k , with the H_c kept changing. As shown in Fig. 4, when t increases from 10 nm to 25 nm, the H_c monotonically increases and reaches its maximum when t = 25 nm. According to random anisotropy model,^[17] H_c shows positive correlation to the D^6 (D denotes

the grain size), and from XRD patterns in Fig. 1 we have already known that the $Fe_{80}Ni_{20}$ –O grain size becomes bigger as *t* increases. While *t* increases from 25 nm to 30 nm, H_c becomes smaller. This is because the grain growth is interrupted by the SiO₂ interlayer, so the grain size is nearly equal to the thickness of magnetic layer. Meanwhile, the ferromagnetic exchange coupling length (L_{ex}) of Fe₈₀Ni₂₀–O is about 26 nm. Therefore, a bigger magnetic layer thickness will result in the larger value of *D* than that of L_{ex} . Then the H_c has a negative correlation to the *D*, and a smaller H_c will be achieved when *t* is larger than 25 nm.



Fig. 3. (color online) In-plane hysteresis loops of the $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer thin films with the values of magnetic layer thickness *t* in a range from 10 nm to 30 nm. The unit 1 Oe = 79.5775 A·m⁻¹.



Fig. 4. (color online) Dependences of coercivities (H_{ce} and H_{ch}) on magnetic layer thickness *t* of the $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer thin films.

Figure 5 shows the dependences of M_s and ρ on t in a range from 10 nm to 30 nm. As t increases, the magnetic volume fraction increases and leads to M_s increasing from 1.01 T to 1.42 T. Meanwhile, more metal phase fractions implies lower resistance when the electrons move. As a consequence, the M_s increases monotonically as t turns thicker while the ρ decreases.

As far as the high frequency permeability is concerned, we find that the real component of the permeability (μ') turns a little larger with *t* increasing from 10 nm to 30 nm while the ferromagnetic resonance frequency (f_r) remains almost unchanged at about 2.5 GHz as shown in Fig. 6. However, for the film with a larger value of *t*, the resonance frequency peak becomes wider. According to the XRD results, the grain size of the film becomes larger as the magnetic layer thickness increases, which will result in strong magnetocrystalline anisotropy and evident dispersion in H_k . Each of the two factors cause an adverse effect on the rotation of magnetic moment when operating in GHz range, which makes the resonance frequency peak wider.



Fig. 5. (color online) Dependences of M_s and ρ on magnetic layer thickness *t* of the [Fe₈₀Ni₂₀–O/SiO₂]_{*n*} multilayer thin films.



Fig. 6. (color online) High-frequency permeability spectra for the $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer thin films with different values of magnetic layer thickness *t*.

According to the above discussion, we find that variation in magnetic layer thickness does not exert an obvious effect on the magnetic layer properties, particularly the resonance frequency. The H_k could not be varied in a board range simply by changing the magnetic layer thickness. On the other hand, the ferromagnetic resonance frequency remains almost unchanged no matter how the magnetic layer thickness is changed. Moreover, film with a thicker magnetic layer gives rise to a worse effect on high frequency property due to its larger magnetocrystalline anisotropy, dispersion in H_k and low resistivity. In our paper, high-frequency magnetic properties are achieved in the film with a real permeability of 300 and a resonance frequency of 2.5 GHz when t = 10 nm.

4. Conclusion

In the present work, a series of $[Fe_{80}Ni_{20}-O/SiO_2]_n$ multilayer films with different magnetic layer thickness values is prepared by alternately magnetron sputtering. The structures, magnetic properties, and high frequency permeabilities of the multilayer films are systematically analyzed. All films present evident IPUMA and high M_s . Besides, the films with thin magnetic layer thickness values exhibit good high frequency properties. For our typical sample with t = 10 nm, the saturation magnetization is 1.01 T and the resistivity equals 113 $\mu\Omega \cdot cm$. When operating in GHz range, the real part of the permeability could remain at 300 in GHz range and the resonance frequency ap-

plication. Meanwhile, further studies are needed to extend the resonance frequency into a larger range.

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