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Magnetic properties and microstructure investigation of electrodeposited FeNi/ITO films with different thickness

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ABSTRACT

FeNi alloy thin films with different thickness deposited on Indium Tin Oxide (ITO) conductive glass substrates from the electrolytes by electrodeposition method have been studied by magnetic force microscopy (MFM), scanning electron microscopy (SEM) and ferromagnetic resonance (FMR) technique. For these films possessing an in-plane isotropy, the remanence decreases with the increasing of film thickness and the critical thickness that a stripe domain structure emerges is about 116 nm. Characteristic differences of the FMR spectra of different thickness are also observed. The results show that the resonance field at high measured angle increases firstly then decreases with increasing thickness, which may be related to the striped domain structure.

1. Introduction

Magnetic thin films have been attracted substantial interest due to their present and potential applications in various magnetic technologies especially the fundamental differences in magnetic and electronic properties from their bulk counterparts [1–3]. Soft magnetic films with excellent high frequency characteristics are widely applied to micro devices such as micro-inductors, micro-transformers, magnetic recording heads and near-field electromagnetic noise absorbers [4]. Soft magnetic films can be fabricated by several methods, such as molecular beam epitaxy (MBE) [5], sputtering [6], thermal evaporation [7], electroless plating [8] and electrodeposition [26–29]. The electrodeposition method has been widely used in many industrial productions because of its low capital cost, high deposition rate, easy operation and requiring relatively inexpensive equipment. In addition, the properties of the materials can be regulated by controlling solution concentration and deposition parameters.

Permalloy is the iron–nickel alloy with a higher permeability in a weak magnetic field. Permalloy can be classified to different types, the alloy at the content of nickel in 45–50% has the highest saturation magnetization, and 70–81% has the highest permeability [30,31]. FeNi thin films, as a very important soft magnetic material and good physical properties for applications, have been studied for a long time by various experimental techniques [9–11]. FeNi based alloys with or without stripe domain [12] have been reported in soft magnetic films with monofilm or multilayers onto different substrate, such as FeNi/Cu/FeNi multilayer films [13], FeNi films and FeNi/Ti films [14] and $Ni_{75}Fe_{16}Cu_5Mo_4$ films [15]. All these reports show that the films perform different properties as the thickness changes. Dependence of the FeNi thin films on thickness have been researched; however, it focused on a narrow range of film thickness, further studies are still necessary to provide more documents for the FeNi thin films.

Ferromagnetic resonance (FMR) is a powerful technique to determine the magnetic anisotropy of thin films and multilayers [16,17]. For the characterization of magnetic thin films, the method has been widely used [19], and the properties of many different FeNi-based thin films with different layers have been studied for a long time by FMR [16,18–23]. However, these previous results about FeNi films or multilayers by FMR focused on the influence of different temperatures or substrates on the resonance spectrum of the films, there have been few attempts to investigate the delicate morphology, domain structure as well as the FMR spectra for a series of FeNi films with different thickness.

In this work, FeNi thin films were electrodeposited by controlling electrodeposition condition and series of different thickness (from 23 nm to 371 nm) of FeNi alloy films onto ITO conductive glass substrates were obtained successfully. The influence of thickness on morphology, domain structure and FMR spectra are then investigated.

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2. Experimental

All deposition experiments were performed at room temperature using a stationary three-electrode cell with an electrochemical workstation (CHI 860D), which the reference electrode was a saturated calomel electrode (SCE) and a platinum (Pt) strip with an area of 4 cm^2 was used as counter electrode. The working electrode was ITO conductive glass. The electrolyte was in a single mixed solution composed of 0.05 M iron(II) sulfate heptahydrate, 0.05 M nickel(II) sulfate heptahydrate combined with 0.5 M boric acidas as a pH buffer, 1 g/L ascorbic acid, 2 g/L glycine and 2 g/L saccharine as complexing agents to keep the ferrous iron and nickel from oxidizing. The electrochemical potential was −1.3 V and all chemicals were in reagent grade and solved in distilled water. The experiments remained every solution concentration unchanged so that we could regulate the electrodeposition time in order to obtain the films with different thickness.

The thicknesses of the films were measured with a Surface Profile-meter (Dektak 8). Inductively couple plasma-atomic emission spectrometry (ICP–AES) was utilized to determine the elemental compositions of the films. The crystal structure was characterized by X-ray diffraction (XRD) (PANaluticalX'Pert) with Cu-Ka radiation. Magnetic properties of the FeNi films were measured by a vibrating sample magnetometer (VSM) (Lakeshore 7304). A magnetic force microscopy (MFM) (Asylum Research MFP3D) was used to study the domain structure of the films. During all the measurement process of MFM, the direction of magnetic field paralleled to the surface of FeNi thin films. The morphology of the sample was characterized by a field emission scanning electron microscopy (SEM) (Hitachi S-4800, Japan) equipped with an energy-dispersive X-ray spectrometer, The ferromagnetic resonance measurements were performed by using an X-band spectrometer (f = 9 GHz, JEOL, JESFA300).

3. Results and discussion

Table 1 shows the film thickness as a function of the deposition time. As can be seen, the thickness of FeNi films increases with the increasing deposition time at the same deposition condition. Since the solution concentration changed slightly with the deposition time during the deposition course, so the film thickness was not linearly proportional to the deposition time. The elemental composition of the films was analyzed by ICP–AES, and the atomic rate of Fe:Ni was about 1:1.

Fig. 1 shows the XRD patterns of FeNi films with different thickness. FeNi films with fcc structure are obtained for each thickness, and the two peaks are indexed as (110) and (211) reflection. At first, the spectra of ITO are observed clearly, but with the increasing thickness. The increasing reflection intensity of FeNi alloy covers the signal of ITO substrate. FeNi reflection intensity becomes more evident while the spectra intensity of ITO conductive glass grows weaker and disappears in the thicker thickness (355 nm, 371 nm).

Table 1
Film thickness dependent on deposition time in FeNi thin films.

Time (s)	50	100	200	300	400	500
Film thickness (nm)	23	63	116	189	355	371

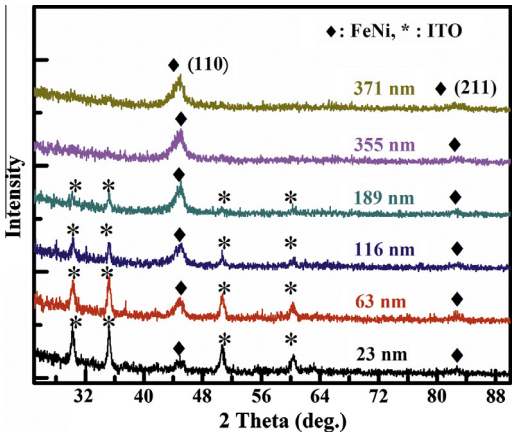

Fig. 1. XRD spectra for electrodeposited FeNi films with different thickness.

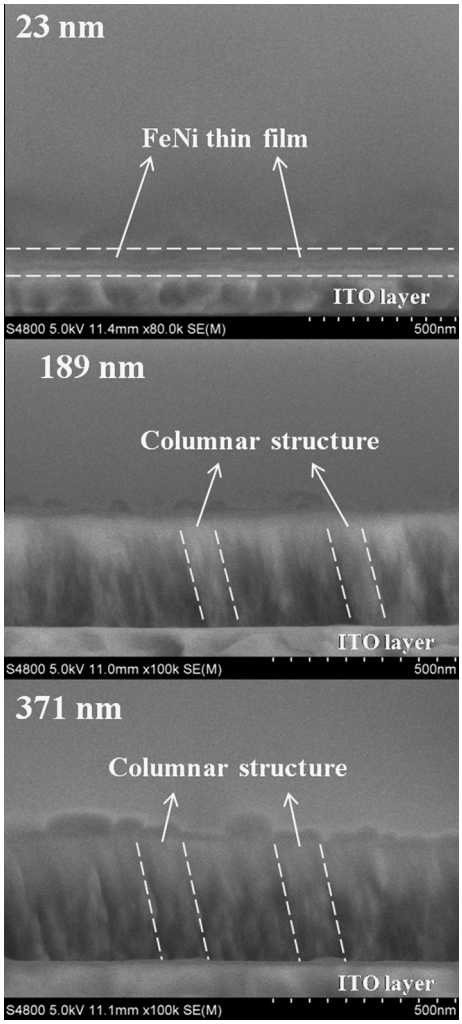

Fig. 2. The cross-section SEM of FeNi thin films: 23 nm, 189 nm and 371 nm.

Fig. 2 shows a cross-section SEM micrograph of FeNi films. The dashed line severs as guide to the eye. It shows that the FeNi films in the thickness of 189 nm and 371 nm emerge the columnar crystalline microstructure. As the thickness decreases to several tens of nanometers (like 23 nm), the columnar nanocrystalline microstructure disappear. Only a relatively tough surface can be seen.

Further investigation of static magnetic properties of FeNi films has been carried out by VSM. Fig. 3 shows the in-plane hysteresis loops depended on the film thickness. The thickness dependence of remanence ratio is also shown in the inset of Fig. 3. When the thickness increased to 116 nm, the remanence ratio decreases to about 0.7. A further increase in thickness causes a continuously reduction of the remanence ratio. From the results of Fig. 2, this behavior of hysteresis loops (189 nm, 355 nm, 371 nm) may be correlated with the presence of columnar crystalline structure, which results in an out-of plane magnetization component. As the thickness increase, the aspect ratio of columnar nanocrystalline increases, which enhances the out-of plane magnetization component. Therefore, the in-plane remanence ratio decreases.

The columnar microstructure in Fig. 2 and the hysteresis loops in Fig. 3 were encouraged to investigate the domain structures in FeNi films with MFM directly. Fig. 4 depicts the magnetic domain image of FeNi thin films with different thicknesses (23 nm,