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MAGNETIC AND ELECTRICAL PROPERTIES OF [NiFe/SiO_2]x N MULTILAYER THIN FILMS

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The ferromagnetic material/SiO_2 multilayer films exhibit very good soft magnetic properties attributed to nanocrystalline metallic phases. Concurrently, high resistivity of ferromagnetic material/SiO_2 multilayer thin films receives much attention from the standpoint of low core-loss for high frequency use. Permalloy NiFe is an important material widely used in magnetic industry because of its permeability and low anisotropy. This paper reports some results concerning the effects of the composition and thermal treatment conditions on the structural, electrical and magnetic properties of [FeNi/SiO_2]×N thin films. The [FeNi(6nm)/(SiO_2)(2nm)]×30 films exhibit high resistivity over 10^{-2} mΩ-m, while superior soft magnetic properties, specific for FeNi thin films are maintained. The [FeNi/(SiO_2)]×30 annealed films show good magnetic properties of saturation magnetization ≅ 1670×10^{-7} Tm^3/kg and coercivity ≅ 1520 A/m. This class of magnetic materials may be interesting for thin - film inductors which can operating at high frequencies.

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1. Introduction

New materials based on nanosized metal have attracted much attention in recent years due to their fundamental interest as well as their potential applications in many technological areas [1]. Magnetic/nonmagnetic multilayer thin films are of great interest for high frequency circuit applications [2,3]. The ferromagnetic material/SiO_2 systems are composite materials consisting of nanometer-order granular and insulator layers. Their electrical and magnetic properties are strongly dependent on composition and microstructure. This fact allows the optimization of the characteristics required for different kinds of magnetic microdevices as R.F magnetic microinductors. The thin films of a granular ferromagnetic material/SiO_2 system exhibit excellent soft magnetic properties such as high saturation magnetization ($4\pi M_s$), low coercivity (H_c), high effective permeability (μ_{eff}) and electrical properties such as very high resistivity. It is well know that FeNi alloy thin film is very important soft magnetic materials due to their high magnetic moment and low magnetostriction. Investigation on high purity FeNi alloys allowed us to determine the conditions yielding optimum soft magnetic properties. Such characteristics are obtained when the competitive growth of BBC and FCC phases of FeNi is induced, producing smallest grain size and minimum internal stresses. Its electrical and magnetic characteristics, however, often make the material too lossy to be used at above ~ 100 MHz. This paper reports some results concerning the effects of the composition and thermal treatment conditions on the structural, electrical and magnetic properties of [FeNi/SiO_2]xN thin films. The dependence of the resistivity, saturation magnetization and coercivity on the bilayers FeNi/SiO_2 number (N) and annealing temperature of these thin films were investigated.

2. Experimental

The [$Fe_{20}Ni_{80}$/SiO_2]xN multilayer films were prepared using a conventional R.F. diode sputtering system by sequential deposition in vacuum from two elemental targets, disc of $Fe_{20}Ni_{80}$

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alloy and disc of SiO_2. The thickness of layers was controlled by sputtering time and measured by using a Film Thickness Monitor (resolution 0.1 nm) during deposition process. The total thickness of FeNi layers in multilayer system was of 360 nm.

The samples were deposited on various substrates, depending on the intended measurements. The substrates were cooled during deposition.

For resistivity measurements and structure analysis, we deposited samples on glass substrates. The resistivity was measured, before and after the thermal treatment, using the standard four-point probe method.

The mainly magnetic characteristics (saturation magnetization M_s and coercive field H_c) of the samples deposited on glass substrates were measured using a vibrating sample magnetometer, in a magnetic field of up to 1.5×10^3 kA/m.

The structure of the $[Fe_{20}Ni_{80}/SiO_2]$xN multilayer films was investigated using X-ray diffraction (XRD) analysis. An X-ray diffractometer with a monochromatized Mo-Kα radiation (λ = 0.71069 Å) was used, in a Bragg-Brentano arrangement.

The microstructure of $[Fe_{20}Ni_{80}/SiO_2]$xN multilayer films was analysed by transmission electron microscopy (TEM), using standard copper 'microscope grids' coated with an evaporated carbon (8 – 10 nm) thin films as substrates. $[Fe_{20}Ni_{80}$ (6 nm)/SiO_2(2 nm)]×10 multilayer films with a total thickness of about 80 nm were used for TEM analysis. The electron microscopy studies were carried out with a JEOL –200 CX microscope.

The as – deposited $[Fe_{20}Ni_{80}/SiO_2]\times N$ multilayer films were subsequently annealed, in vacuum, at temperatures between 250 °C and 400 °C, in order to enhance and stabilize their physical properties.

3. Results and discussion

For gigahertz band applications we studied magnetic multilayer granular films for which the resistivity must be as high as possible. The magnetic and resistive properties of $[FeNi/SiO_2]$xN multilayer thin films strongly depend on the number $FeNi/SiO_2$ bilayers (N) and thickness of metallic and SiO_2 layer. We studied the dependence of the resistivity of the $[FeNi/SiO_2]$xN thin films on the value of the number $FeNi/SiO_2$ bilayers (N). The values of the resistivity, for as-deposited and thermally treated samples (at. 350 °C, for 2 h, in vacuum) are presented in Table 1.

Table 1. Values of the resistivity for as-deposited and annealed $[FeNi/SiO_2]\times N$ thin films.

Samples	Resistivity (mΩ·m)	
	As -deposited	After annealing at 350 °C
FeNi	0.98×10^{-3}	0.72×10^{-3}
[FeNi(18nm)/SiO2(6nm)] × 10	1.5×10^{-2}	9.3×10^{-3}
[FeNi(12nm)/SiO2(4nm)] × 15	9.6×10^{-2}	6.4×10^{-2}
[FeNi(6nm)/SiO2(2nm)] × 30	0.62	0.48

The resistivity values increase with increasing the number $FeNi/SiO_2$ bilayers (N) for as-deposited samples. For all the samples the resistivity values decrease after annealing at temperature of 350 °C. The decrease of resistivity after the thermal treatment is due to an increase of the metallic grain dimension because of the recrystallization phenomena in the metallic layers, fact also suggested by XRD and TEM analysis. It is possible that the recrystallization process is associated with a diffusion process at $FeNi/SiO_2$ interfaces.

In Fig. 1 are presented the XRD patterns of $Fe_{20}Ni_{80}$ and $[Fe_{20}Ni_{80}/SiO_2]$xN thin films, after annealing. The X-ray diffraction pattern of $Fe_{20}Ni_{80}$ thin film after annealing at 350 °C (curve a) shows a nanocrystalline structure indicated by the presence of small sharp peaks. XRD patterns corresponding to $[Fe_{20}Ni_{80}/SiO_2]\times N$ thin films after successive annealing in vacuum, at temperatures up to 300 °C do not exhibit evident changes as compared to X-ray diffraction patterns of as-deposited samples, indicating that no major structural changes were produced by these treatments.

The XRD patterns of [$Fe_{20}Ni_{80}$/SiO_2]×N thin films (curve b and c) show a nanocrystalline or amorphous structure, as follows: nanocrystalline structure indicated by the presence of small broad peaks for [$Fe_{20}Ni_{80}$/SiO_2]×10; amorphous structure for [$Fe_{20}Ni_{80}$/SiO_2]×30 (curve c), indicated by a broad peak at $2\theta = 19$ deg., corresponding to the FCC (Fe,Ni) reflection.

Fig. 2 shows the electron micrographs and electron diffraction patterns of [$Fe_{20}Ni_{80}$(6 nm)/SiO_2(2 nm)]×10 thin films, for the as-deposited state (Fig. 2a) and after annealing at 350 °C (Fig. 2b). In general, the microstructure of metal/insulator thin films consists of approximately spherical, very small metal particles separated by smaller barriers of insulator material.

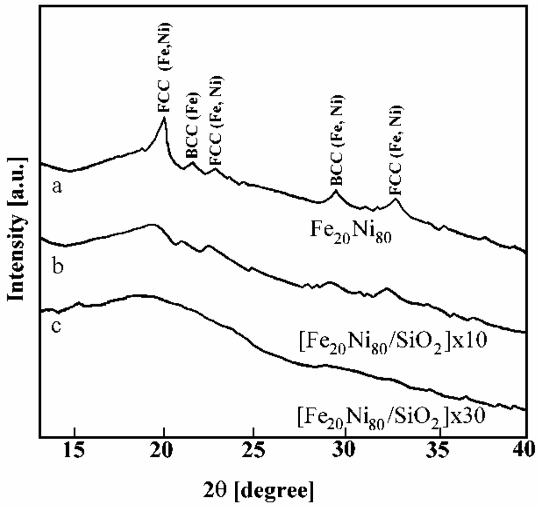

Fig. 1. X-ray diffraction patterns of $Fe_{20}Ni_{80}$ and [$Fe_{20}Ni_{80}$/SiO_2]xN /SiO_2]xN thin films, after annealing at 350 °C.

The electron micrograph for of [$Fe_{20}Ni_{80}$ (6 nm)/SiO_2(2 nm)]×10 thin films shows a labyrinth structure consisting of short filamentary chains with interconnected FeNi particles and the dispersed insulating materials, filling in the free spaces. Most of FeNi particles are discontinuous, but a significant fraction exists as small chains with bridges. Microstructure of the multilayers of [$Fe_{20}Ni_{80}$ (6 nm)/SiO_2(2 nm)]×10 consists of approximately spherical, very small FeNi particles separated by narrow barriers of SiO_2. The FeNi particles are distributed uniformly in SiO_2. Both micrographs reveal fine metallic particles embedded in an amorphous matrix of SiO_2. For as-deposited films the particle size values are about 10 nm and after annealing at 350 °C the particle size values range between 15 and 20 nm.

The increasing sharpness of the electron diffraction patterns after annealing at 350 °C (Fig. 2b) is caused by the metallic particles growth. The changes in samples' microstructure after annealing at 350 °C are related to observed changes of the resistivity values (Table 1).

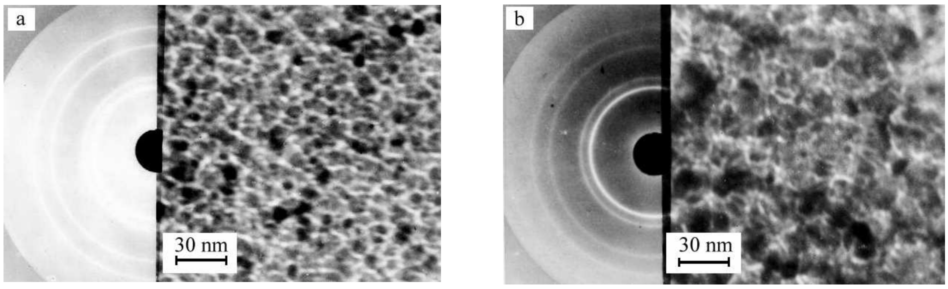

Fig. 2. TEM micrographs and electron patterns of [$Fe_{20}Ni_{80}$ (6 nm)/SiO_2(2 nm)]×10 thin films for as-deposited state (a) and after annealing at 350 °C (b).

Fig. 3 shows the hysteresis loops at room temperature for selected samples with the applied magnetic field parallel to the sample plane. One can see that the values of the saturation magnetization for FeNi (360 nm) and [FeNi(6 nm)/(SiO_2)(2 nm)]×30 thin films increase, while the coercive field values decrease, after annealing at 350 ^oC. The [FeNi/(SiO_2)]×30 annealed films exhibit a saturation magnetization, $M_s \cong 1750\times10^{-7}$ Tm^3/kg and a coercive field, $H_c \cong 620$ A/m.

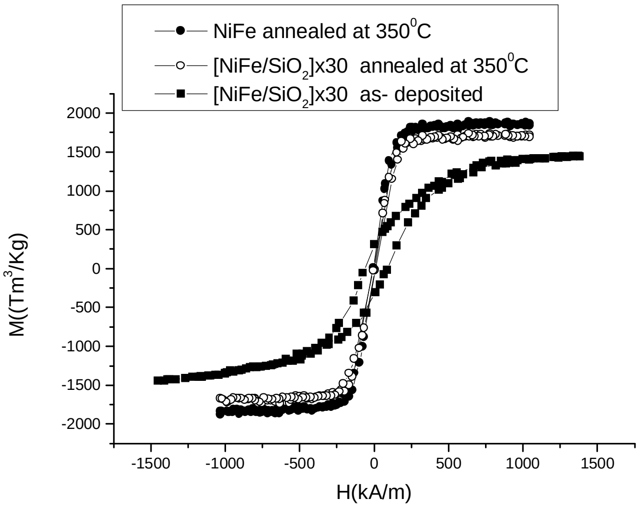

Fig. 3. Magnetic hysteresis loops of FeNi and [FeNi (6 nm)/SiO_2(2 nm)]×30 thin films, in as-deposited and annealing state.

The compositional changes in [FeNi/(SiO_2)]×N multilayer films occur by variation of the FeNi/SiO_2 number layers (N) and their correlation with the associated changes in the basic morphological parameters, mainly govern the electrical and magnetic response of these materials. We got magnetic films showing not only high resistivity, but also excellent soft magnetic properties.

Recently [4] we have shown that in [FeCoB/(SiO_2)]×N sputtered thin films one observes different physical properties by changing the thickness for FeCoB and SiO_2 layers. For n>30 the multilayer exhibits high resistivity and the excellent soft magnetic properties specific to FeCoB films are maintained.

By analyzing the presented results it can be seen that by controlling of layers thickness in multilayer systems, the nanogranular thin films with good resistive and soft magnetic properties were obtained. Future work includes the optimization of the micromagnetic structure of these thin films by growth on suitable substrates.

4. Conclusions

The [FeNi(6nm)/(SiO_2)(2nm)]×N thin multilayer films exhibit high resistivity over 10^{-2} mΩ-m, while the excellent soft magnetic properties, specific for FeNi thin films, are maintained. The [FeNi(6nm)/(SiO_2)(2nm)] × 30 annealed films show good magnetic properties of saturation magnetization ≅ 1750 × 10^{-7} Tm^3/kg and coercive field ≅ 620 A/m. The excellent electrical and soft magnetic behavior is a consequence of their ultrafine metallic grain structure. By analyzing the presented results it can be seen that [FeNi/(SiO_2)]×N thin films are interesting for micromagnetic devices including magnetic thin-film inductors, which can operate at high frequencies.

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