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May 01, 1990

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Recommended Citation

Harris, V. G.; Oliver, S. A.; Nowak, W. B.; and Vittoria, C., "Magnetic and microwave properties of ion-beam-sputtered amorphous $Fe_xCo_{80-x}B_{15}Si_5$ films" (1990). *Mechanical and Industrial Engineering Faculty Publications*. Paper 31. http://hdl.handle.net/2047/d20000722

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Magnetic and microwave properties of ion-beam-sputtered amorphous $Fe_xCo_{80-x}B_{15}Si_5$ films

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Magnetically soft amorphous films of $Fe_xCo_{80-x}B_{15}Si_5$ ($x = 0, 6, 23, 40, 70, 80$) were ion beam sputter deposited onto fused quartz for static and microwave magnetic characterization. Films ranged in thickness from 220 to 260 nm and were deposited at rates of 0.1–0.2 nm/s. Saturation magnetization, coercivity, and loop squareness values were extracted from hysteresis loops generated by a vibrating sample magnetometer. Ferromagnetic resonance measurements were taken using a 9.5-GHz cavity with the applied magnetic field both parallel and perpendicular to the plane of the film, yielding values for the g factor, anisotropy field, effective magnetization, and linewidth. Well resolved quadratic spin-wave resonance spectra allowed for the deduction of exchange stiffness constants. Hysteresis loops showed well-defined uniaxial in-plane anisotropies for Fe-rich films, with easy axis loop squareness decreasing with decreasing Fe content. Saturation magnetization and effective magnetization values were found to reach a maximum at $x = 70$ for those compositions investigated. These films were found to have soft magnetic properties comparable to the $Fe_xNi_{80-x}B_{15}Si_5$ alloy films previously investigated.

INTRODUCTION

Amorphous ferromagnetic alloys, consisting of transition metals (TM = Fe, Co, Ni) and metalloids (M = B, Si, C, P), have been extensively studied for their excellent mechanical, corrosion resistant (with Cr added), and soft magnetic properties. This unique combination of properties, coupled with the higher electrical resistivities necessary to reduce eddy current losses at high frequencies, make these materials excellent alternatives to existing polycrystalline metallic alloys for microwave magnetic device applications. Amorphous TM-M alloys have been studied as both ribbons[1-4] and films.[5-12] However, further investigation of films is needed prior to their widespread acceptance, development, and deployment in microwave magnetic devices.

Our interest in the Fe-Co-B-Si system stems from previous investigations of ion beam sputtered (IBS) films of the Fe-Ni-B-Si system,[8-11] which displayed soft magnetic properties comparable to polycrystalline permalloy, in addition to the desirable properties characteristic of amorphous materials.

EXPERIMENTAL PROCEDURE

Films were deposited at ambient temperatures onto fused quartz substrates by IBS deposition. Details of the deposition process have been previously reported.[10,11] Targets are 8-cm-diam disks of hot-pressed 99.5% purity powders. The holder assembly was rotated at 2 rpm during deposition to improve thickness and composition uniformity across the substrate.

Deposition rate and film thickness were monitored *in situ* with a quartz crystal oscillator and confirmed by surface profilometer traces of a film step created from masking the substrate during deposition. Deposition rates were typically 0.1–0.2 nm/s resulting in film thicknesses ranging from 220 to 260 nm.

All $Fe_xCo_{80-x}B_{15}Si_5$ ($x = 0,6,23,40,70,80$) films were deposited at beam conditions determined optimal for soft magnetic properties in a previous investigation of beam parameter effects on soft magnetic properties of $Fe_{40}Co_{40}B_{15}Si_5$ films.[11]

Reflection electron diffraction (RED) was used qualitatively to confirm the amorphouslike state of the films. Representative films were characterized chemically by Rutherford backscattering spectroscopy (RBS)[13] and Auger electron spectroscopy (AES) and were found to be within $\pm 3\%$ of target compositions. All film compositions will be referred to by the nominal composition of the target sputtered.

Several 5.3-mm-diam disks were cut from the films using a diamond corer. These samples were used in collinear four-point probe resistivity measurements, magnetic characterization by vibrating sample magnetometer (VSM), and ferromagnetic resonance (FMR) measurements. VSM measurements, made with a dc magnetic field applied parallel to the film plane, generated high resolution hysteresis loops from which values of the coercive field (H_c), saturation magnetization ($4\pi M_s$), and loop squareness ($Sq = I_r/I_s$) were extracted. X-band cavity ferromagnetic resonance (FMR) measurements were performed with the applied field (H) lying both parallel and perpendicular to the plane of the film allowing for deduction of in-plane and perpendicular anisotropies ($H_k, H_u^{\perp}$), effective magnetization ($4\pi M_{eff}$), and g factor. Analysis of spin-wave resonance (SWR) spectra allowed for calculation of exchange stiffness constants (A). In addition, a microwave swept frequency transmission technique[10] measured the zero-field-resonance (ZFR) frequency, i.e., frequency at which the film is magnetically resonant with $H = 0$.

RESULTS AND DISCUSSION

The deposition parameters chosen were those found to minimize contaminants trapped within the film during deposition. This was accomplished by selectively choosing beam parameters to generate higher deposition rates, while

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TABLE I. Static magnetic properties of IBS $Fe_xCo_{80-x}B_{15}Si_5$ films extracted from hysteresis loop measurements by vibrating sample magnetometer, also listed are miscellaneous physical properties. (Note: E and H indicate orientation of applied field relative to the films' easy and hard axes.)

x (at. % Fe)	t (nm) ($\pm 3\%$)	Sq (E/H)	H_c (Oe) (E/H)	$4\pi M_{sat}$ (kOe) ($\pm 2\%$)	ρ_e ($\mu\Omega$ cm)	ρ_d (g/cm^3)
0	260	0.45/0.48	36.1/36.3	10.56	329	7.57. $\pm$ 0.28
6	231	0.80/0.36	0.11/0.16	11.43	150	···
23	229	0.95/0.05	1.05/2.15	13.98	196	···
40	233	0.93/0.05	0.54/0.65	13.51	161	8.48 $\pm$ 0.76
70	230	0.95/0.07	2.48/2.50	15.33	192	···
80	220	0.97/0.04	2.78/1.05	13.17	176	7.64 $\pm$ 0.31

maintaining sputtered impurities at an acceptable level. The primary sputtered contaminants detected were tungsten and carbon, generated from incidental sputtering of the ion source's neutralizer filament and acceleration grid assembly, respectively. Their presence was confirmed in chemical analysis by both RBS and AES. In particular, the presence of tungsten is believed to be detrimental to soft magnetic properties; the effect of carbon is not yet fully understood.[11]

Film densities were found for films $x = 0,40,80$ by depositing a series of thicker films (≈ 1 μm) onto thin coverglasses. Measured densities (Table I) were found to agree, within error bars, with previously reported densities for similar TM-M alloy ribbons.[1,14] Room-temperature electrical resistivities are also listed in Table I. In a previous study[11] a clear dependence was observed between film resistivity values and the base pressure attained prior to deposition; this was attributed to gases (working and residual) trapped within the film during deposition.

Hysteresis loops of films produced from sputtering targets: $x = 23,40,70,80$ displayed well-defined easy and hard axes with coercivities ranging from 0.11 to 2.78 Oe (Table I). The coercivities for some films were found to be unusually high for amorphous materials which, due to their lack of atomic periodicity, are expected to possess values less than 100 mOe.[5,6] The dominant domain wall pinning (DWP) mechanism leading to these uncharacteristically high coercivities is believed to be structural inhomogeneities arising from gases trapped during deposition. The presence of tungsten in these films may also contribute significantly as DWP centers.

Hysteresis loops of the Co-rich films ($x = 0,6$) indicate that the easy axes lie out of the film plane. The hysteresis loop of $Co_{80}B_{15}Si_5$, indicative of perpendicular anisotropy,[6] is presented in Fig. 1(b). Representative easy and hard axes loops for Fe-rich films are presented in Fig. 1(a). The overall behavior of loop squareness with composition is illustrated in Fig. 2. It is clear that the films' easy axes align out of the film plane with decreasing Fe content. Perpendicular anisotropy values (Table II) ($H_u^{\perp} = 4\pi M_{eff} - 4\pi M_{sat}$) do not reflect this tendency, however, this may be concealed within the magnitude of the error associated with the saturation magnetization values.

Saturation and effective magnetization values were found to increase with iron content, peaking at $x = 70$, for those compositions investigated (see Tables I and II). Similar trends have been reported in investigations of polycrystalline Fe-Co alloys and Fe-Co-B alloy ribbons, where the saturation magnetization maximum was reported at $x = 65$.[1,2] The values of saturation magnetization presented here agree with those reported elsewhere for sputtered films of similar compositions,[5-12] however, they are consistently low compared with results reported for ribbons.[2] This may be attributed to the presence of silicon as a glass stabilizer, whose valence electrons contribute to the d band of the transition metals, diminishing the alloy's net magnetization.[2]

FMR deduced anisotropy field values were found to de-

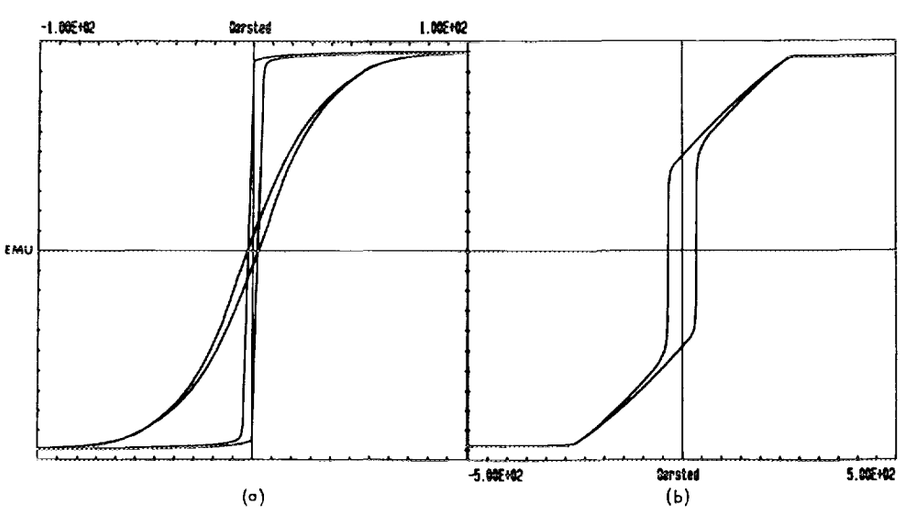

FIG. 1. Representative hysteresis loops of (a) Fe-rich films ($x = 40,70,80$) with applied field along easy and hard axes, and (b) $Co_{80}B_{15}Si_5$ film displaying perpendicular anisotropy.

TABLE II. Microwave magnetic properties of IBS $Fe_xCo_{80-x}B_{15}Si_5$ films deduced from ferromagnetic resonance measurements.

x (at. % Fe)	$4\pi M_{eff}$ (kOe) ($\pm 2\%$)	ΔH (Oe) ($\pm 2\%$)	$A(10^{-6})$ (ergs/cm) ($\pm 8\%$)	$H_u^{\perp}$ (kOe) ($\pm 8\%$)	g ($\pm 2\%$)	H_k (Oe)	f_o (GHz) ($\pm 10\%$)
0	10.79	29	0.99	0.23	2.13	10 ± 2	1.54
6	12.13	32	1.17	0.70	2.18	25 ± 2	1.37
23	13.21	26	1.25	−0.77	2.17	35 ± 10	0.97
40	14.00	24	1.37	0.49	2.14	12.5 ± 2	0.94
70	14.35	35	1.53	−0.98	2.14	6 ± 1	0.66
80	13.14	34	0.73	−0.03	2.13	3.5 ± 1	0.90

crease with increased iron content attaining a low of 3.5 Oe at $x = 80$, shown by Table II. Although anisotropy has been found to be sensitive to processing,[12,15] the observed trend appears to be one related to composition. Surprisingly, the $Co_{74}Fe_6B_{15}Si_5$ film, reported extensively as a near-zero magnetostrictive composition,[4,7,15,16] had the highest as-deposited in-plane anisotropy value of those compositions investigated. Preliminary heat treatment results show hard axis field annealing to be effective in aligning the easy axis in the film plane and reducing the anisotropy field value from 28 to 10.5 Oe. Films of other compositions showed similar improvements.[17] These results may indicate the presence of substantial amounts of film stress often found in sputtered films.

Perpendicular FMR measurements displayed well-defined spin-wave resonance (SWR) spectra for all films. Fe-rich films ($x \geqslant 40$) possessed a higher field mode well above that predicted from the quadratic spin-wave model. This was also observed in earlier results of $Fe_xNi_{80-x}B_{15}Si_5$ films[10] and was attributed to inhomogeneities in magnetization and/or anisotropy near the interfacial regions. In contrast, the SWR spectra of Co-rich ($x < 40$) films were predicted accurately by the quadratic SWR model, once conductivity effects were included. Values for the exchange stiffness constant (A) are listed in Table II, as well as results for the perpendicular linewidth (ΔH), g factor, and ZFR frequencies. All g-factor values lie within the range of the polycrystalline values for Fe and Co; $g_{Fe} = 2.091$ and $g_{Co} = 2.187$.[18] Experimental line widths were found to be less than or comparable to IBS permalloy films.[8] In addition, the films were found by ZFR measurements to experience magnetic resonance below 2 GHz without an applied magnetic field.

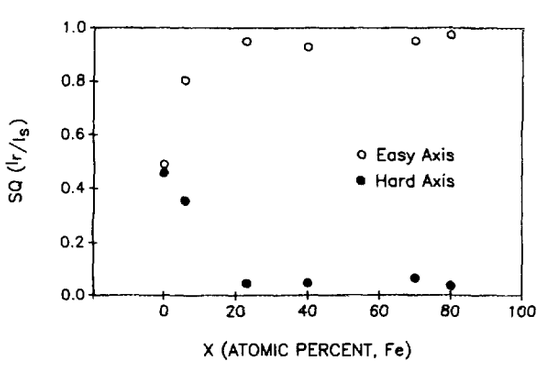

FIG. 2. Hysteresis loop squareness is plotted vs composition illustrating the tendency of the film's easy axes to align outside the film plane with decreasing iron content.

CONCLUSIONS

These films were found to possess static and microwave magnetic properties which lend themselves to microwave magnetic applications. Specifically, the films display soft magnetic properties comparable to polycrystalline permalloy, with higher magnetization values ($4\pi M_s$,$4\pi M_{eff}$), and electrical resistivities 2–5 times greater. Therefore, these films are excellent alternatives for high frequency applications where minimizing eddy current losses is of primary importance. In comparison to the $Fe_xNi_{80-x}B_{15}Si_5$ films previously investigated, these films displayed magnetization values 5%–20% greater. However, in their as-deposited state, anisotropy field values were higher than those reported for both permalloy and the $Fe_xNi_{80-x}B_{15}Si_5$ films.

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