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Correlation of magnetic properties and local atomic structure observed by electron-yield EXAFS in amorphous $\text{Co}_{74}\text{Fe}_6\text{B}_{15}\text{Si}_5$ thin films

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Thin-film specimens of amorphous $\text{Co}_{74}\text{Fe}_6\text{B}_{15}\text{Si}_5$ were subjected to heat-treatment procedures followed by room-temperature magnetic and structural characterization in an effort to quantitatively correlate magnetic property changes to local atomic structure. Fourier transforms of electron-yield extended x-ray-absorption fine structure spectra show second and higher atomic shell ordering about transition-metal ions to occur at $T_{\text{ann}} \approx 300^\circ\text{C}$. This ordering evolves to a body-centered atomic structure with heat treatments at higher temperatures. Accompanying this structural evolution is an expansion of the nearest-neighbor distance, disruption of the spin-wave-resonance spectra, and general deterioration of the soft magnetic properties.

I. INTRODUCTION

Amorphous magnetic materials possess a metastable short-range-order atomic structure, which often requires a stabilizing heat-treatment procedure prior to application. The irreversible effects of this heat treatment on the atomic structure and magnetic properties have yet to be fully understood. A clear understanding of these effects would undoubtedly refine processing technology and enhance the search for novel magnetic materials.

This paper reports on a joint magnetic and structural study of soft magnetic amorphous thin films which employs extended x-ray-absorption fine structure (EXAFS) analysis as an elemental specific, local structural probe. An initial study showed the viability of EXAFS as an effective technique for providing information of the atomic environment of transition-metal ions in thin-film specimens.¹ However, the limited number of samples used and indications of magnetic nonuniformities in some annealed film specimens precluded determination of most trends. Presented here are the results of a more extensive study which details local structural changes undergone by transition-metal ions and corresponding film magnetic properties, as a function of annealing temperature.

II. EXPERIMENTAL PROCEDURES

A 150-nm-thick film was deposited by ion-beam sputtering (IBS) from a $74\text{Co}-6\text{Fe}-15\text{B}-5\text{Si}$ pressed powder target. To aid in structural analysis, standards of elemental iron and cobalt of comparable thickness were also deposited. Details of the deposition process and results of magnetic and chemical analyses of films have been presented elsewhere.^{2,3}

Ten 5-mm-diam disk specimens, cut from the as-deposited film using a diamond corer, underwent room-temperature vibrating sample magnetometer (VSM) measure-

ments to determine each specimen's static magnetization behavior and to ensure magnetic uniformity of the group. All annealing procedures took place under argon flow in an oven interfaced to the VSM with a saturation field of 500 Oe applied in the film plane parallel to the hard axis. During heat treatment the specimens were maintained at the selected annealing temperature for a period of 20 min.

After annealing all specimens underwent room-temperature VSM, X-band cavity ferromagnetic resonance (FMR), and four-point probe electrical resistivity measurements. In addition, EXAFS spectra for the transition-metal ions were collected at room temperature using the U. S. Naval Research Laboratory's materials analysis beamline at the National Synchrotron Light Source (Brookhaven National Laboratories, Upton, NY).

X-ray absorption spectra were collected using an electron-yield technique where the sample is mounted as the cathode in a helium gas ionization chamber.⁴ The generated sample current, composed mainly of Auger electrons arising from the absorption process, is normalized relative to the incident photon flux. Measured EXAFS spectra typically encompass photon energies extending ≈ 100 eV below the Fe and Co *K*-edges to 500–700 eV above each edge. Following standard EXAFS analysis,⁵ the absorption spectrum above the edge is isolated, a linear background subtracted, and converted from energy space to photoelectron wave-vector (*k*) space. The *k*-space spectrum then undergoes Fourier transformation. The resulting Fourier transformed data represent the atomic environment of the central TM ion uncorrected for electron phase shifts. Due to beamline constraints of incident photon energy, EXAFS spectra were only available for the transition-metal ions.

III. RESULTS

VSM measurements were performed on annealed film specimens with the dc magnetic field applied both parallel and perpendicular to the film plane. Figure 1(a) shows the room-temperature saturation magnetization (as $4\pi M_{\text{sat}}$) of annealed specimens normalized to the as-deposited value

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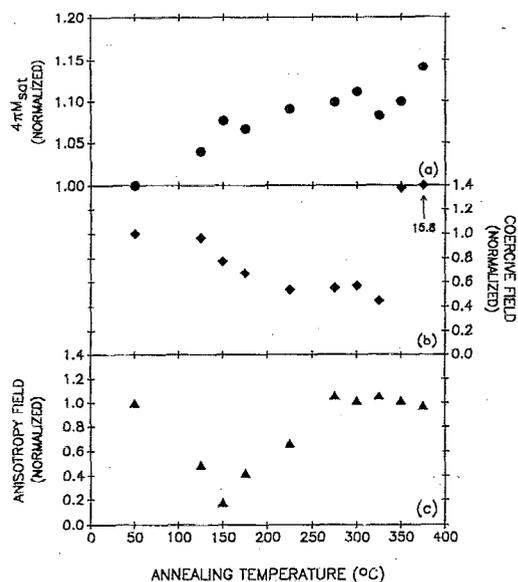


FIG. 1. Magnetic properties ($4\pi M_{\text{sat}}, H_a, H_c$) as a function of annealing temperature. Values have been normalized to the as-deposited value.

of 11.45 ± 0.49 kOe. Results show gradual increases in $4\pi M_{\text{sat}}$ values with specimen annealing temperatures to 300°C [see Fig. 1(a)]. FMR measurements show increases in effective magnetization ($4\pi M_{\text{eff}}$) and A/M_{sat} values with increasing annealing temperature, T_{ann} , where A is the exchange stiffness constant. Since M_{sat} values were measured to increase with T_{ann} , it is implied that A is also increasing with T_{ann} . By taking the difference between $4\pi M_{\text{sat}}$ and $4\pi M_{\text{eff}}$ we can infer the change in perpendicular anisotropy field (H_u^\perp) with T_{ann} . We find slight increases in H_u^\perp in films where some long-range order (LRO) has been induced. Perpendicular FMR measurements on specimens annealed above 325°C show the presence of resonant absorption lines above the uniform FMR mainline. These absorptions have been attributed to magnetic nonuniformities near the interfacial regions.⁶

Coercive field values (H_c) for all of the annealed specimens are presented in Fig. 1(b) normalized to their as-deposited values. Decreases in easy-axis H_c begin at 150°C . This correlates with full easy-axis rotation during annealing in a magnetic field.¹ Significant increases in H_c are observed in specimens annealed at 350°C and 375°C , and are attributed to domain-wall pinning by precipitated crystallites.

In-plane uniaxial anisotropy field (H_a) was measured using both a VSM technique³ and a FMR technique. Results for these two techniques were found to agree for all specimens annealed below 325°C . Figure 1(c) illustrates the H_a values determined by FMR after annealing, normalized to the as-deposited value of 22.6 ± 1.3 Oe. Anisotropy values are observed to decrease immediately upon annealing, attaining a minimum of 4 Oe at 150°C . Specimens annealed at higher temperatures show increase in H_a .

Fourier transformed data of EXAFS spectra for the Co

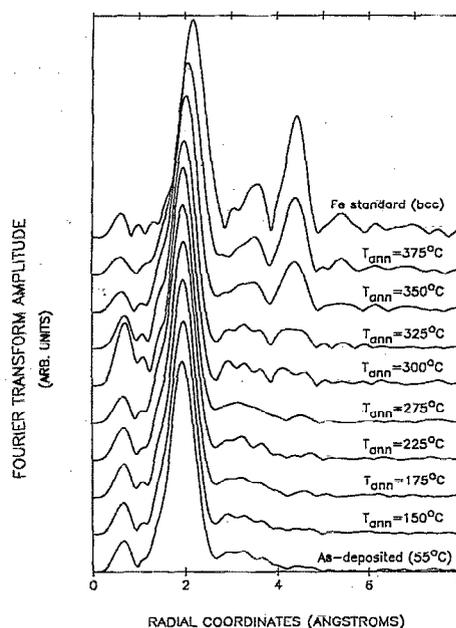


FIG. 2. Fourier transforms of EXAFS spectra of Co in annealed specimens (scaled and shifted vertically to allow direct comparison). The Fourier transform of the bcc Fe standard is presented for comparison.

ion in the as-deposited and annealed specimens are presented in Fig. 2. The Fourier transform of the Fe ion in the polycrystalline Fe standard (upper curve) is presented for qualitative comparison. Although this is useful for recognizing structural trends, discrepancies will exist in quantitative comparisons arising from differences in the electron phase shift of the two ions. The Fourier transform of the as-deposited specimen is dominated by a large peak centered at 1.96 \AA arising from nearest-neighbor (NN) atom scattering. The absence of peaks appearing above the signal-to-noise ratio at radial distances greater than this NN peak indicates the lack of an ordered atomic lattice. Comparing this transform to the transforms of annealed specimens it is apparent that only subtle structural change occurs in specimens annealed at temperatures below 275°C . However, the transform of the specimen annealed at 300°C illustrates development of higher-order peaks above signal-to-noise over the range of $4\text{--}5 \text{ \AA}$. This split peak is observed to form a single peak in specimens annealed at higher temperatures ($T_{\text{ann}} > 350^\circ\text{C}$). The transforms of specimens annealed at 350°C and 375°C closely match the Fourier peaks above 3 \AA in the bcc profile of the Fe standard. This behavior is also observed in Fourier transformed data of Fe ions in these specimens. Concurrent with the development of higher-order atomic peaks is an increase in NN Fourier peak distance. This NN expansion is also observed in Fourier transforms of the Fe ion EXAFS data. Room-temperature electrical resistivity values correlate closely with the development of LRO observed in Fourier transforms. In particular, values are measured to decrease linearly with increased T_{ann} for samples annealed at $T_{\text{ann}} \geq 300^\circ\text{C}$.

IV. DISCUSSION

The structural information deduced from Fourier transforms of the EXAFS spectra of annealed specimens (see Fig. 2) will be addressed as three regions: region I, $T_{\text{ann}} < 275^\circ\text{C}$; and region II, $300^\circ\text{C} < T_{\text{ann}} < 325^\circ\text{C}$; region III, $T_{\text{ann}} > 350^\circ\text{C}$.

Over region I the NN distance remains constant and there is no evidence of higher atomic shell ordering. Any structural changes occurring over this region are subtle and not of a long-range order. Correspondingly, values of H_c and H_a are largely dominated by structural and magnetic relaxation in the film. Specifically, H_a values are observed to decrease immediately as a result of heat treatment, attaining a minimum of 4 Oe at 150°C . Coercive field values begin to decrease at this same temperature. This temperature was determined from an earlier study to correspond to a state of magnetic relaxation sufficient to promote full easy-axis rotation during annealing with an externally applied field parallel to the hard axis.¹ Decreases in H_c are believed to result from enhanced domain-wall mobility initially allowed for by stress relief, and at higher temperatures, by the annihilation of trapped volumes which are incurred in the film during processing. Values of H_a are measured to increase after reaching a minimum value at 150°C . The origins of this behavior are believed to be due to increased magnetic ordering induced by magnetic field annealing. Values of $4\pi M_{\text{sat}}$ are seen to increase continuously from the as-deposited value with increased annealing temperature. Because NN distance over this range remains unchanged these increases are attributed to changes in exchange interaction between NN magnetic ions fostered by increased NN shell ordering. Further analysis of the EXAFS data using the ratio method and/or nonlinear fitting⁵ may provide quantitative information about this ordering.

Region II encompasses the annealing range of $300^\circ\text{C} < T_{\text{ann}} < 325^\circ\text{C}$. The Fourier transforms of EXAFS spectra corresponding to these specimens show evidence of ordering in a small volume of the film. Precipitation of crystallites at this low temperature [$T_x \approx 400^\circ\text{C}$ (Ref. 1)] is unusual. The structural changes observed to occur in this region are supported by perpendicular field FMR measurements which display extraneous absorptions above the uniform FMR mainline. Such behavior has been attributed to magnetic nonuniformities near the interfacial regions, such as variations in local anisotropy fields and/or magnetization.⁶ We attribute this behavior in our specimens to nucleation and growth of crystal phases at the free surface. Supporting this hypothesis are significant decreases in electrical resistivities first observed in specimens annealed at 300°C . This is presumably due to the decreased scattering of valence electrons in the crystalline regions which have formed at this temperature. Continued decreases in H_c indicate that the atomic ordering which occurs in this region is insufficient to hinder domain-wall mobility.

Specimens annealed at temperatures greater than 325°C are designated to be in region III. In this region higher-order peaks in Fourier transformed data most closely match those of a body-centered structure and indi-

cate ordering over an extended region of the film. Previous investigation of amorphous-to-crystalline transition of Fe-Co-B metallic glasses [$(\text{Fe}_{100-x}\text{Co}_x)_{83}\text{B}_{17}$ as ribbons] found crystallization products to consist predominantly of TM borides and Co-Fe solid solutions of varying composition and structure [$0 < x < 84$, bcc; $76 < x < 96$, fcc; $x > 96$, hcp (Ref. 7)]. The appearance of the bcc signature in Fourier transforms of specimens over this temperature range can be envisioned if Co borides precipitate from the amorphous matrix leaving behind the proper ratio of Co to Fe to form a bcc Co-Fe solid solution.

Concurrent with the upper values of these Fourier peaks, FMR spectra indicate gross magnetic nonuniformities and H_c values undergo large increases. In addition, H_a values undergo relative decreases due to the cumulative effects of magnetocrystalline anisotropy contributions in a polycrystalline-amorphous matrix.

V. CONCLUSIONS

We present magnetic and local structural properties of annealed, originally amorphous, $\text{Co}_{74}\text{Fe}_6\text{B}_{15}\text{Si}_5$ thin-film specimens. Electron-yield EXAFS analysis of annealed specimens indicate the onset of LRO near 300°C . This ordering is observed to evolve to a body-centered structure concurrent with increases in NN distance. Corresponding to the onset of LRO in specimens annealed at $T_{\text{ann}} > 325^\circ\text{C}$ is deterioration of soft magnetic properties, most notably, large increases coercive field and FMR linewidth. Progressive increases in $4\pi M_{\text{sat}}$ values are observed prior to the onset of ordering and are attributed to changes in the exchange interaction between NN magnetic ions fostered by an increase in NN ordering.

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