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Structural and magnetic characterization of amorphous Gd₂Fe₁₄B thin films

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Thin amorphous films of nominal composition Gd₂Fe₁₄B were fabricated on Si (100) substrates using ion-beam sputtering for the purpose of establishing their structural and magnetic properties at room temperature. X-ray-diffraction scans performed on as-deposited and annealed films revealed an amorphous structure. The as-deposited saturation magnetization was found to be 8101 G at room temperature suggesting ferrimagnetic ordering with anti-parallel coupling similar to other heavy rare earth-transition metal systems. Results from in-plane and perpendicular ferromagnetic resonance experiments conducted at 9.108 GHz showed the films to be inhomogeneous. This was evident from the presence of two in-plane resonance lines and spin-wave spectra that were not quadratic. Annealing at 300 °C for 30 min significantly reduced the absorption of the second in-plane resonance and restored classical n^2 law spin-wave behavior. It is speculated that the origin of the inhomogeneities is due to the kinetics of the deposition process where the increased mobility of the condensing Fe atoms leads to interspersed amorphous regions which are rich in Fe and Gd, respectively.

I. INTRODUCTION

The discovery of the RE₂Fe₁₄B system in 1984 (where RE=rare earth) has led to a considerable amount of research into the magnetic properties of this new class of permanent magnets.^{1,2} Although studies have been conducted on bulk crystalline and amorphous materials,³ to the best of our knowledge, none have specifically addressed sputtered amorphous thin films because the primary focus was on recrystallization of Nd₂Fe₁₄B permanent magnets.^{4,5} Since the amorphous state displays its own unique behavior, we present results on thin films sputtered from stoichiometric Gd₂Fe₁₄B sintered targets for the purpose of studying amorphous and gently annealed structures. As Gd is an *S* state ion, it is expected that this alloy will yield narrow ferromagnetic resonance (FMR) linewidths and multiple spin waves allowing for characterization using FMR techniques.

II. EXPERIMENTAL APPROACH

Thin films were sputtered from a stoichiometric Gd₂Fe₁₄B sintered target using a 5-cm diam ion beam operating at 1 keV. The working gas was gettered argon and the vacuum was maintained at 10⁻⁴ Torr during sputtering. Films were deposited onto Si (100) substrates at room temperature after first baking the chamber to achieve a base vacuum at 3 × 10⁻⁸ Torr. Films of thickness 223 nm were deposited at a rate of 0.1 nm/s and coated with approximately 10 nm of germanium for oxidation protection. After being fractured from the Si wafer, selected specimens were annealed in lots of 100, 200, and 300 °C, in gettered argon.

Structural characterization was undertaken using glancing incidence angle x-ray diffraction (GIA-XRD).

Films were analyzed using a 7.5 kW CuK α source with the incident x-ray flux oriented at a 4.5° glancing angle. As-deposited and annealed specimens were examined to confirm the presence of the amorphous state and to monitor for recrystallization. Static magnetic properties were evaluated using a vibrating sample magnetometer (VSM) with the applied field oriented parallel and perpendicular to the film surface. Saturation magnetization tests were conducted using the in-plane configuration with saturation fields of 10 000 Oe. FMR was conducted at 9.108 GHz using a TE₁₀₂ resonant cavity. Tests were performed with the applied field parallel and perpendicular to the film plane. The effective *g* factor g_{eff} and uniaxial perpendicular anisotropy constant K_u^1 were determined using the resonance equations for thin magnetic films,

$$\omega^2/\gamma^2 = (H_r^{\parallel} + 4\pi M - H_A^{\perp})(H_r^{\parallel}) \quad (1)$$

and

$$\omega/\gamma = H_r^{\perp} - 4\pi M + H_A^{\perp}, \quad (2)$$

where H_A^{\perp} and γ are the perpendicular anisotropy field and gyromagnetic ratio, respectively, and H_r^{\parallel} and H_r^{\perp} are the resonant fields when the applied field is applied parallel and perpendicular to the film plane, respectively. Use of Eqs. (1) and (2) allows for calculation of g_{eff} and K_u^1 , where $g_{\text{eff}} = \gamma\hbar/\beta$ (β and \hbar are the Bohr magneton and Planck's constant, respectively), and $H_A^{\perp} = 2K_u^1/M$. The presence of multiple spin waves in the absorption spectra (with the field oriented perpendicular to the film plane), allowed for the calculation of the exchange stiffness constant *A*, using the resonance equation

$$\frac{\omega}{\gamma} = H_r^{\perp} - 4\pi M_{\text{eff}} + \frac{2A}{M} \left(\frac{\pi}{t} \right)^2 n^2, \quad (3)$$

where $4\pi M_{\text{eff}} = 4\pi M - H_A^{\perp}$, *t* is the film thickness, and *n* is the spin wave order number.

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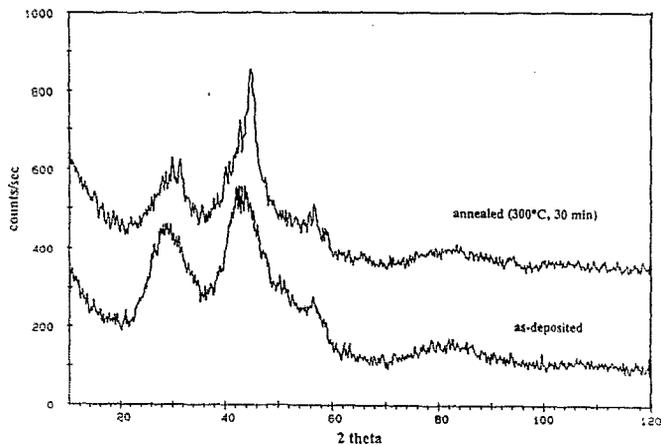


FIG. 1. GIA-XRD spectra for $Gd_2Fe_{14}B$ thin films ($\theta=4.5^\circ$, $t=223$ nm, 7.5 kW).

III. RESULTS

The results of the GIA-XRD scans from the as-deposited and annealed specimens suggest that the films are amorphous (Fig. 1). This conclusion is made after noting the presence of broad peaks (typical of amorphous materials), and the absence of any substantial diffraction peaks. Although there is some evidence of ordering for the specimen annealed at 300 °C, (i.e., the presence of a small peak at $2\theta=44.7^\circ$), it is felt that this reflects only the onset of recrystallization. To identify this peak, an as-deposited film was annealed at 500 °C for 10 min to promote recrystallization. The subsequent x-ray spectra revealed peaks that were indexed to α -Fe and the α -Fe (110) interplanar spacing was found to correspond to the small peak in Fig. 1.

Hysteresis loops with the applied field taken along the in-plane easy axis revealed the films to be magnetically soft (Fig. 2). Coercive fields for the as-deposited and annealed specimens were 3.8 and 5.0 Oe, respectively. Confirmation that the materials are ferrimagnetically ordered may be

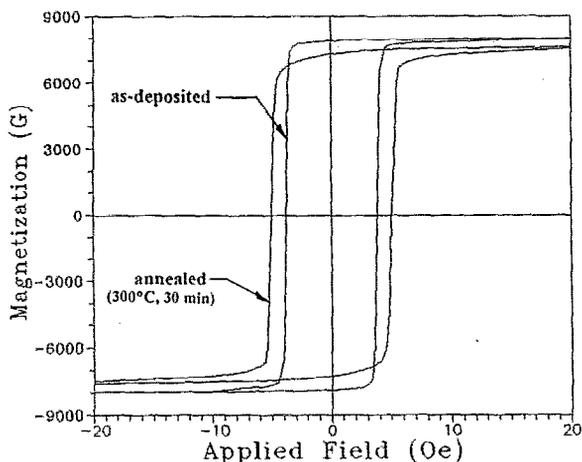


FIG. 2. In-plane hysteresis loops with H_{ex} parallel with the easy axis (RT).

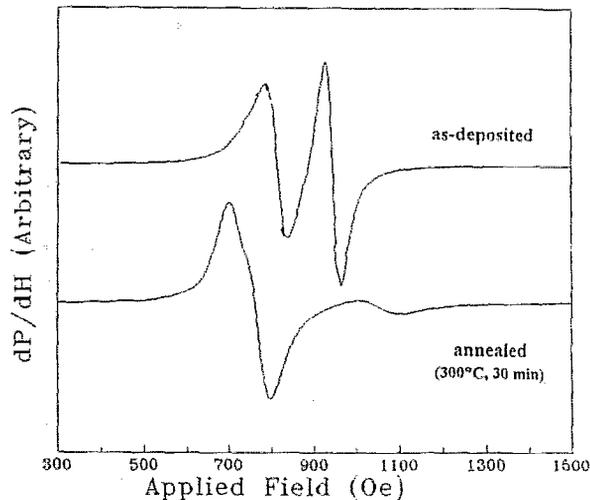


FIG. 3. In-plane FMR spectra at 9.108 GHz at RT showing the presence of two resonances.

inferred from the magnitude of the saturation magnetization (8101 G at room temperature), which compares with values extrapolated for the amorphous Gd-Fe system below the compensation composition.⁶ The behavior of this system is thought to be similar to rare-earth-transition-metal (RE-TM) systems where the magnetic sublattices couple parallel and antiparallel for the light and heavy RE's, respectively.⁷ The saturation magnetization and Curie temperatures showed no significant changes with annealing. The Curie temperature was determined to be 590 K, and $4\pi M_s$ showed no sign of compensation behavior in the range of 100–650 K.

FMR spectra obtained at 9.108 GHz with the applied field aligned parallel to the film plane revealed the presence of two resonances (Fig. 3), where the linewidths ΔH_r of the first and second resonance were 50 and 37.5 Oe, respectively. Annealing at 300 °C significantly reduced the amplitude of the second resonance and increased the linewidths to 95 and 93 Oe, respectively. The linewidths derived from the perpendicular resonance spectra were 43 and 40 Oe for the as-deposited and 300 °C specimens, respectively.

The perpendicular FMR spectra obtained at room temperature showed the presence of spin waves for both the as-deposited and 300 °C annealed specimens (Fig. 4). Using Eqs. (1) and (2), and $4\pi M_s$ from the VSM results, K_u^{\perp} and g_{eff} were calculated using data similar to that shown in Figs. 4 and 5. Table I summarizes the results of those calculations. The presence of a second peak in the in-plane data (Fig. 3) allowed for recalculation of K_u^{\perp} and g_{eff} using the second resonance and the main line from the perpendicular data. Since the recalculated value for K_u^{\perp} remained mostly unchanged, only the recalculated values for g_{eff} are included in Table I.

Exchange stiffness constants were evaluated for the as-deposited and annealed specimens using Eq. 3 and the slope from the plots in Fig. 5. The main line was selected as $n=0$ and sequential lines were selected for $n=1,2,\dots$, etc.

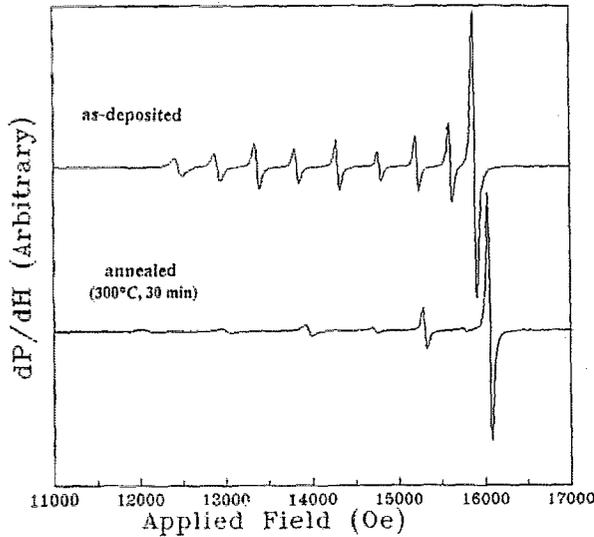


FIG. 4. Perpendicular FMR spectra at 9.108 GHz at RT showing the main line and spin-wave spectra.

Since the as-deposited spin wave spectra was not quadratic, resulting in a nonlinear plot, the slope was estimated using the spin wave spacing furthest from the main line. Values for A were determined to be 0.49×10^{-6} and 1.3×10^{-6} erg/cm for the as-deposited and 300 °C specimens, respectively.

IV. DISCUSSION AND CONCLUSIONS

As shown in Fig. 1, the as-deposited and annealed films were amorphous. As the work of Mimura *et al.* showed that sputtered Gd-Fe films with less than 20% Gd recrystallize during room-temperature deposition; the fact that these films are amorphous indicates the influence of the boron.⁸ The results from the FMR, analysis revealed negative values of K_u^1 indicating the presence of large in-plane anisotropy fields. Since results from in-plane angular variation tests, obtained using both VSM and FMR provided no evidence of preferred orientation, it is apparent that this anisotropy field acts on the magnetization in a manner similar to the demagnetizing field. To the best of

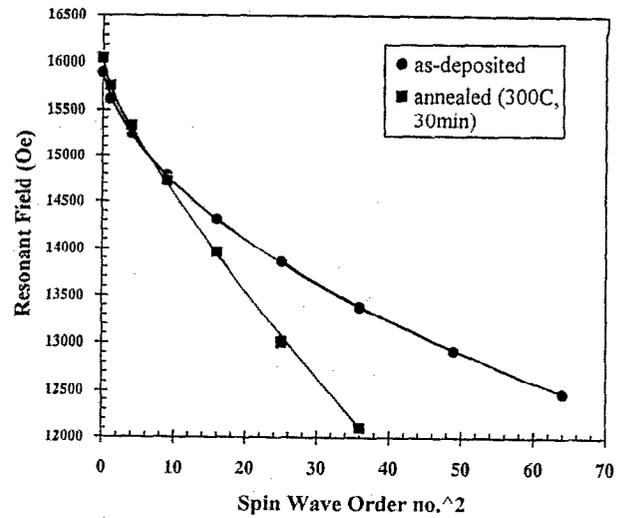


FIG. 5. Spin-wave resonant fields vs n^2 at RT.

our knowledge the magnitudes of K_u^1 are larger than any reported for Gd-Fe films. The effect of annealing was to increase K_u^1 .

The inhomogeneous nature of the films is evident from a variety of sources including: (i) the presence of an additional peak in the in-plane FMR spectra; (ii) the difference in linewidths between the in-plane and perpendicular orientation; (iii) the nonquadratic behavior of the spin-wave spectra; and (iv) the presence of two broad peaks in the x-ray data. The fact that gentle anneals reduced the magnitude of the second in-plane resonance peak and restored the quadratic nature of the spin wave spectra suggests that annealing resulted in internal reordering of the amorphous structure. The presence of two in-plane resonances, both of which yield distinct values of g_{eff} , suggest the presence of two interspersed amorphous "phases." It is speculated that these regions are a result of the kinetics of the deposition process, where the increased mobility of the condensing Fe atoms as compared to the Gd results in interspersed Fe- and Gd-rich amorphous regions with different local magnetic properties.

TABLE I. Magnetic parameters for $\text{Gd}_2\text{Fe}_{14}\text{B}$ films as determined from VSM and FMR data, and the resonance equations.

Specimens/ processing	Anisotropy field			Effective g factor	Effective g factor (2nd res. line)
	$4\pi M_s$ (25 °C) (G)	H_u^1 (Oe)	$K_u^1 \times 10^{-6}$ (erg/cm ³)		
FMR 105 as-deposited	8101	-4277	-1.38	2.00	1.87
FMR 106 100 °C, 30 min	8231	-3946	-1.29	2.02	1.88
FMR 107 200 °C, 30 min	8206	-4402	-1.44	2.01	1.84
FMR 108 300 °C, 30 min	7914	-4969	-1.56	1.97	1.70

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