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The role of iron in magnetic damping of $\text{Mg}(\text{Al,Fe})_2\text{O}_4$ spinel ferrite thin films

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We have investigated magnesium aluminum ferrite thin films with a range of iron concentrations and identified the optimal iron content to obtain high crystalline quality thin films with the low magnetic damping required for spin current-based applications. Epitaxial $\text{MgAl}_{2-x}\text{Fe}_x\text{O}_4$ films with $0.8 < x < 2.0$ were grown by pulsed laser deposition (PLD) on single crystal MgAl_2O_4 (MAO) substrates and were characterized structurally and magnetically. We find that the $x = 1.5$ composition minimizes the room-temperature magnetic damping with a typical Gilbert damping parameter of $\alpha_{eff} = 1.8 \times 10^{-3}$. This minimized damping is governed by a competition between the more robust magnetic ordering with increased iron content, x , and the more defective structure due to larger film-substrate lattice mismatch with increased iron content. The temperature-dependent magnetization curves indicate that T_c is suppressed below room temperature for iron content $x \leq 1.2$ and eventually suppressed entirely for $x = 0.8$. X-ray magnetic circular dichroism results indicate that for all x the magnetic moment is dominated by Fe^{3+} cations distributed in a 60:40 ratio on the octahedral and tetrahedral sites, with minimal contribution from Fe^{2+} cations. Films with $x = 1.4 - 1.6$ exhibit very strong ferromagnetic resonance and low Gilbert damping with $\alpha_{eff} = (1.8 - 6) \times 10^{-3}$, making them ideal candidates for microwave and spintronic applications.

Spinel ferrites have long been studied as more cost-effective alternatives to garnet ferrites for high-frequency microwave applications.[1–4] Of particular importance for pure spin current generation and above resonance circulators are materials with a narrow ferromagnetic resonance (FMR) linewidth and low Gilbert damping. [1, 5, 6] Garnet ferrites have been incorporated in heterostructures demonstrating spin current generation and spin current switching. [7–13] In addition, doping studies on $\text{Y}_3\text{Fe}_5\text{O}_{12}$ (YIG) and barium ferrite with various metallic cations have allowed for modulation of the structural and magnetic properties. These studies have shown favorable results for decreasing the resonance linewidth. [14–17] As an alternative to the garnet ferrites, we have recently reported doped nickel and magnesium ferrites with resonance linewidths as low as ≈ 5 Oe at 10 GHz and Gilbert damping parameters as low as $\alpha < 0.002$. [18–22] These previous studies have demonstrated both the viability of spinel ferrites as low-loss spin current sources and the versatility of the spinel crystal structure. We have also recently reported that the spinel ferrite $\text{MgAl}_{0.5}\text{Fe}_{1.5}\text{O}_4$ (MAFO) has a number of advantages over YIG, including lower external field requirements for magnetization precession and lower growth temperatures (450 °C as opposed to ≈ 750 °C for the garnet ferrites). Most recently, we have demonstrated successful spin pumping from spinel ferrites into heavy metals and isostructural integration with other spinel ferrites. [20, 22, 23]

To date, $\text{MgAl}_{0.5}\text{Fe}_{1.5}\text{O}_4$ is the most promising low-

loss magnetic insulator among epitaxial spinel ferrite thin films.[19–21] This particular composition of MAFO exhibits a Gilbert damping parameter as low as $\alpha \approx 0.0015$. In order to understand the role of iron stoichiometry on damping in magnesium aluminum ferrite films, we have studied the structure, composition, static magnetic, and ferromagnetic resonance properties of a series of $\text{MgAl}_{2-x}\text{Fe}_x\text{O}_4$ films where $x = 0.8 - 2.0$. As the composition varies from $x = 0.8$ to 2.0, the lattice mismatch with the MgAl_2O_4 substrate increases. Although all films exhibit epitaxy and are coherently strained to the substrate as evident from reciprocal space mapping, films with $x = 1.8$ and 2.0 show evidence of decreased film quality and increased mosaic spread. Temperature-dependent magnetization measurements show that films with $x \leq 1.2$ undergo magnetic transitions below room temperature with ferrimagnetic order completely suppressed for $x = 0.8$ in the measured temperature range. Room-temperature magnetic hysteresis loops show that films with $1.4 \leq x \leq 1.6$ exhibit coercivities $H_c < 0.5$ mT, with $x \geq 1.8$ films showing increased coercivities corresponding to the decreased film quality seen in XRD. X-ray magnetic circular dichroism (XMCD) indicated that in all compositions, the magnetism is dominated by Fe^{3+} cations. Finally, Gilbert damping parameters extracted from room temperature ferromagnetic resonance measurements range from $\alpha_{eff} = (1.8 - 6) \times 10^{-3}$ with the $x = 1.5$ composition yielding the lowest damping parameter. From these results we can conclude that $\text{MgAl}_{0.5}\text{Fe}_{1.5}\text{O}_4$ represents a compromise between a large

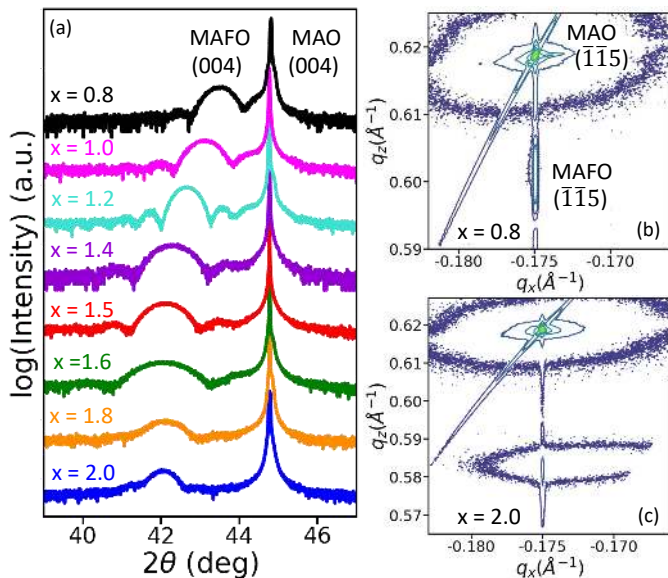


FIG. 1. (a) $2\theta - \omega$ XRD scans for 11 nm thick films with different iron contents showing clear Laue oscillations for $x \leq 1.6$. There is also a systematic increase in the out-of-plane lattice parameter as the iron content is increased up to $x = 1.6$. Labels correspond to the iron content. (b) RSM of the $(\bar{1}\bar{1}5)$ Bragg reflection of an $x = 0.8$ film showing that the film is coherently strained to the substrate. (c) RSM of the $(\bar{1}\bar{1}5)$ Bragg reflection of an $x = 2.0$ film, which is also coherently strained.

72 magnetic moment and a low concentration of lattice de-
73 fects to yield low damping for application in low-loss spin
74 current generation and microwave device applications.

75 Epitaxial thin films of MAFO were deposited on as-
76 received (001)-oriented single crystal MgAl_2O_4 (MAO)
77 substrates by pulsed laser deposition. A KrF 248nm ex-
78 cimer laser was incident on a pressed stoichiometric tar-
79 get of $\text{MgAl}_{2-x}\text{Fe}_x\text{O}_4$ for $x = 0.8, 1.0, 1.2, 1.4, 1.5, 1.6, 1.8$
80 and 2.0 with a fluence of $\approx 2 \text{ J/cm}^2$. The depositions
81 were performed at a substrate temperature of 450°C in
82 10 mTorr of O_2 . Deposition rates were calibrated via x-
83 ray reflectivity. All films are approximately 11 nm thick
84 as this was found to be the optimal thickness for the
85 lowest Gilbert damping in $x = 1.5$ films. [19, 21]

86 In order to structurally characterize our films, we per-
87 formed x-ray diffraction using a PANalytical X'Pert
88 reflectometer. Our results show that MAFO films are
89 epitaxial with the underlying MAO substrates and are of
90 high crystalline quality.

92 Figure 1(a) shows x-ray diffraction scans of MAFO
93 films with varying compositions. Laue oscillations are
94 observed around the (004) Bragg reflection for films with
95 $x < 1.6$, indicating excellent crystallinity. As the iron
96 content is increased, there is a monotonic increase (up
97 to $x = 1.6$) and then saturation in the out-of-plane lat-
98 tice constant as the films undergo a larger tetragonal
99 distortion to remain coherently strained to the MAO
100 substrate. Figure 1(b) shows a reciprocal space map

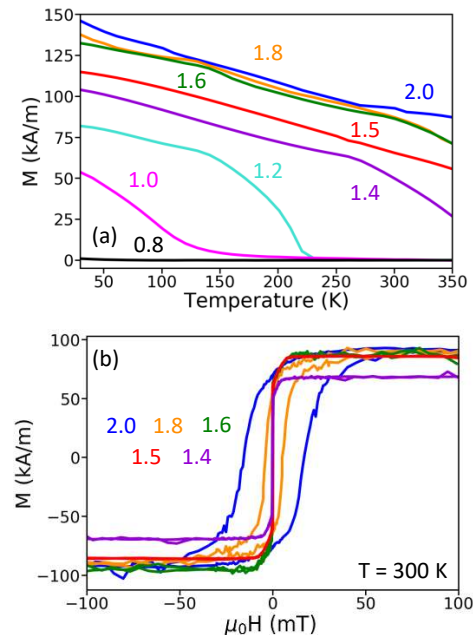


FIG. 2. (a) Magnetization measured as a function of temper-
ature. Films with $x = 1.2, 1.0$ undergo ferrimagnetic transi-
tions with $T_C = 225$ and 150 K , respectively. Composi-
tions where $x \leq 0.8$ do not become magnetic in the meas-
ured temperature range. (b) Room-temperature magnetic hysteresis
loops for the compositions that are magnetic at room tem-
perature.

101 (RSM) of an $x = 0.8$ film. The alignment of the film
102 and substrate peaks of the $(\bar{1}\bar{1}5)$ Bragg reflection in Fig-
103 ure 1(b) confirms that the film is coherently strained
104 on the substrate. Typical ω -rocking curves for films in
105 the $x \leq 1.6$ range have a full-width at half-maximum
106 of $\approx 0.045\text{-}0.060^\circ$, which is comparable to the substrate,
107 indicating low mosaic spread and high film crystallinity.

108 Structural quality of the films deteriorates as x in-
109 creases to 2.0 (MgFe_2O_4) and the lattice mismatch with
110 the underlying substrate increases. For $x = 1.8$ and
111 $x = 2.0$, the Laue oscillations disappear as the film qual-
112 ity degrades. Figure 1(c) shows an RSM of the $(\bar{1}\bar{1}5)$
113 Bragg reflection of an $x = 2.0$ film. While the film re-
114 mains coherently strained, the broader peak and the sur-
115 rounding “cloud” indicates there is a higher degree of
116 film mosaicity than in the lower compositions. This is
117 confirmed by the broader omega rocking curve with a
118 typical full-width at half-maximum of $\approx 0.085^\circ$ for the
119 $x = 1.8, 2.0$ films in contrast to the films with lower iron
120 content.

121 To characterize the static magnetic properties of our
122 films, the temperature and field dependence of the mag-
123 netization was measured using a Quantum Design Ever-
124 cool SQUID in the reciprocal sample option mode with
125 a sensitivity of $5 \times 10^{-12} \text{ Am}^2$. These measurements in-
126 dicate that increased iron content is correlated with the
127 Curie temperature as well as the saturation magnetiza-

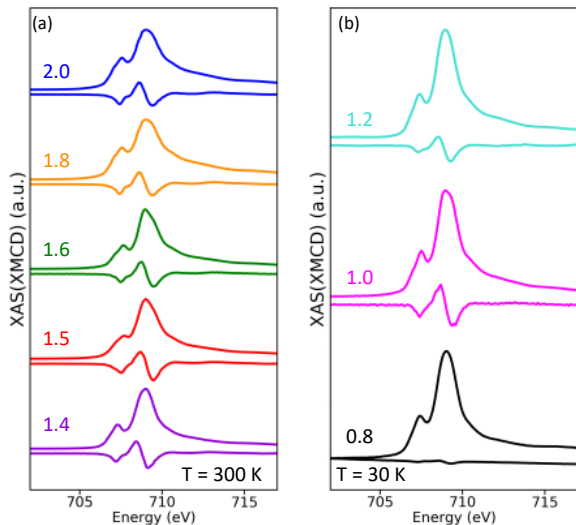


FIG. 3. (a) XAS and XMCD taken at room temperature. (b) XAS and XMCD taken at $T = 30$ K, below the T_C for $x = 1.2$ and 1.0.

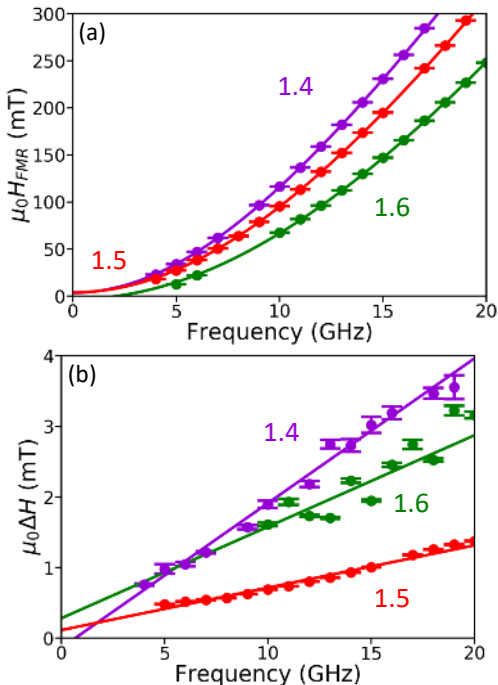


FIG. 4. (a) FMR resonant field as a function of frequency with Kittel equation fits (b) FMR linewidth as a function of frequency with fits to the linewidth equation.

tion.

Magnetization as a function of temperature is shown in Figure 2(a). Films with $1.4 < x < 2.0$ are ferrimagnetic at room temperature, and films with lower iron content of $x = 1.2$ and 1.0 undergo magnetic transitions with $T_C = 225$ and 125 K, respectively. There was no detectable magnetic signal in $x = 0.8$ films above the diamagnetic signal of the substrate.

A comparison of the magnetization values at low temperatures for the entire range of iron concentrations indicates that increased iron content increases the overall magnetization of the magnesium aluminum ferrite film. A similar trend is observed at room temperature for those samples that exhibit long range magnetic order as observed in the hysteresis loops taken along the [100] axis for $1.4 \leq x \leq 2.0$ films in Figure 2(b). Films with $1.4 \leq x \leq 1.6$ exhibit very low coercivity ($\mu_0 H_c < 0.5$ mT) and an increasing saturation magnetization with increasing iron content. Films with $x = 1.8$ and 2.0 show much higher coercivities ($\mu_0 H_c = 10$ and 20 mT, respectively) and are slower to saturate. This is likely due to defect pinning and frustrated magnetism attributed to poorer film quality and demonstrates the importance of coherent film growth in obtaining soft magnetism. [24, 25] The static magnetometry results indicate that there is an optimal window of the iron content in the $x = 1.4 - 1.6$ range where the films are ferrimagnetic at room temperature and exhibit soft magnetism that is ideal for spintronic applications such as pure spin current generation and spin-transfer torque.

In order to understand the origin of the soft magnetism in our samples with respect to iron valence and site distribution, we performed x-ray absorption spectroscopy (XAS) and x-ray magnetic circular dichroism (XMCD) at beamlines 6.3.1 and 4.0.2 at the Advanced Light Source at Lawrence Berkeley National Laboratory.

Measurements were performed in an applied field of 400 mT along the circularly polarized x-ray beam, incident at 30° grazing from the film plane. The spectra in Figure 3(a) were taken at room temperature for $1.4 < x < 2.0$, and at $T = 30$ K (below the T_C for $x = 1.2$ and $x = 1.0$) for $0.8 \leq x \leq 1.2$ in Figure 3(b).

The XAS and XMCD (defined as the difference between the + and - polarizations) spectra indicate that the magnetism in our films is derived from Fe^{3+} magnetic ions. In Figure 3, the peak at 709.2 eV is attributed to tetrahedrally coordinated Fe^{3+} and the peak at 710.0 eV to octahedrally coordinated Fe^{3+} [26, 27]. From the XMCD spectra, we note that the octahedrally coordinated Fe^{3+} ions align parallel to the field, while the tetrahedrally coordinated ions align antiparallel to the field. We have also compared our spectra to calculated spectra for Fe^{2+} and do not find that the inclusion of Fe^{2+} improves the fit to our measured XMCD spectra. [28] We note that measurements were performed in the total electron yield configuration, which is only sensitive to the top 5 nm of the sample. It is therefore possible that the bulk of the sample could contain a higher proportion of Fe^{2+} cations, however our ferromagnetic resonance measurements (see below) suggest that this is not the case. The predominance of Fe^{3+} and the absence of any signature of Fe^{2+} is particularly relevant for efficient spin current generation, because Fe^{3+} is an $L = 0$ ion that minimizes magnetic anisotropy and spin-orbit coupling. Experimental reference spectra can be used to determine the distribution of Fe^{3+} ions on the tetrahe-

TABLE I. Summary of Kittel equation and Gilbert damping parameters for the measured compositions.

x	g	$\mu_0 M_{eff}$ (T)	$ H_{4,\parallel} $ (mT)	$\alpha_{eff} (\times 10^{-3})$
1.4	2.12 ± 0.01	0.90 ± 0.02	9.11 ± 0.17	6.1 ± 0.2
1.5	2.08 ± 0.03	1.19 ± 0.04	8.44 ± 0.10	1.8 ± 0.01
1.6	2.12 ± 0.01	1.55 ± 0.03	8.43 ± 0.11	3.8 ± 0.8

194 dral (Fe_{Th}^{3+}) and octahedral (Fe_{Oh}^{3+}) sites. We can repro-
 195 duce our spectra using a combination of GaFeO_3 (100%
 196 Fe_{Oh}^{3+}) and $\gamma\text{-Fe}_2\text{O}_3$ (37.5% Fe_{Th}^{3+} , 62.5% Fe_{Oh}^{3+}) to give
 197 a 60:40 $\text{Fe}_{Oh}^{3+}:\text{Fe}_{Th}^{3+}$ ratio, as confirmed by our previous
 198 study. [19, 26, 27]

199 Previous dynamic magnetic characterization indicates
 200 that we can achieve low magnetic damping with a Gilbert
 201 damping parameter as low as <0.002 . [19–22] To char-
 202 acterize spin current generation efficiency, we perform
 203 broadband FMR measurements at room temperature in
 204 a coplanar waveguide setup. The resulting spectra were
 205 fit to the sum of the symmetric and antisymmetric com-
 206 ponents of the Lorentzian derivative, and the resonant
 207 field, H_{FMR} , and linewidth, ΔH , were extracted from
 208 the fit.

209 We find that, of the room-temperature ferrimagnets,
 210 only the $x = 1.4, 1.5$ and 1.6 compositions show strong
 211 FMR (high signal-to-noise ratio) in our setup. Figure
 212 4(a) shows the resonant field, H_{FMR} , as a function of
 213 frequency, f , for these compositions. In order to under-
 214 stand this trend, we analyze the frequency dependence
 215 of the FMR field in terms of the Kittel equation: [6]

$$f = \frac{g\mu_B}{h} \mu_0 [(H_{FMR} + H_{4,\parallel}) (H_{FMR} + M_{eff} + H_{4,\parallel})]^{1/2} \quad (1)$$

216 where g is the Landé g-factor, μ_0 is the permeability of
 217 free space, μ_B is the Bohr magneton, h is Planck's con-
 218 stant, $H_{4,\parallel}$ is the in-plane cubic anisotropy field, and
 219 M_{eff} is the effective magnetization that accounts for the
 220 out-of-plane uniaxial anisotropy field. The three Kittel
 221 equation parameters are summarized in Table 1. That
 222 $g \approx 2$ for all compositions implies low spin-orbit coupling,
 223 as to be expected from the magnetic contribution of Fe^{3+}
 224 ions discussed above. In addition, the effective magneti-
 225 zation, M_{eff} , increases as a function of increasing iron
 226 content since the saturation magnetization also increases
 227 in this composition range. $H_{4,\parallel}$ is approximately 8-9 mT
 228 for all measured compositions within experimental error,
 229 which is not so surprising given that the strain state (see
 230 Figure 1) of the films in this composition range does not
 231 vary significantly.

232 The figure of merit for magnetic damping is the Gilbert
 233 damping parameter which is minimized for the $x = 1.5$
 234 iron composition at <0.002 . The magnetic damping is
 235 deduced from the frequency dependence of the FMR
 236 linewidth for $x = 1.4, 1.5$, and 1.6 as shown in Figure
 237 4(b). Fitting the FMR linewidth ΔH as a function of
 238 frequency, f , to the linewidth equation yields the Gilbert

239 damping parameter, α_{eff} :

$$\Delta H = \Delta H_0 + \frac{h\alpha_{eff}}{g\mu_0\mu_B} f \quad (2)$$

240 where ΔH_0 is the zero-frequency linewidth. The Landé g
 241 factor is determined via the frequency dependence of the
 242 resonant field, as discussed above. From Table 1, we note
 243 that the $x = 1.5$ film composition minimizes the Gilbert
 244 damping parameter with $\alpha_{eff} = (1.8 \pm 0.01) \times 10^{-3}$, and
 245 thus is the best candidate for microwave device low-loss
 246 pure spin current generation applications.

247 According to our FMR results, the $x = 1.5$ composition
 248 of MAFO yields the lowest damping parameter. This can
 249 be understood from our structural characterization mea-
 250 surements, which indicate the film quality increases as
 251 the iron content decreases and the MAFO lattice param-
 252 eter more closely matches that of the MAO substrate. As
 253 the iron content is increased, films undergo more tetrag-
 254 onal distortion to remain coherently strained to the sub-
 255 strate, resulting in a larger mosaic spread for films with
 256 $x > 1.6$. Additionally, static magnetization at room tem-
 257 perature suggests that the softest magnetism is observed
 258 for $1.4 \leq x \leq 1.6$, which is critical for observing strong
 259 FMR signals. Therefore the $x = 1.5$ composition repre-
 260 sents an excellent compromise between high enough iron
 261 content for magnetic ordering well above room temper-
 262 ature and a low-enough lattice mismatch with the sub-
 263 strate to yield a high quality film with a low concentra-
 264 tion of defects. Our results highlight the interplay be-
 265 tween film quality, magnetic ordering, and cation chem-
 266 istry in obtaining films with low magnetic damping.

267 In summary, we have performed a systematic study
 268 of the role of iron content on the structural and mag-
 269 netic properties of epitaxial $\text{MgAl}_{2-x}\text{Fe}_x\text{O}_4$ films. As
 270 the iron content is increased we see a monotonic increase
 271 in the out-of-plane lattice parameter and mosaic spread.
 272 From static magnetometry, we measure an increase in the
 273 Curie temperature as well as the saturation magnetiza-
 274 tion at low temperatures as the iron content is increased.
 275 XAS and XMCD show that the magnetism is derived
 276 from Fe^{3+} magnetic moments with minimal spin-orbit
 277 coupling. Finally, ferromagnetic resonance studies indi-
 278 cate that the $x = 1.5$ composition gives the lowest Gilbert
 279 damping parameter due to a compromise between struc-
 280 tural quality and magnetic response. Our results indicate
 281 that the $\text{Mg}(\text{Al,Fe})_2\text{O}_4$ system is an excellent candidate
 282 for pure spin current sources, and demonstrates the ver-
 283 satility of the spinel crystal structure for obtaining a wide
 284 range of structural and magnetic properties.

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