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Effect of FeF₂ on magneto-optic response in FeF₂/Fe/FeF₂ sandwiches

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Abstract. The room temperature optical constants n and k of MBE grown FeF₂ films are reported. Because of poor chemical stability, FeF₂ had to be coated with a protective Au layer. Reflection spectral ellipsometry in the photon energy range between 1.3 and 5.2 eV was performed on structures with a typical profile Au(0.5 nm)/FeF₂(120 nm)/Au(30 nm)/Ag(20 nm)/Fe(0.6 nm) grown on GaAs(001) substrate. The spectra of n and k in FeF₂ were subsequently employed in the design of FeF₂/Fe/FeF₂ sandwiches considered as magneto-optic (MO) sensors for weak microwave currents. Their MO response was evaluated using reflection MO (Kerr) spectroscopy at polar magnetization. The present results may be of interest in MO studies of magnetic nanostructures with Fe/FeF₂/Fe, including MO magnetometry and MO magnetic domain imaging.

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1. Introduction

Interest in FeF₂ is motivated by exchange bias studies [1-4] since anti-ferromagnet FeF₂ has a strong uniaxial anisotropy that makes modeling of the exchange bias Fe/FeF₂/Fe nanostructures easier. The effect of exchange bias on the magnetic characteristics of Fe/FeF₂/Fe can be studied using fixed wavelength magneto-optic (MO) magnetometry in reflection. Chemical compatibility with Fe was found by Lopusnik et al. [5] to be favorable for magneto-optic (MO) reflection sensors based on FeF₂/Fe/FeF₂ sandwiches operating at a Ti: sapphire laser wavelength of 810 nm (1.53 eV). The sensors, which exploit strong MO activity in reflection of Fe in the infrared spectral region, were considered for the detection of weak magnetic fluxes up to microwave frequencies with submicron resolution. In the sandwich nanostructure, the MO response can be tuned to its maximum by a proper choice of the Fe layer thickness and that of the sandwiching FeF₂ layers. In the practical design, an Au layer protects the FeF₂/Fe/FeF₂ sandwich which is deposited on a reflecting Ag or Au layer. The entire structure is grown on a GaAs substrate with a Fe seed layer to form an epitaxial Ag or Au reflector.

An evaluation of the MO response in the Fe/FeF₂/Fe exchange bias and FeF₂/Fe/FeF₂ sensor structures requires the optical and MO constants for all layer media. These are available in the literature except those for ultrathin FeF₂ films. Their bulk counterpart, FeF₂ single crystals have rutile-type structure (D_{4h}) and become anti-ferromagnetic at temperatures $T < T_N = 78.4$ K [6]. Infrared optical absorption, magnetic circular dichroism (MCD), and light scattering studies of FeF₂ have focused on anti-ferromagnetic resonance [6], optical transitions with spin change [7, 8], light scattering by magnons [9], Raman optical activity [10], etc. Giordano et al. measured the infrared reflectivity spectrum at photon energies between 0.05 and 0.6 eV on an optically clear light yellow 4 mm thick FeF₂ single crystal [11]. McClure et al. [8] and Cheng et al. [9] reported two bands in the absorption spectrum: One centered near 2.67 eV and another stronger one centered near 3.20 eV. However, above the photon energy of 0.08 eV, no systematic information on optical spectra in FeF₂ is available.

The present work reports on the room temperature optical constants n and k of submicron FeF₂ films in the photon energy range between 1.3 and 5.2 eV (240-950 nm), determined from reflection spectroscopic ellipsometry (SE). The films were MBE grown on noble metal reflectors separated from GaAs (001) substrates by a seed Fe layer. A thin protecting Au layer was added on the top of the structure since the FeF₂ films were found chemically unstable when exposed to ambient humidity. The n and k extracted from the *ex situ* SE data were then used for the design of FeF₂/Fe/FeF₂ structures with an optimized MO response, in reflection, at polar magnetization, and normal light incidence, for a wavelength of 810 nm. The FeF₂/Fe/FeF₂ structures were characterized using MO reflection (Kerr) spectroscopy at polar magnetization. In the spectral range from 1.4 to 4.5 eV, the best theoretical fit to the MO experimental data was achieved by replacing the sharp Fe/FeF₂ interfaces with ≈ 2 nm thick transition layers. These transition layers were characterized by the optical and MO constants of an effective medium consisting of Fe (50%) and FeF₂ (50%).

2. Samples

The optical constants n and k in FeF₂ were computed from SE experiments using two structures denoted as Sample A and Sample B (fig. 1). Their composition profiles are listed in table 1.

Table 1. Samples for spectral ellipsometry.

	Au [nm]	FeF ₂ [nm]	Au[nm]	Ag[nm]	Fe[nm]	GaAs
A	0.5	120	30	20	0.6	Substrate
B	0.5	120	60	6.5	0.6	Substrate

Table 2. Multilayers with optimized MO response.

	Au [nm]	FeF ₂ [nm]	Effective medium [nm]	Fe [nm]	Effective medium [nm]	FeF ₂ [nm]	Ag [nm]	Fe [nm]	GaAs
C	0.5	2.5	-	8	-	50	50	0.6	Substrate
C1	0.3	3	-	8.5	-	63	50	0.6	Substrate
C2	0.3	1	2	6.8	2	58	50	0.6	Substrate
D	0.5	2.5	-	8	-	70	50	0.6	Substrate
D1	0.3	3	-	9	-	98	50	0.6	Substrate
D2	0.3	1	2	7.3	2	90	50	0.6	Substrate

The n and k of FeF₂ extracted from the SE data on samples A and B were used in the design of Au/FeF₂/Fe/FeF₂/Ag/Fe/GaAs multilayers, samples C and D, with an optimized MO response at the desired wavelengths. The structures of samples C and D are shown in figure 2, while individual layer thicknesses for samples C and D are listed in table 2. The details of the sample preparation were given in the previous paper [5]. We would like to point out that fluorides on metallic templates frequently grow in columnar fashion, and may form rough interfaces between Fe/FeF₂ [12, 13].

Figure 1. Structure of FeF₂ sandwiches.

Figure 2. Structure of $\text{FeF}_2/\text{Fe}/\text{FeF}_2$ multilayers: abrupt interfaces (a) and effective medium model with transition interface layers (b).

3. Spectroscopic ellipsometry of FeF_2

The $\text{Au}/\text{FeF}_2/\text{Au}/\text{Ag}/\text{Fe}/\text{GaAs}$ sandwiches were characterized using a UVISSEL spectroscopic ellipsometer (Horiba Jobin Yvon) with the optical element sequence PSMA (Polarizer-Sample-Modulator-Analyzer). The phase of the probing polarized monochromatic beam is modulated using a photoelastic modulator (PEM), and the resulting AC photoelectronic signal at the photodetector output is converted to DC using a lock-in amplifier. The spectra were measured at photon energies ranging from 1.3 to 5.2 eV (from 240 to 960 nm) in 0.02 eV steps, and at the incidence angles of 60° , 70° and 75° . Two azimuth setting for the PEM, $M = 0^\circ$ and $M = 45^\circ$, had to be used to acquire unique values of the ellipsometric angles ψ and Δ . Figure 3 shows the experimental and modeled ψ and Δ for Sample A at the incidence angle of 70° . Sample B produced similar results, deviating by less than 1% from Sample A, and thus only Sample A is shown and discussed for simplicity. The modeling procedure is described next.

The analytic complex relative scalar permittivity in the polycrystalline FeF_2 layer, $\epsilon(E)$, was computed using SCOUT software for optical spectroscopy [14]. The Kim extended oscillator model [15], developed from the work by Kim et al. [16], was chosen as the most appropriate for FeF_2 . In the model, $\epsilon(E)$, in the energy range of 1-6 eV, can conveniently be expressed in terms of a sum of three oscillators and a constant term (ϵ_∞), i.e.

$$\epsilon(E) = \epsilon_\infty + \sum_{i=1}^3 \chi_e^{(i)}(E) \quad (1)$$

where the i -th component of electric susceptibility is given by

$$\chi_e^{(i)}(E) = \frac{E_p^{(i)2}}{E_0^{(i)2} - E^2 - i\Gamma^{(i)}(E)E} \quad (2)$$

Figure 3. Measured and modeled spectra of the ellipsometric parameters, ψ and Δ , in Sample A at an incidence angle of 70° .

Here $E_0^{(i)}$ denotes the resonance photon energy and $E_p^{(i)}$ accounts for the oscillator strength. The damping, $\Gamma^{(i)}(E)$, is assumed to be a function of the photon energy, E , and is given by

$$\Gamma^{(i)}(E) = \Omega_r^{(i)} \exp \left[-\frac{1}{1 + \sigma^{(i)2}} \left(\frac{E - E_0^{(i)}}{\Omega_r^{(i)}} \right)^2 \right] \quad (3)$$

where $\Omega_r^{(i)}$ is the damping constant. The parameter $\sigma^{(i)}$, the so-called Gauss-Lorentz switch, is allowed to vary between 0 and ∞ . The value $\sigma^{(i)} = 0$ corresponds to a Gaussian line shape, while for $\sigma^{(i)} > 5$ the profile is considered Lorentzian. The contribution to $\epsilon(E)$ originating from the spectral range above the maximal $E_0^{(i)}$ is approximated by a constant background term, $\epsilon_\infty - 1$. The model has been successfully applied to a broad class of metals [17], semiconductors, and dielectrics [18].

For CaF_2 and SrF_2 , both transparent fluorides, $\epsilon(E)$ between 1 and 6 eV can be approximated with a single Lorentz oscillator term with $E_0^{(i)} \approx 15.5$ eV assigned to electric dipole allowed inter-band transitions [19, 20]. In FeF_2 , there are also contributions to $\epsilon(E)$ due to $3d$ electrons of Fe^{2+} . The modeling process started with six fitting parameters, i.e., the optical constants of FeF_2 and the thicknesses of the four topmost layers $\text{Au/FeF}_2/\text{Au/Ag}$ with a fixed thickness of the Fe seed layer. Fixed parameters included the Lorentz oscillator at $E_0^{(3)} \approx 15$ eV, and literature values for the optical constants of Au, Ag, Fe and GaAs [21-23]. The FeF_2 layer thickness, $t=118$ nm, deduced from modeling, was reasonably close to $t=120$ nm measured with a thickness monitor during growth. Using the modeled t and $\epsilon(E)$, SCOUT software provided ψ and Δ consistent with the experiment, as shown in figure 3. The parameters entering Eq. (1) for $\epsilon(E)$ are collected in table 3. The procedure provided $\sigma^{(i)}$ at the resonance photon energies for the first ($E_0^{(1)} \approx 3.409$ eV) and second ($E_0^{(2)} \approx 5.049$ eV) oscillators of 0.119 and 0.841, respectively, corresponding to Gaussian profiles. The exact position of $E_0^{(3)}$ has a negligible effect on the parameters of the first and second oscillators, and consequently on the permittivity in the spectral region of interest ($E < 6$ eV). For example, a shift of $E_0^{(3)}$ to 15.5 eV ($E_p^{(3)} \approx 20$ eV) modified the permittivity by less than 0.1%. The large separation between the strong allowed transition centered at $E_0^{(3)}$ and the transitions involving $3d$ electrons, centered at $E_0^{(1)}$ and $E_0^{(2)}$ is typical for fluorides [24].

Figure 4. Real (a) and imaginary (b) parts of the complex relative permittivity in FeF_2 as a function of photon energy, E , determined from spectral ellipsometry on Sample A.

Table 3. Parameters entering the analytic expression for the dielectric function with a constant background term, $\epsilon_\infty - 1 = 0.612$.

	$E_0^{(i)}$ [eV]	$E_p^{(i)}$ [eV]	$\Omega_f^{(i)}$ [eV]	$\sigma^{(i)}$
$\chi_e^{(1)}$	3.409	1.206	1.328	0.119
$\chi_e^{(2)}$	5.049	2.143	1.433	0.841
$\chi_e^{(3)}$	15.000	20.000	0.25	>5

Figure 4 displays the dielectric function, i.e., the complex relative permittivity, $\epsilon(E)$, in FeF_2 as a function of photon energy, E . The real index of refraction (n) and the extinction coefficient (k) in FeF_2 as a function of wavelength (λ) are collected in table 4. The corresponding absorption coefficient, $\alpha = 4\pi k / \lambda$ in FeF_2 as a function photon energy, E , is displayed in figure 5.

Figure 5. Absorption coefficient, $\alpha = 4\pi k / \lambda$, of FeF_2 as a function of photon energy, E , determined from ellipsometry measurements on Sample A.

Table 4. Optical constants n and k of polycrystalline FeF_2 as a function of wavelength, λ .

λ [nm]	n	k	λ [nm]	n	k
235	1.84375	0.15733	400	1.97849	0.08298
245	1.88023	0.17467	425	1.98678	0.06481
255	1.91734	0.17021	450	1.98721	0.04764
265	1.94265	0.15116	475	1.98325	0.03436
275	1.95361	0.12881	500	1.97771	0.02494
285	1.95434	0.11045	525	1.97206	0.01842
295	1.95006	0.09844	550	1.96686	0.0139
305	1.94467	0.09238	575	1.96229	0.01073
315	1.94053	0.09074	600	1.95832	0.00846
325	1.93876	0.09183	700	1.94712	0.00392
335	1.93965	0.09411	800	1.94051	0.00227
345	1.94301	0.09636	900	1.93629	0.00153
350	1.94547	0.09719	1000	1.93341	0.00114
375	1.9623	0.0955			

In the transparent fluorides CaF_2 and SrF_2 , $k \approx 0$, and n varies between 1.42 (1.5 eV) and 1.48 (5.0 eV) [19, 20]. The higher values of n , non-zero k and α in FeF_2 (figures 4 and 5), originate from electron transitions involving $3d$ levels of Fe^{2+} ions in octahedral coordination [24, 25].

4. MO spectroscopy of FeF₂/Fe/FeF₂

The use of reflection MO (Kerr) spectroscopy for the characterization of FeF₂/Fe/FeF₂ multilayers is illustrated on Samples C and D. MO Kerr rotation and ellipticity spectra at polar magnetization and nearly normal incidence were measured in the photon energy range from 1.3 to 4.5 eV (275-950 nm). The optical element sequence in the set-up consisted of 450W Xe arc lamp – monochromator – polarizer (0°) – calibrated DC Faraday rotator – AC Faraday rotator – calibrated phase plate (0° or 90°) – sample – analyzer (90°) – photomultiplier. Samples were subjected to a magnetic field of ± 0.3 Tesla perpendicular to their surface (polar magnetization, $\pm \mathbf{M}$). This field is about seven times smaller than the field required to saturate thin iron films (2.1 Tesla) out of plane. The rotation spectra were determined with the phase plate removed, i.e. at zero phase retardation, from the current difference in the DC Faraday rotator at $\pm \mathbf{M}$. The procedure was repeated with the phase plate of a known retardation, δ , inserted into the sequence. The ellipticity was deduced from the compensation currents in the DC Faraday rotator at zero and δ retardations. Figure 6 shows the MO polar Kerr rotation and ellipticity spectra in Samples C and D. The spectra display maxima in the MO response near 1.5 eV, as required for the sensor operation. The structure centered near 3.9 eV, more distinct in Sample D, is due to the shifted plasma resonance in Ag [26]. The spectra were simulated with the help of a multilayer model with abrupt interfaces [27], schematically shown in figure 2a, using the optical constants of FeF₂ from table 4. For the other layer media, the literature values were used [21-23, 28, 29]. The thicknesses of the four topmost layers (Au/FeF₂/Fe/FeF₂) were allowed to vary. The results are included in table 2 as entries C1 and D1. The model correctly reproduced both the position of the maxima in MO response at 1.53 eV, trends in the spectra, and provided refined thicknesses for the layers. However, the thickness values for the bottom FeF₂ layer were significantly higher compared to those evaluated during the growth. The abrupt interface model, however, ignores the roughness at FeF₂/Fe interfaces. To account for this effect, a more realistic model with transition layers at the Fe/FeF₂ interfaces, schematically shown in figure 2b, was employed. The addition of transition layers at both Fe/FeF₂ interfaces, with a total thickness of 4 nm, and characterized with the optical and MO constants of an effective medium [30] consisting of Fe (50%) and FeF₂ (50%), improved agreement between the thickness values evaluated during the growth (entries C and D in table 2) and the modeling (entries C2 and D2 in table 2). In the model with transition layers, the MO maxima near 1.53 eV are shifted by +0.03 eV with respect to those obtained from the model with abrupt interfaces. Up to this small shift, the MO spectra computed with these models are practically the same. Only the spectra for the effective medium model with transition layers are therefore included in figure 6. Two transition layers sandwiching Fe of equal thickness (2 nm) were considered but the model was found insensitive to thickness redistribution between the two Fe/FeF₂ interfaces, provided that the total transition layer thickness was kept at 4 nm.

Figure 6. Magneto-optical Kerr rotation (circles) and ellipticity (triangles) spectra as a function of photon energy, E , measured at polar magnetization ± 0.3 Tesla in Samples C and D. The computed spectra are represented with solid lines.

5. Summary

The paper reports the room temperature permittivity, $\epsilon(E)$, of polycrystalline submicron FeF_2 films deduced from spectroscopic ellipsometry on sandwiches at photon energies, E , between 1.3 and 5.2 eV. The $\epsilon(E)$ spectra display features typical for a dielectric 3d compound with a high density of magnetic cations, and can be conveniently expressed analytically as a sum of three physically reasonable oscillators. The two lower oscillators correspond to 3d electron transitions involving Fe^{2+} pairs coupled via F^- , the mechanism typical for other compounds with high density magnetic cations coupled by superexchange [31]. Their contributions to $\epsilon(E)$ are superimposed on a wing of an allowed electric dipole transition with a well separated resonance around 15 eV. A lower degree of covalency in FeF_2 results not only in much lower Néel temperature (T_N), but also in lower oscillator strengths of 3d electron transitions. This, in turn, results in a two orders in magnitude lower absorption coefficient (figure 5) compared to antiferromagnetic and ferrimagnetic oxides.

The $\epsilon(E)$ spectra enabled a more precise evaluation of the magneto-optic (MO) response in $\text{FeF}_2/\text{Fe}/\text{FeF}_2$ sandwiches considered for MO sensing of microwave current with submicron resolution. The measured maximum MO rotation at a 15% saturation of Fe reached 1 degree. MO studies of $\text{FeF}_2/\text{Fe}/\text{FeF}_2$ structures supported the expected presence of intermediate mix layers at the Fe/FeF₂ interfaces. Their contribution to MO response was approximated using effective medium theory. The information on $\epsilon(E)$ of FeF_2 (not available in the literature so far) will be useful in interface studies of layered systems with FeF_2 by means of MO magnetometry, MO magnetic domains imaging, MO spectroscopy, and spectroscopic ellipsometry. In particular, it may be used for selective sensing of Fe layers in exchange coupled Fe/FeF₂/Fe sandwiches. The optical constants of FeF_2 submicron films may serve as a guide to optical constants of bulk FeF_2 .

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