Broadband Electron Spin Resonance Study in a Sr$_2$FeMoO$_6$ Double Perovskite

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1. INTRODUCTION

Double perovskites with the general formula A$_2$BB′O$_6$ (A = divalent or trivalent cation; B and B′ = transition metals) are made up of perovskite blocks, where the transition metal ions are alternatively occupied by B and B′ ions. They show a number of interesting magnetic and electric properties arising from the band structure and a variety of magnetic interactions between B and B′ ions. $^{1-4}$ A$_2$FeMoO$_6$ shows ferromagnetic ordering near and above room temperature ($T_C = 420, 330$, and $320 \, K$ for $A = \text{Sr, Ba, and Ca}$, respectively). The discovery of giant magnetoresistance due to tunneling between ferromagnetic grains at room temperature and prediction of half-metallicity in Sr$_2$FeMoO$_6$ made this series of double perovskites more popular in recent years.$^{5-7}$ Although the origin of ferromagnetism was attributed to the ferromagnetic superexchange interaction between Fe$^{3+}$ (3$d^6$, $S = 2$) ions, with Mo$^{5+}$ (4$d^3$) playing no role in magnetism in earlier studies, later studies favored an interpretation in terms of the ferromagnetic interaction between Fe$^{3+}$ (3$d^5$, $S = 5/2$) mediated by the itinerant down-spin $t_{2g}$ electron of Mo$^{5+}$ (4$d^3$, $S = 1/2$) that couples antiferromagnetically with the up-spin $t_{2g}$ electrons of Fe$^{3+}$ ions.$^{8,9}$ Static (dc) magnetization alone does not distinguish between Fe$^{3+}$–Mo$^{5+}$ and Fe$^{3+}$–Mo$^{5+}$ combinations because both can give a saturation magnetization of 4 Bohr magneton/formula unit. Experiments such as electron spin resonance (ESR), Mossbauer spectroscopy, or X-ray magnetic circular dichroism (XMCD) can be used to probe valance states of the Fe ions.$^{10}$ Based on ESR investigations in the paramagnetic state over a wide temperature range ($T = 125–560 \, K$), Causa et al.$^{11}$ and Niebieskikwiat et al.$^{12}$ conclude that Fe exists in the trivalent state and Flores et al.$^{13}$ attributed the line width variation to the divalent state of Fe in Sr$_2$FeMoO$_6$. However, these ESR studies were done at a single frequency ($f = 9.2 \, GHz$) due to limitations of cavity resonance-based conventional ESR spectrometers.

Sr$_2$FeMoO$_6$ being ferrimagnetic at room temperature and highly spin-polarized, has potential for spintronic applications. Resonant absorption of microwaves at multiple frequencies is essential to extract damping parameters that determine the switching time of magnetization. Motivated by the absence of literature on the dynamic magnetic behavior of Sr$_2$FeMoO$_6$ and lack of ESR work below room temperature, we explore microwave absorption in this compound at different frequencies ($f = 2–18 \, GHz$) and over a wide temperature range ($T = 390–10 \, K$) using a coplanar waveguide-based spectrometer. This enables us to get an accurate estimation of the gyromagnetic ratio, $\gamma$, which is related to the spectroscopic $g$-factor. $\gamma = (g \mu_B / \hbar)$, where $\mu_B$ is the Bohr magneton, $\hbar$ is the reduced Planck’s constant, and $g$ is the spectroscopic splitting factor. 

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factor or Lande g-factor. An accurate determination of g is of paramount importance since it is an intrinsic property of the material related to the relative spin and orbital moments. One can also obtain information about spin–orbit interactions, relaxation mechanisms, and damping mechanisms originating from intrinsic or extrinsic considerations.

Here, we have observed that g is not independent throughout the temperature range; it is \( \sim 2.01 \) at \( T \geq T_C \) and decreases slowly, reaching \( \sim 1.2 \) at 10 K. Spin relaxation time or damping is found to be intrinsic Gilbert type at \( T \geq 300 \) K and at lower temperatures, extrinsic contributions dominate.

2. RESULTS AND DISCUSSION

Figure 1 shows the powder X-ray diffraction (XRD) pattern of the synthesized \( \text{Sr}_2\text{FeMoO}_6 \). All peaks can be assigned to the tetragonal crystal structure (space group I4/mmm). The inset shows order-related peak that appear at 2\( \theta \sim 19.5^\circ \). Thus, we have observed that an antiferromagnetic coupling between the Fe\(^{3+}\) and Mo\(^{5+}\) sites is present at \( T = 10 \) and 325 K. The inset of (a) shows the magnetic field dependence of \( M \) at 10 K and (b) \( \ln(\rho) \) vs 1/\( T \) plot. The solid line shows the fitted curves. (S = 5/2) and Mo (S = 1/2) sublattices induces a ferrimagnetic half-metallic state with saturation magnetization (\( M_S \)) of 4 \( \mu_B \). Thus, a perfect alternating order of Fe and Mo ions is needed to reach the saturation. In the present sample, the nonsaturation of magnetization might be related to the AS defect (Mo at the Fe site and vice versa), which alters the local interactions and favors antiferromagnetic (AFM) superexchange antisite interactions (Fe–O–FeMo or Mo–O–MoFe).

Several experimental and theoretical studies demonstrated the possible relation between the magnetic parameters (\( T_C \) and \( M_S \)) and the structural off-stoichiometry (i.e., Fe, Mo, and oxygen vacancies and AS disorder) in double perovskites. Alonso et al. elaborated that with an increase in the antisite defect density (\( y \)), \( M_s \) decreases at a rate of (4.0–7.7)\( \mu_B \), where \( y \) is the antisite concentration, but \( T_C \) tends to increase due to the direct AFM interaction between neighboring Fe ions. Similarly, oxygen nonstoichiometry influences the magnetic parameters \( M \) and \( T_C \) differently; an increase of oxygen vacancies in \( \text{Sr}_2\text{FeMoO}_6 \) lowers the magnetization value but increases the later.

Hoffmann et al. found that \( T_C \) increased at a rate of +15 K per atom % of oxygen vacancies.

Since magnetization is not saturated at 70 kOe, we can obtain an approximate value of \( M_s \) from the law of approach to saturation (LOAS) given by the equation

\[
M = M_s \left( 1 - \frac{a}{H} - \frac{b}{H^2} \right) + \chi H
\]

(1)

where the parameters \( a \) and \( b \) are related to the microstress and the first-order magnetocrystalline anisotropy coefficient (\( K_1 \)), respectively, and \( \chi \) represents forced magnetization in the system. The anisotropy field (\( H_K \)) can also be estimated from the fitted parameter \( b \) using the equations \( b = \left( \frac{8}{105} \right) \times \left( \frac{K_1}{\mu_0 M_s} \right)^2 \) and \( H_K = \frac{2K_1}{\mu_0 M_s} \). The estimated values of \( M_s \) and \( H_K \) are 2.6\( \mu_B \) f.u. and 1.09 kOe, respectively. Although the \( M_s \) value

Figure 1. Observed and refined room-temperature XRD patterns for the \( \text{Sr}_2\text{FeMoO}_6 \) sample. The inset shows order-related peak that appear at 2\( \theta \sim 19.5^\circ \).

Figure 2. Temperature dependence of (a) magnetization (\( M \)) in a magnetic field of \( H = 1 \) kOe and (b) resistivity (\( \rho \)) for \( H = 0 \) kOe. (c) Field dependence of \( \rho \) at \( T = 10 \) and 325 K. The inset of (a) shows the magnetic field dependence of \( M \) at 10 K and (b) \( \ln(\rho) \) vs 1/\( T \) plot.
observed here is significantly lower than the expected $M_s$ of the bulk material, it is comparable with the value reported for Sr$_2$FeMoO$_6$ prepared by the same technique\textsuperscript{19} and the obtained $T_C$ is still favorable for the application purpose.

The temperature dependence of the resistivity ($\rho$) of Sr$_2$FeMoO$_6$ shows a semiconducting nature (Figure 2b). $\rho(T)$ increases monotonically with decreasing temperature and a rapid increase occurs below 20 K. The estimated activation energy, obtained from the fitting of the $\rho(T)$ curve with the Arrhenius equation ($\rho = \rho(0) \exp \left( -E_a/k_B T \right)$) in the temperature range $T = 290–390$ K, is $E_a \approx 85.67$ meV (inset of Figure 2b). Magnetoresistance (MR) is obtained from the standard definition $-\text{MR}$% = $\left[ \left( \rho(H) - \rho(0) \right) \times 100 \right]/\rho(0)$, where $\rho(H)$ and $\rho(0)$ are the resistivities at $H$ and the 0 kOe magnetic field, respectively. At 10 K, Sr$_2$FeMoO$_6$ shows negative MR with an absolute value of 10.3% in a magnetic field of 70 kOe, whereas MR is negligibly small (\textsim 0.7% at 70 kOe) at $T_C$ (325 K) as shown in Figure 2c. The MR at low fields (<1 kOe) is due to tunneling between ferrimagnetic grains and linear MR at higher fields is most likely due to scattering by disordered spins at the grain boundaries.\textsuperscript{1,2}

Figure 3a shows the magnetic field-dependent resonance spectra, plotted as the field derivative of power absorption ($dP/dH$) measured at a fixed frequency $f = 9$ GHz for different temperatures. At 370 K, the spectra are a single Lorentzian type. As the temperature decreases, the intensity of the $dP/dH$ spectra reaches a maximum at 325 K, which indicates the change in the magnetization states. The intensity of the signal again decreases below $T_C$ and $dP/dH$ is dominated by noise below 50 K.

The spectra were fitted to a linear combination of symmetric and an antisymmetric Lorentzian function (Figure 3b) given by

$$
\frac{dP}{dH} = A_{\text{sym}} \frac{4\Delta H(H - H_{\text{res}})}{[4(H - H_{\text{res}})^2 + (\Delta H)^2]^2} - A_{\text{asym}} \frac{(\Delta H)^2 - 4(H - H_{\text{res}})^2}{[4(H - H_{\text{res}})^2 + (\Delta H)^2]^2} + C
$$

where $H_{\text{res}}$, $\Delta H$, and $C$ are the magnetic resonance field, peak-to-peak line width, and offset, respectively. An example of the fit is shown for 300 and 350 K in Figure 3b. The above equation is equivalent to the Dyssonian equation, frequently used to fit the ESR data for conducting samples.\textsuperscript{29} The extracted $H_{\text{res}}$ and $\Delta H$ are summarized in Figure 3c. Two observations are evident. First, $H_{\text{res}}$ is nearly temperature independent in the paramagnetic state but shifts to a lower field as the temperature decreases below $T_C$. The decrease of $H_{\text{res}}$ is due to the continuous increase in the effective internal fields, such as the anisotropy and saturation magnetization. Second, the variation of the line width $\Delta H(T)$ value closely follows $M(T)$. Above $T_C$, $\Delta H$ shows an increasing trend, whereas $H_{\text{res}}$ remains almost constant. A similar trend in $\Delta H(T)$ was also observed in Ba$_2$FeMoO$_{6-\delta}$ where the temperature variation of the line width also does not show any peak below $T_C$ as the temperature decreases to 50 K. The presence of a maximum in $\Delta H(T)$ would indicate an electronic conduction process between the multivalence Fe$^{3+}$–Fe$^{2+}$ ions. Fe$^{2+}$ ions with a nonzero orbital momentum open up an additional slow-relaxation process in the crystal structure and give rise to a characteristic maximum in the $\Delta H(T)$ curve due to direct phonon modulation or indirect magnon modulation.\textsuperscript{16} The absence of maximum in $\Delta H(T)$ confirms the absence of Fe$^{2+}$ ions in our sample.

Broadband ferromagnetic resonance (FMR) spectra for multiple frequencies ($f = 2–18$ GHz) at selected dc magnetic fields were measured to calculate the gyromagnetic ratio ($\gamma$). Since $\gamma = g\mu_B/\hbar$, we can obtain the spectroscopic splitting factor or “Lande g-factor” of the material. Figure 4a shows the frequency derivative of the power absorption ($dP/df$) at room temperature as a function of the frequency of the microwave

Figure 3. (a) Magnetic field dependence of the resonance spectra plotted as the derivative of the absorbed power ($dP/dH$) at different temperatures, (b) Lorentzian fit of the $dP/dH$ line shape taken at $T = 300$ and 350 K, and (c) variation of line width ($\Delta H$) and resonance field ($H_{\text{res}}$) with temperature.
signal. The zero-crossing point of the spectrum can be identified as the resonance frequency \( f_{\text{res}} \). We can see that \( f_{\text{res}} \) increases with increasing strength of the static magnetic field. The recorded power absorption vs frequency pattern was fitted with the same Lorentzian equation (eq 2) to accurately determine \( f_{\text{res}} \) for all \( H_{dc} \) at temperatures down to 10 K (fitting is shown by the solid lines in the same figure).

Once the resonance frequencies are extracted for different fields and temperatures, \( f_{\text{res}} \) vs \( H_{dc} \) is plotted in Figure 4b. \( f_{\text{res}}(H_{dc}) \) is linear above \( T_C \); however, as we cool down the sample, \( f_{\text{res}}(H_{dc}) \) shows a continuous change in the slope. Nonlinearity increases as temperatures is lowered much below \( T_C \). These curves were fitted with Kittel’s expression for in-plane FMR

\[
f_{\text{res}} = \frac{1}{2\pi} \sqrt{(H_k + H_{dc})(H_k + M_{eff} + H_{dc})}
\]

where \( M_{eff} \) is the effective magnetization. At \( T \geq T_C \), the extracted value of \( \frac{f}{2\pi} \) (\( \sim 2.83 \) MHz/Oe) corresponds to a g factor of 2.02, which suggests the presence of paramagnetic \( \text{Fe}^{3+} \) ions. It also confirms that no \( \text{Fe}^{2+} \) ions were probed in the FMR measurement as the g value expected from the band calculation of the localized \( \text{Fe}^{2+} (3d^8) \) ion is much higher (\( g \approx 3.4 \)). Tovar et al. suggested that the itinerant electrons of both Fe and Mo as well as the localized core spins of \( \text{Fe}^{3+} \) contribute to the effective g-factor in \( \text{Sr}_2\text{FeMoO}_6 \). In the paramagnetic region, they found \( g = 2 \) for the localized and delocalized spins for \( \text{Fe}^{3+} \) and they are temperature independent. However, we find that the extracted value of effective g factor (\( g_{\text{eff}} \)) is not temperature independent in the magnetically ordered state; \( g_{\text{eff}} \) decreases slowly below \( T_C \) as shown in the inset of Figure 4b. This change in \( g_{\text{eff}} \) possibly arises from spin–orbit or crystal fields affecting the localized electrons, as \( g_{\text{eff}} = g (1 \pm \chi/\Delta) \), where \( \Delta \) is the crystal-field splitting and \( \chi \) is the spin–orbit coupling constant.

We have also collected \( dP/dH \) spectra as a function of the dc magnetic field for selected frequencies in paramagnetic and ferromagnetic regions. The resonance conditions obtained here match nicely with the frequency sweeps described earlier.

We employ the same method of Lorentzian function fitting to identify the parameters, \( H_{\text{res}} \) and \( \Delta H \). Figure 5a shows the acquired data points and the corresponding fitting for \( T = 300 \) K. \( H_{\text{res}} \) decreases monotonically with decreasing frequency of the microwave field from 15 to 3 GHz. The evolution of \( \Delta H \) with frequency for temperatures 10–100 and 150–350 K is shown in Figure 5b,c, respectively. The line width linearly varies with frequency from 350 to 200 K, however, the line width for a particular frequency increases with a decrease in temperature.

According to the Landau–Lifshitz–Gilbert (LLG) model of spin relaxation, the intrinsic line width is proportional to the frequency and is given by

\[
\Delta H = \Delta H_0 + \frac{4\pi \alpha f}{\gamma}
\]

where \( \alpha \) is the unitless Gilbert damping parameter that contains both the intrinsic and extrinsic contributions and \( \Delta H_0 \) is a frequency-independent extrinsic factor attributed to inhomogeneous line width broadening. At 350 K, \( \Delta H \) follows the LLG model and gives an intrinsic value of \( \alpha \sim 0.115 \pm 0.003 \). The slope of the linear \( \Delta H \) vs \( f \) curve is higher at 350 K than at 325 and 300 K. We estimate \( \alpha \sim 0.032 \pm 0.005 \) at room temperature and it is three orders of magnitude higher than the damping factor obtained from the angular dependence study of a \( \text{Sr}_2\text{FeMoO}_6 \) thin film. As shown in the figure, \( \Delta H \) shows Gilbert-like damping at temperatures below but close to \( T_C \). Interestingly, far below the room temperature, the damping parameter could not be extracted using eq 4 as \( \Delta H(f) \) shows a minimum and linearity is confined to a narrow-frequency range above the minimum. The frequency at which the line width shows a minimum increases with decreasing temperature. While the minimum occurs at 6 GHz when \( T = 200 \) K, it occurs at 15 GHz when \( T = 10 \) K. It has been reported that the FMR line width is enhanced at low frequencies due to a nonuniform magnetization distribution or spatial inhomogeneities in the magnetic property apart from field fluctuations and magnetic noise.
power-law term of the excitation frequency is added to eq 4 to \( \Delta H \) fitting

\[
\Delta H = \Delta H_0 + \frac{4 \pi f}{\omega_0} + \left( \frac{\eta_0}{f} \right)^{\beta/2}
\]

(5)
The last term in eq 5 is usually neglected in FMR studies since line width is reported mostly for the applied field greater than the anisotropy field. We obtain \( \beta = 1 \) at 200 K and its value increases to 4 for 10 K. This indicates the growing contribution of low-field losses in \( \text{Sr}_2\text{FeMoO}_6 \). As observed in the magnetization results \( M(H) \) at 10 K, the saturation is not achieved even at 5 T field at low temperature due to the presence of AS defects in the lattice.

3. CONCLUSIONS
We have investigated the magnetic resonance over a frequency range of 2–18 GHz in polycrystalline \( \text{Sr}_2\text{FeMoO}_6 \). Our results indicate that the Lande \( g \)-factor is \( \sim 2.02 \) in the paramagnetic state corresponding to the presence of an \( \text{Fe}^{3+} \) ion and it decreases with decreasing temperature in the ferromagnetic state. The resonance field smoothly decreases, and the line width increases with decreasing temperature in the ferromagnetic regime. \( \Delta H \) obtained from the dc magnetic field swept shows linear growth with frequency at \( T \geq 300 \) K in agreement with Gilbert damping, whereas below that temperature, the nonlinear behavior of \( \Delta H \) vs \( f \) indicates the existence of multiple damping factors in the composition. These findings highlight a major difference in the information that can be obtained from a cavity resonance technique reported before and a broadband technique employed here.

4. EXPERIMENTAL DETAILS
\( \text{Sr}_2\text{FeMoO}_6 \) was synthesized by the sol–gel reaction method using high-purity \( \text{Sr} (\text{NO}_3)_2 \), \( \text{Fe} (\text{NO}_3)_3 \cdot 9\text{H}_2\text{O} \), and \( \text{MoO}_3 \) powders. Stoichiometric amounts of the salts were well mixed in an aqueous solution of 1 M citric acid at 60 \(^\circ\)C for a day. Later, the precursors were kept at 500 \(^\circ\)C for 6 h in air. Finally, the pelletized sample was annealed at 1200 \(^\circ\)C for 6 h in a slightly reducing atmosphere (5% \( \text{H}_2 \), 95% Ar), which is necessary to avoid molybdenum evaporation and limit the concentration of the antisite defects in \( \text{Sr}_2\text{FeMoO}_6 \). Crystalline structure analysis of the double perovskite was performed using a Philip’s XPERT MPD powder X-ray diffractometer with a Cu K\( \alpha \) (1.542 Å) radiation source. Magnetization measurements were performed using a physical property measuring system (PPMS), equipped with a vibrating sample magnetometer. The frequency and magnetic field dependence of the derivative of microwave power absorption of a 4 \( \times \) 4 \( \times \) 1 mm\(^3\) pellet were measured using a broadband ferromagnetic resonance spectrometer (NanOsc Phase-FMR from Quantum Design Inc.) that makes use of the coplanar waveguide method. The flow of microwave (MW) current in the coplanar waveguide creates a magnetic field \( H_{\text{MW}} \) perpendicular to the direction of the MW current and the dc magnetic field provided by the superconducting coils in the PPMS. Thus, the dc and MW magnetic field configurations are similar to those in a conventional microwave cavity-based electron spin resonance spectrometer. The field and frequency swept resonance spectra at room temperature were also repeated for the sample in the powder form, and the resonance fields and line widths were found to be identical for both bulk and powder samples, which suggests that the microwave penetrates through the bulk of the sample.

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Notes
The authors declare no competing financial interest.

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