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# A microwave power absorption characterization of $YMnO_3$

G. Alvarez [a,*], H. Montiel [b], M.A. Castellanos [c], J. Heiras [d], R. Valenzuela [a]

[a] Departamento de Materiales Metálicos y Cerámicos, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, A.P. 70-360, Coyoacan DF 04510, Mexico
[b] Centro de Ciencias Aplicadas y Desarrollo Tecnológico de la Universidad Nacional Autónoma de México, México DF 04510, Mexico
[c] Facultad de Química, Universidad Nacional Autónoma de México, Cd. Universitaria, México DF 04510, Mexico
[d] Centro de Ciencias de la Materia Condensada, Universidad Nacional Autónoma de México, Apartado Postal 2681, Ensenada BC 22800, Mexico

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ABSTRACT

We report on the effects of temperature and dc magnetic field on the microwave power absorption measurements at X-band (8.8–9.8 GHz), in powder samples of $YMnO_3$ (YM). Two techniques are used: magnetically modulated microwave absorption spectroscopy (MAMMAS) and low-field microwave absorption spectroscopy (LFMAS). The measurements were performed in the 77–520 K temperature range. MAMMAS response showed distinctive features associated with microwave absorption by magnetic and electric dipoles; at low and high temperatures, the paramagnetic and dielectric absorptions of microwave are dominant, respectively. The profiles obtained by plotting the slope vs. temperature of the LFMAS line, while cooling or heating, are similar to those detected by the MAMMAS technique. We conclude that both measurements are a manifestation of the same response to electromagnetic absorption, in which the same physical processes take place.



## 1. Introduction

Yttrium manganite $YMnO_3$ (YM) crystallizes in two possible structural phases [1]: hexagonal and orthorhombic. An antiferromagnetic ordering occurs in both structural phases, but a ferroelectric ordering occurs only in the hexagonal phase [2], which belongs to the noncentrosymmetric $P6_3cm$ space group. For the hexagonal-YM, structural studies together with theoretical calculations recently revealed a ferroelectric transition at high temperature [3–5], $T_{FE} \sim 914$ K. This ordering is originated by the buckling of $MnO_5$ polyhedral accompanied by the displacements of Y ions, which lead to net electric polarization [6]. Upon cooling below $T_N = 70$ K (Néel temperature), Mn spins have an antiferromagnetic ordering with moments aligned on the *ab* plane in a 120° structure [4,5,7,8], forming a triangular antiferromagnetic structure. The hexagonal-YM belongs to an interesting class of materials known as ferroelectromagnets [3] or multiferroics [4], in which ferroelectric and antiferromagnetic ordering coexist at low temperature.

Evidence of coupling between the ferroelectric and magnetic ordering in this material has been reported [5]. The study of this material may offer an insight into the conditions of occurrence and interdependence of ferroelectricity and magnetism by examining the coupling between the two ordering phenomena. This coupling can result in the so-called magnetoelectric effect, where the magnetic (dielectric) properties of the material may be altered by the onset of the electric (magnetic) transition or by the application of an electric (magnetic) field. To investigate this coupling, the material is typically excited with a magnetic (electric) field and the observation of a typical electric (magnetic) response such as dielectric permittivity (magnetic susceptibility) is carried out [3,9]. Additionally, anomalies in the dielectric constant and loss tangent have been observed near the Néel temperature [5], when the material is driven through a phase transition.

Electric and magnetic dipoles are strong absorbers of microwaves [10]. For a pure dielectric material, only the dielectric losses contribute to the energy absorption of the electromagnetic wave, while for a pure magnetic material, the effect of the magnetic losses dominates over the dielectric loss. However, for multiferroic materials, the microwave absorption is due to a competition between the dielectric and magnetic losses, which originates from the combination of both ferroelectric and magnetic phases. Information on the absorption processes of each kind of dipole and their coupling is expected to be obtained from microwave experiments.

Recently, we have implemented two experiments to measure the microwave power absorption (MPA) as a function of temperature or dc applied magnetic field in multiferroic materials [11,12]. They are known as magnetically modulated microwave

* Corresponding author. Tel.: +52 55 5622 4653.
*E-mail address:* memodin@yahoo.com (G. Alvarez).

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absorption spectroscopy (MAMMAS) and low-field microwave absorption spectroscopy (LFMAS), respectively. These techniques have recently been used to detect the magnetic/electric transition in multiferroic $Pb(Nb_{0.5}Fe_{0.5})O_3$ [12]. Additionally, these techniques provide information on the temperature or dc magnetic field dependence of MPA in each material. More importantly, these techniques can distinguish between different dissipative dynamics of microwave absorbing centers [11–19].

The material under study has shown to have a coupling between the electric and magnetic phenomena [3,9]; the MPA is therefore expected to be due to both magnetic and electric dipoles contained in the material.

In this paper we present MAMMAS results on hexagonal-YM, showing distinctive features associated with the absorption of microwaves by magnetic or electric dipoles. The LFMAS results confirm our assignment for the involved absorption processes.

## 2. Sample preparation and experimental details

Polycrystalline samples of $YMnO_3$ were prepared from stoichiometric amounts of $Y_2O_3$ (99.99%, Aldrich) and $MnCO_3$ (99.9%, Alpha Aesar) by the standard solid-state reaction method. Reagents were ground and mixed to obtain an homogeneous mixture, which was decarbonated at 500 °C, followed by sintering at 1200 °C for 6 h under air atmosphere. X-ray diffraction powder analysis showed a single hexagonal $YMnO_3$ phase.

The microwave investigations used a JEOL JES-RES 3X spectrometer operating at X-band (8.8–9.8 GHz) adequately modified for the above-described techniques [11,20]. In these techniques, MPA response with modulation of an applied dc magnetic field ($H_{dc}$) is measured. These modulated MPA spectra can be recorded either as the temperature of the sample is scanned in presence of a constant $H_{dc}$ (MAMMAS measurements), or as this $H_{dc}$ is scanned at a constant temperature (LFMAS measurements). In both cases, the ac magnetic field of modulation ($H_{mod}$) is superimposed to $H_{dc}$ and the microwave signal is detected synchronously with the modulation using a phase-sensitive detector [11,20].

The MAMMAS signal was registered with an $H_{dc}$ of 600 Oe, an amplitude $H_{mod}$ of 4 Oe, an incident microwave power of 7 mW with a constant gain. A slow temperature sweep (~1 K/min) was used.

LFMAS measurements were performed using a JEOL ES-ZCS2 zero-cross sweep unit that digitally compensates any remanence in the electromagnet, thus allowing measurements to be carried out by cycling $H_{dc}$ about its zero value continuously from −1000 to +1000 Oe with a standard deviation of less than 0.2 Oe for the measured field; these cycles have their analog in the magnetic hysteresis measurements. In this technique the sample is zero-field cooled or heated to the desired temperature, and then it is maintained at a fixed temperature with a maximum deviation of ±1 K during the whole LFMAS measurement (~4 min of sweep).

MAMMAS and LFMAS spectra were recorded in the 77–300 and 300–520 K temperature regions. The complete scheme and details concerning the experimental set-up for the MAMMAS and LFMAS measurements can be found elsewhere [11,20].

## 3. Results and discussion

Fig. 1 shows the MAMMAS response that decreases monotonically as temperature decreases from 300 K, following an approximate decay exponential (which we call absorbing process A), reaching a minimum value at $T_{min}$ = 112.4 K. To better understand this behavior, we compare the MAMMAS response of a well known ferroelectric material, $BaTiO_3$ powders in the 77–300 K temperature range, see inset in Fig. 1. This profile shows a continuous

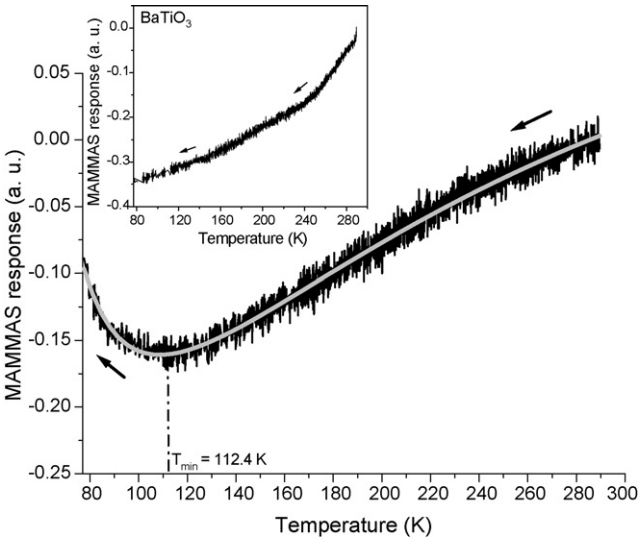


**Fig. 1.** MAMMAS response of hexagonal-YM for decreasing temperature; the solid line corresponds to a fit of exponential type, $0.11 + 0.03\,e^{185.38/T} - 0.74\,e^{-T/183.76}$. For comparison, the inset shows the MAMMAS response of $BaTiO_3$ powders in the 77–300 K temperature range.

decrease of exponential type with decreasing the temperature. Since $BaTiO_3$ has only electric dipoles, all of its microwave absorption is due to dielectric losses. For the behavior similarity between the MAMMAS profiles, we propose that the process A is due to the electric dipoles with long-range co-operative interactions in the hexagonal-YM; these dipoles can be driven by a microwave field by increasing their oscillating energy and therefore leading to absorption. When temperature decreases, the thermal excitation also decreases, and the electrostatic interactions increase. Electric dipoles have a diminished freedom to move, and therefore, absorption decreases.

As the temperature is decreased further into the $77\,K < T < T_{min}$ range, the MAMMAS response increases and another microwave energy-absorbing process sets-in, different from the previous behavior. This process follows an approximate growing exponential function (absorbing process B). We propose that the process B is originated mainly by the paramagnetic dipoles, as they have effectively exhibited this behavior [12,17]; then for low temperature, the effect of magnetic loss is dominant over the one from dielectric loss, but more evidence is gathered below.

Electron paramagnetic resonance (EPR) is the most powerful spectroscopic method available for obtaining local structural information and symmetry of paramagnetic ions incorporated in the structure [21,22]. This technique allows the investigation of the nature of magnetic phases in materials at different temperatures [13,17,23,24]. Fig. 2 shows EPR spectra ($dP/dH$ vs. magnetic field) for the hexagonal-YM, at a few selected temperatures in the 100–300 K range. EPR spectra show two symmetric Lorentzian lines along the entire temperature range, and they are labeled as 1 and 2. The broad signal 1 suggests a strong dipole–dipole interaction, due to the high concentration of $Mn^{3+}$ spins in the hexagonal-YM; where these $Mn^{3+}$ ions are located at the 6c sites, surrounded by a trigonal bipyramid of oxygen ions [1]. The inset of Fig. 2 displays the $g$-value as function of temperature for signal 1; it decreases continuously with the decrease in temperature. The linewidth of signal 1 broadens as the temperature is lowered, suggesting the existence of short-range antiferromagnetic correlations in the paramagnetic phase. This temperature dependence of our EPR data can be understood in terms of a universal behavior for the spin dynamics observed in many classes of manganites [25] that reveal

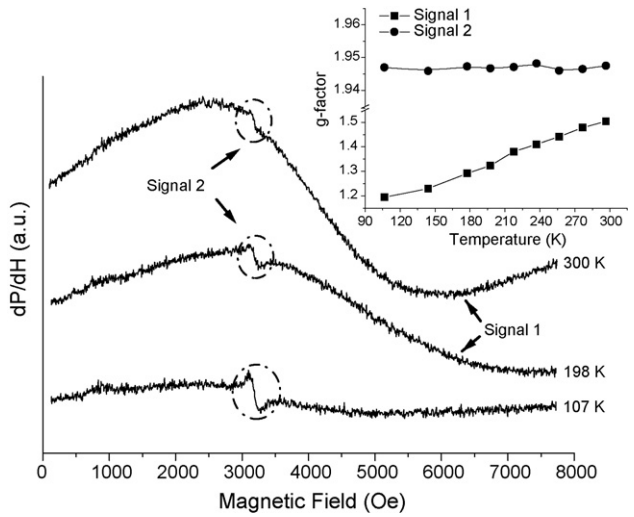


**Fig. 2.** EPR spectra of hexagonal-YM for selected temperatures in the 100–300 K range. The inset shows the temperature dependence of the *g*-factor in signals 1 and 2 for the same temperature range; curves connecting points are only guides for the eye.

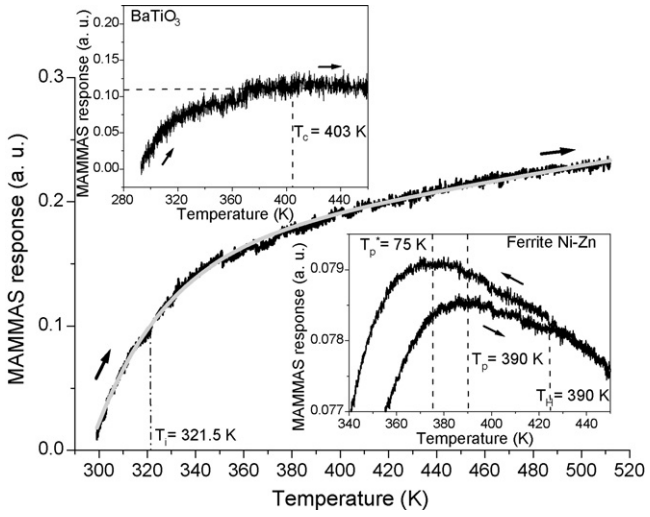


**Fig. 3.** MAMMAS response of hexagonal-YM recorded for increasing temperature; the solid line corresponds to a fit of the type $334.56–334.5\,e^{-T/1{,}000{,}000} - 3081.84\,e^{-T/30}$. For comparison, the insets show the MAMMAS response of ferrite Ni–Zn and $BaTiO_3$ powders, respectively, in the 300–520 K temperature range.

a strong magnetic coupling well above $T_N$. This behavior is similar to that found in the paramagnetic phase of systems exhibiting spin-freezing phenomena at low temperature (spin glasses), and can be understood as a manifestation of the presence of short-range magnetic correlations at $T \geq T_N$. The thin signal 2 is located approximately at all the temperatures in the same resonant field, ~3254 Oe, and it can be assigned either to the presence of ions of $Mn^{2+}$ ions [21,22] found as impurity traces from the starting reagents. The inset of Fig. 2 also shows the *g*-factor vs. temperature for the signal 2, where the *g*-factor is independent of the temperature, and this behavior confirms the paramagnetic nature of this signal [17,21,22].

On the other hand, the microwave power absorption in materials originates from magnetic losses or dielectric losses such as reorientation of magnetic domains, and electric charge displacement, respectively. The time-averaged power *P*, dissipated in a system carrying a current density *j* in an alternating electric field $E = E_0\,e^{-i\omega t}$ or by its magnetic component in an alternating magnetic field, $H = H_{mw}\,e^{-i\omega t}$, can be expressed as [26]:

$$P = \tfrac{1}{2}\omega(\varepsilon_0\,\mathrm{Im}(\varepsilon)E_0^2 + \mu_0\,\mathrm{Im}(\mu)\,H_{mw}^2) \quad (1)$$

where $\varepsilon_0$ and $\mu_0$ are the dielectric constant and the permeability of free space, respectively; $\mathrm{Im}(\varepsilon)$ and $\mathrm{Im}(\mu)$ are the imaginary part of the dielectric constant and the magnetic permeability, respectively, and $\omega = 2\pi f$, where *f* is the frequency. It can be observed that the microwave power absorption varies linearly with frequency, while the imaginary part of both the magnetic permeability and the dielectric constant depend on the square of the intensity of the magnetic and electric fields. For a constant frequency (and amplitude) of applied fields, the microwave power absorption should follow a behavior similar to $\mathrm{Im}(\mu)$ or $\mathrm{Im}(\varepsilon)$ when temperature is varied.

Fig. 3 shows the MAMMAS response recorded in the heating run for hexagonal-YM. Starting from room temperature, as the temperature is increased, the MPA increases monotonically and follows an exponential type law. We have recently shown that the paramagnetic microwave absorption should follow a continuous decrease when increasing the temperature [12]. Since the observed MAMMAS profile for hexagonal-YM shows a different behavior, we propose that this profile is due also to electric dipoles, and is the continuation of the process A to high temperatures. For a comparison, we also show the MAMMAS response of $BaTiO_3$ powders in the 300–460 K temperature range, see left-inset of Fig. 3. It shows a continuous increase of MPA when increasing the temperature, following also an exponential type law, and for $T > 403$ K the MPA reaches a constant value. This behavior is indicative of the gradual vanishing of the absorption process by electric dipoles with long-range interactions, and it is due to the ferro-paraelectric phase transition in $BaTiO_3$; the Curie temperature ($T_c = 403$ K) obtained by the MAMMAS technique is in a good agreement with the reported values [27,28]. Therefore, by similarity with $BaTiO_3$, we can assign the MPA profile for hexagonal-YM in the 300–520 K temperature range as associated with dielectric losses.

Additionally, a change in slope is observed in the MAMMAS response (Fig. 3), with an inflection point at $T_i = 321.5$ K. This change indicates the presence of an additional absorption process, C, which we propose as originated by relaxation effects or by a change of the transport mechanisms for the electric dipoles at those temperatures. The MPA is related to the imaginary part of the dielectric constant Eq. (1). As the material under study is a dielectric, the only current which can flow is the Maxwell displacement current induced by the microwave electric field. Since the sample is placed at the center of a resonant cylindrical cavity [20], the MPA must follow the behavior of $\varepsilon$ and satisfy the boundary conditions (inside the sample: $D_{normal} = \varepsilon E_{in}$ and outside the sample: $E_{out} = D_{out} \sim 0$; *D* is the displacement and $\varepsilon$ is the permittivity). Therefore, the features in the plot of the temperature dependence of the dielectric constant ($\varepsilon$ vs. *T*), associated with a physical process, should be reflected by some feature of the MAMMAS response. The temperature dependence of the dielectric constant, as well as impedance measurements at several selected rf frequency values, are reported in Ref. [9] on polycrystalline $YMnO_3$; this measurements show relaxations close to the transition temperature $T_i$. The thermal variations of MPA are therefore indicative of a dielectric process, A, and the set-in of another dynamics of absorption, C, are mainly due to relaxations of the electric dipoles.The previous behaviors contrast with those observed in magnetically ordered materials as it appears in Fig. 3, where the MAMMAS response for $Ni_{0.35}Zn_{0.65}Fe_2O_4$ polycrystalline ferrite in the 340–450 K temperature range is shown in right-inset. The MPA was taken from measurements in both heating and cooling directions in an attempt to look for a change in microwave absorption regime associated with a magnetic phase transition. During

heating (cooling), this signal increases monotonically as temperature increases (decreases) from 360 K (450 K), reaching a maximum value at $T_p$ = 390 K ($T_p^* = 375$ K), as shown in the right-inset of Fig. 3. As temperature increases (decreases) further, $T > T_p$ ($T < T_p^*$), the MAMMAS response decreases and another magnetic process sets-in, modifying its modulated microwave absorption and suggesting a magnetic transition. For $T > T_H$ (=424 K), MAMMAS response does not show thermal hysteresis in the heating and cooling cycles, as shown in the right-inset of Fig. 3, this is only observed when $T \leq T_H$; the merging point ($T_H$) clearly indicates the onset of the magnetic ordering. The $T_H$ value is in very good agreement with the value of Curie temperature ($T_c$) measured by means of the thermal variation of the initial permeability [29].

We turn now to the LFMAS results. Figs. 4(a) and 5(a) show the LFMAS spectra ($dP/dH$ vs. magnetic field, around zero field) recorded in the 77–300 and 300–520 K temperature ranges, respectively. We observe that all the curves exhibit a linear behavior with a positive slope and non-hysteretic traces. The positive slope implies that this MPA is a minimum around zero magnetic field and that it increases with field; in other words, this is a magnetic field-dependent absorption. The LFMAS line shows the absence of any irreversible microwave energy absorption process. This linear behavior of MPA for the hexagonal-YM has also been observed in other materials [12,30], but this behavior strongly contrasts with that shown by Ni–Zn [13] and cobalt [19] ferrites. LFMAS signal is closely related with the low-field magnetization of the sample in the long-range order regime; i.e., LFMAS is absent in the paramagnetic phase and emerges as the temperature is decreased below $T_c$.

Let us remember that in a dielectric material the Maxwell displacement current, $j = d(\varepsilon\varepsilon_0 E)/dt$, is induced by the microwave electric field $E$. Since $j = \sigma E$, where $\sigma$ is the microwave conductivity ($\sigma = (1/2)\varepsilon_0 \mathrm{Im}(\varepsilon)\omega$), we describe the magnetic field dependence of the microwave absorption, $P = \sigma E_0^2$, in terms of changes in microwave conductivity $\sigma(H)$ with an applied static magnetic field $H$. Experimentally the observed MPA at high temperatures where the dominant response is due to electric dipoles, is

$$\Delta P = P(H) - P(0) = \Delta\sigma E_0^2 \tag{2}$$

where $\Delta\sigma = \sigma(H) - \sigma(0)$ is the change in the microwave conductivity induced by the magnetic field $H$. Since dielectric loss in absence of static magnetic field is given by $P(0) = \sigma(0)E_0^2$, the observed response can be written:

$$\Delta P = \left[\frac{\Delta\sigma}{\sigma(0)}\right] P(0) \tag{3}$$

The term $\Delta\sigma/\sigma(0)$ describes relative changes in microwave conductivity induced by the external field, that is, magnetoconductance. The explanation of magnetoresistance in perovskite manganites [31] relies on assumptions about magnetic ordering or exchange coupling between manganese ions with different valency states.

In the present case, the low-field microwave power absorption by electric dipoles should be considered as the enhancement of dielectric loss by the external magnetic field, or microwave magnetoconductance, which leads to

$$\frac{dP}{dH} = \left[\frac{P(0)}{\sigma(0)}\right] \frac{d\sigma}{dH} \tag{4}$$

where the factor $P(0)/\sigma(0)$, the square of the microwave electric field $E_0^2$, is constant.

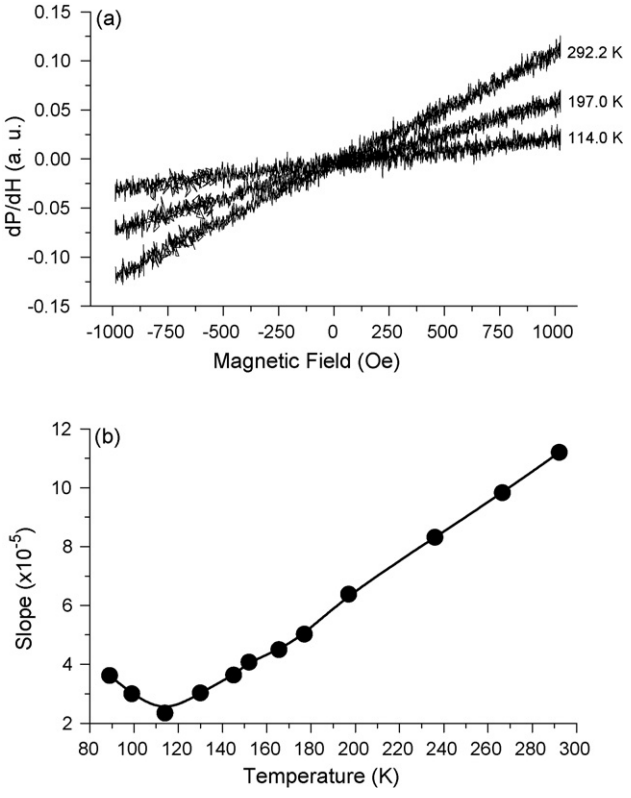


**Fig. 4.** (a) LFMAS spectra of hexagonal-YM for selected temperatures, with magnetic modulation of 0.63 Oe and microwave power 1 mW and (b) the temperature dependence of the absorption line slope for the 77–300 K temperature range. The solid lines are only guides for the eyes.

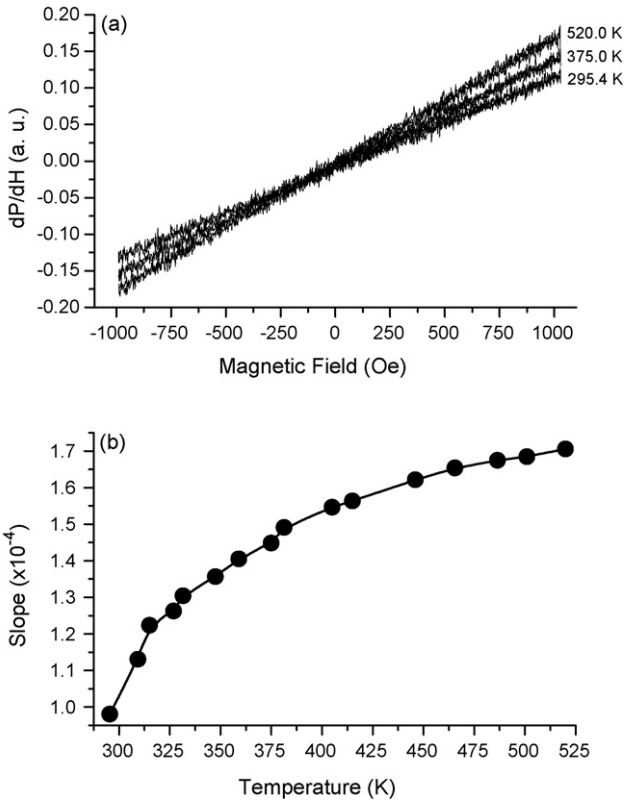


**Fig. 5.** (a) LFMAS spectra of hexagonal-YM for selected temperatures, with a magnetic modulation of 0.63 Oe and microwave power 2 mW and (b) the temperature dependence of the absorption line slope for the 300–520 K temperature range. The solid lines are only guides for the eyes.

For low temperatures, where the dominant response is due to magnetic dipoles, the low-field microwave absorption is given as

$$\frac{dP}{dH} = \frac{1}{2}\omega\mu_0 H_{mw}^2 \frac{d\ \mathrm{Im}(\mu)}{dH} \quad (5)$$

Here, the amplitude of the microwave magnetic field, $H_{mw}$, is constant.

On the other hand, the LFMAS line can be described by the experimental correlation:

$$\frac{dP}{dH} = S(T)H \quad (6)$$

where $S(T)$ depends only on temperature.

To find the nature of the MAMMAS response, we calculated the derivative of the LFMAS line, $d^2P/dH^2$, in the low magnetic field range ($\leq$1000 Oe):

$$\frac{d^2P}{dH^2} = S(T) \quad (7)$$

this value does not depend on the magnetic field.

Figs. 4(b) and 5(b) show the behavior of the slope of the LFMAS line, $S(T)$, in the 77–300 and 300–520 K temperature ranges, respectively. We observe that the profiles obtained are similar to the shape of the corresponding MAMMAS responses. This clearly points to a common origin for both measurements, giving evidence that LFMAS spectra are generated by the same absorption processes above described, namely the absorption dynamics for electric and magnetic dipoles in different regions of temperature. Also, as the MAMMAS responses are similar to the plot of the slope vs. temperature, we can establish an experimental correlation between the MAMMAS response and the second derivative microwave power absorption ($d^2P/dH^2$); i.e., for low magnetic field ($\leq$1000 Oe) the MAMMAS response is proportional to $d^2P/dH^2$.

## 4. Conclusions

MAMMAS and LFMAS techniques provide information on the temperature or dc magnetic field dependence of the modulated microwave power absorption of materials. More important, these techniques can distinguish between different dissipative dynamics of microwave absorbing centers, providing also valuable information about the nature of the microwave absorption within materials. These techniques are very sensitive, requiring a very small amount of material for testing in the form of a cylinder, sphere, film, ribbon or powder; these wireless techniques are non-destructive.

In the particular study of this work, the MAMMAS measurements in the $T_{min} < T < 520$ K temperature range showed two microwave absorption processes, A and C, which are associated with the dynamics of electric dipoles. In the $77\ \mathrm{K} < T < T_{min}$ range, a third absorption process appears (B) which can be associated with the microwave absorption by magnetic dipoles. The LFMAS spectra showed a linear behavior with positive slope and non-hysteretic traces. The spectral changes for the plot of the slope vs. temperature, both during cooling and heating, showed features similar to those of MAMMAS profile; it confirmed that both profiles are due to the same absorption processes. Additionally, we found an experimental correlation of MAMMAS response with the second derivative of the microwave power absorption, $d^2P/dH^2$.

As a general conclusion, we have showed that the modulated microwave power absorption techniques LFMAS and MAMMAS are powerful tools for the research of materials at microwave frequencies.

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