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Experimental demonstration of the nonreciprocity of magnetic composite materials for microwave applications

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The purpose of this article is to bring out the nonreciprocity of ferrimagnetic powder loaded composites. We first describe their technology of preparation. Then we briefly recall the principle of the broadband measurement method used to determine the permeability tensor components of magnetized materials. Our experimental results performed at *X*-band frequencies (8–12 GHz) on two different ferrimagnetic loaded composite samples are presented and discussed. We finally show that for each material under test, the off-diagonal component κ of the permeability tensor, which is at the origin of the nonreciprocal effect, is of a magnitude comparable to the magnitude observed in bulk ferrites. This result proves that powders technology can be used to realize composite materials for nonreciprocal microwave applications.
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I. INTRODUCTION

For the achievement of nonreciprocal devices (circulators, isolators) and tunable circuits (phase shifters, filters) in the centimeter wave range, soft ferrites have been widely used. For future applications on the communication market higher-operating frequencies, small size and low-production cost circuits will be required. For planar magnetic circuits, the typical operating frequency is to a large extend determined by the strength of the anisotropy field $H_a$and the saturation magnetization M_s of the magnetic plate sample.[1] To reduce the size and cost of circuits, the choice of the material fabrication process is constrained by the desire for compatibility with the monolithic microwave integrated circuits (MMIC) technology. It requires a deposit of the magnetic components directly on the MMIC chip. But, the semiconductor substrates used to realize the active functions cannot withstand high-processing temperatures.

To fit the previous schedule of conditions soft ferrites are not the best suited materials. Their moderate anisotropy field and saturation magnetization ($M_s < 400$ kA/m) limit the circuit operating frequency. Moreover, the high-sintering temperatures (1200–1400 °C) needed during their manufacturing process make them incompatible with MMIC chip technology.

The substitute materials we propose consist of mixing various components to obtain a medium with unique properties nonexistent in ferrites. They are composed of ferromagnetic powders embedded in a dielectric binder. To avoid metallic losses in the composite material, electrical insulated ferromagnetic grains with a typical size less than the skin depth must be used. Compared to polycrystalline ferrites, the main interests of these composite materials are (1) a high-saturation magnetization can be reached; and (2) a technological process such as silkscreen printing, which is compatible with the MMIC technology, can be used for the manufacturing of the samples.

The fact that there are no losses due to domain wall motion in composites with single domain particles permits us to use the high level of the real part of the permeability below the gyromagnetic resonance to conceive microwave tunable devices. Indeed, demonstration that ferromagnetic/dielectric composite materials can be used in microwave tunable circuits has been recently done.[2,3] Unfortunately, the electomagnetic properties measurements of the tunable composites made in our laboratory does not exhibit nonreciprocal effects, due to the geometry of the ferromagnetic particles (thin layers or nanowires).

One of our aims is to realize planar nonreciprocal microwave devices whose substrate is a thick ferromagnetic powder loaded composite layer, deposited with the silkscreen printing technique. The first step in this demonstration consists of showing that magnetic matter keeps its field-induced anisotropy properties when it is divided in small size particles. In this article, to valid powders technology for microwave applications, we prove that ferrimagnetic powder loaded composites exhibit a nonreciprocal effect.

II. METHOD OF PREPARATION OF THE COMPOSITES

One well-known way of manufacturing a magnetic composite material is to disperse randomly ferrimagnetic or ferromagnetic particules in an insulating binder. The fabrication process is divided in two main steps.

The first step consists of mixing magnetic powders with a dielectric resin by a humid process using acetone. The mixture is dried under vacuum. A slight grinding using a hand mill enables us to break agglomerates. This process permits us to obtain a homogeneous distribution of the magnetic powder in the composite material.

The second step consists of pressing the mixture in rectangular-shaped matrixes at room temperature (uniaxial pressure less than 10^4 kg/cm^2).

The resin used is soluble in acetone and polymerized at 150 °C during one hour. It works as a binder and, if necessary, as a dilution agent. The magnetic powders are obtained

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either by grinding sintered ferrites or by coprecipitating methods. Our laboratory currently uses magnetic powders whose particle sizes typically range from 0.1 to 150 μm.

For the composite materials studied in this article two different ferrimagnetic powders have been used. The first one has been obtained by grinding (rolls grinder) aluminum substituted yttrium iron garnet (YIG) (Curie temperature $T_c = 195\,°\mathrm{C}$, saturation magnetization $M_s = 63.7$ kA/m). This powder has been provided by Tekelec-Temex Industry at Montreuil, France. A dry riddling insures a grains size less than 20 μm. The second ferrimagnetic loaded composite is composed of a commercially available hexaferrite powder (Tekelec-Temex Industry). The mean grain size is of 50 μm. The material supplier does not provide the composition and the saturation magnetization for the hexaferrite powder. It only gives the value of the gyromagnetic resonance frequency $f_r = 5$ GHz of the ceramic material in a completely demagnetized state. The density of each powder has been measured using an helium pycnometer. We have found a density of 5.14 for the first powder and of 5.28 for the second one.

III. MEASUREMENT METHOD

To exhibit the required properties, the magnetic sample integrated in the nonreciprocal circuit must be placed in a magnetized state applying a dc magnetic field. In this case, its permeability is a tensor quantity which in the Cartesian coordinate system takes the following form:

$$\overleftrightarrow{\mu} = \begin{bmatrix} \mu & 0 & +j\kappa \\ 0 & \mu_y & 0 \\ -j\kappa & 0 & \mu \end{bmatrix} \quad \text{where} \quad \begin{array}{c} \mu = \mu' - j\mu'' \\ \kappa = \kappa' - j\kappa'' \\ \mu_y = \mu'_y - j\mu''_y, \end{array}$$

when the dc field is applied along the y axis. The permeability tensor components are complex owing to the existence of magnetic losses. The off-diagonal component κ is substantial for the realization of circulators and isolators, since it is at the origin of the nonreciprocal effect.

A broadband measurement method of the permittivity $\epsilon = \epsilon' - j\epsilon''$ and the μ and κ permeability tensor components using a rectangular waveguide has been developed in our laboratory.[4] The validation of the measurement technique has been demonstrated for polycrystalline ferrites only.[4]

IV. EXPERIMENTAL RESULTS

A. Description of test device

The sample holder is an X-band (8–12 GHz) rectangular waveguide. Inside dimensions of its cross section are $22.86 \times 10.16\ \mathrm{mm}^2$. A 10.16 mm wide rectangular piece of the test material is inserted into the propagation structure as sketched in Fig. 1.

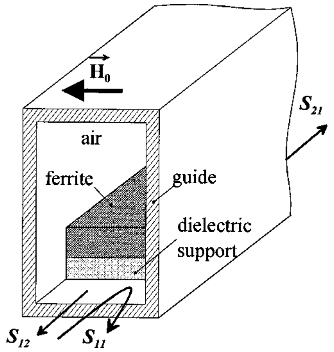

FIG. 1. Rectangular waveguide sample holder and static magnetic-field H_0 direction.

To avoid dimensional resonances, the sample length must be less than $\lambda_g/2$, where λ_g is the wavelength in the sample. The test sample has a typical thickness of a few millimeters. The S-parameter measurements of the device are performed using an HP 8510 B network analyzer setup. The access planes of the sample holder are the reference planes for a thru-reflect-line (TRL) calibration procedure[5] conducted to eliminate systematic errors due to the defect of the cables, coaxial-to-waveguide adapters, and impedance mismatch of the cell. The cell is set in between the poles of an electromagnet, in order to apply to the test sample static magnetic fields up to 1600 kA/m.

B. Permeability tensor measurements

Two $5 \times 10.16 \times 2\ \mathrm{mm}^3$ rectangular composite samples were manufactured using the technological process described in Sec. II. The first one is a YIG loaded composite (loaded factor in YIG $p = 0.55$) and the second one is a hexaferrite loaded composite $(p = 0.60)$. The amount of air bubbles in the first and the second composites are of 15% and of 25% respectively.

Measured μ and κ data as a function of frequency and for different dc magnetic field H_0 strength are given in Fig. 2 for the first composite material.

The YIG-resin composite sample is completely saturated by the external dc field when the gyromagnetic resonance is in the X-band. The relations of Polder and Smit[6] which are usually assumed to apply only to saturated media, cannot be used to validate the measured curves, even if the composite sample is saturated. Indeed, The theory of Polder and Smit was developed for single ferrite crystals and cannot take into account the volume fraction in magnetic inclusions in the composite. However, in analyzing the measured μ and κ data, different observations prove the validity of the experimental results obtained from our technique. The measured

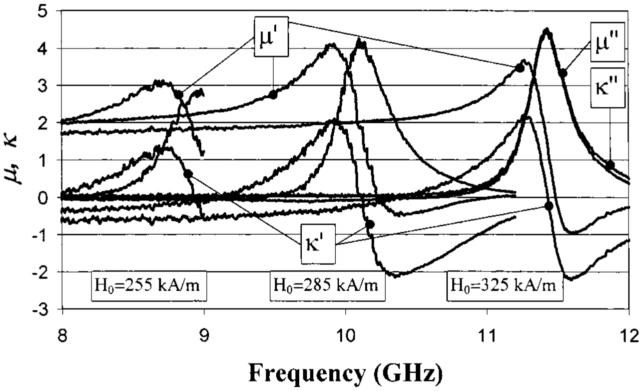

FIG. 2. Measured μ and κ data vs frequency for the YIG loaded composite material $(p = 0.55)$ for different strengths of the external dc magnetic-field H_0.

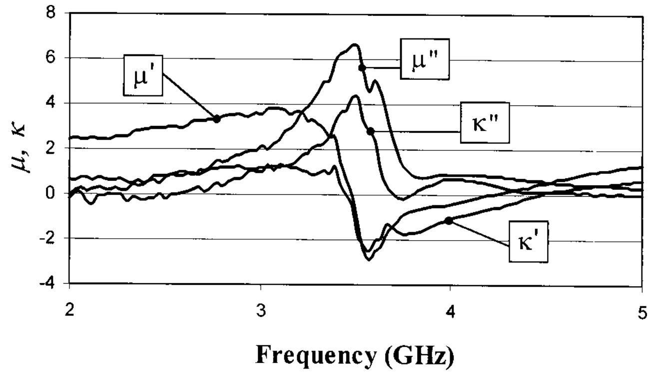

FIG. 3. Measured μ and κ data vs frequency for the sintered YIG ferrite close to saturation ($H_0 = 119$ kA/m).

imaginary parts μ'' and κ'' of the permeability tensor components reveal a peak absorption of the microwave due to the damped precession of the spins around the dc magnetic-field H_0 direction. The frequency where maximal absorption occurs in loss curves is the gyromagnetic resonance frequency f_r. As we can observe in Fig. 2, the gyromagnetic resonance frequency f_r shifts linearly with the dc magnetic field H_0 strength. From those measurements and the well-known theoretical law[1] $f_r = \gamma H_0$, which can be applied to saturated media, it is possible to obtain an experimental value for the gyromagnetic ratio. The measured value for γ is 0.0342 GHz/kA.m^{-1} and is seen to be in close agreement with the theoretical value of 0.0352 GHz/kA.m^{-1}. The requirement of a non-negative energy loss in the composite material imposes the restriction that μ'' and κ'' are positive. This condition is confirmed by the measured loss curves, as seen in Fig. 2.

We have measured the electromagnetic properties of the sintered YIG ferrite at low field in order to compare the magnitude of the permittivity and the permeability tensor components of the YIG in the composite resin state and in the sintered state. The measurement cell used at low field (i.e., low frequency) is composed of a nonreciprocal strip transmission line partly filled with the sample that is to be characterized.[7] The electromagnetic parameters of the sample under test are deduced in the 130 MHz-6 GHz frequency range from the measured scattering matrix of the cell using analytical relations. Measured μ and κ data as a function of frequency for a $5 \times 5 \times 1.85$ mm^3 rectangular sample of the sintered YIG ferrite close to saturation ($H_0 = 119$ kA/m) are given in Fig. 3. We can notice in Fig. 3 that the real part κ' of the off-diagonal component, which is at the origin of the nonreciprocal effect, is of a magnitude comparable to the magnitude of κ' measured on the YIG powder loaded composite in a saturated state (Fig. 2). The complex permittivities of the composite material and the sintered ferrite have been measured using the strip transmission line cell. The measured complex permittivities for the YIG in the composite state and in the sintered state are, respectively, $\epsilon = 12.08$-j 0.012 and $\epsilon = 14.00$-j 0.011 at 3 GHz. The composite does not exhibit higher dielectric losses than the sintered ferrite.

Measured κ', κ'' data versus frequency for the second magnetic composite material are depicted in Fig. 4 for different values of the dc magnetic field, i.e., for a different magnetization state of the material. Contrary to the YIG-resin composite sample, the hexaferrite-resin composite material is not saturated by the dc field H_0 when the gyromagnetic resonance is in the X-band, due to the high value of the anisotropy field of the hexagonal ferrite inclusions. A typical behavior of unsaturated materials can be observed in Fig. 4(b) where loss curves κ'' are sharper when H_0 is increasing.

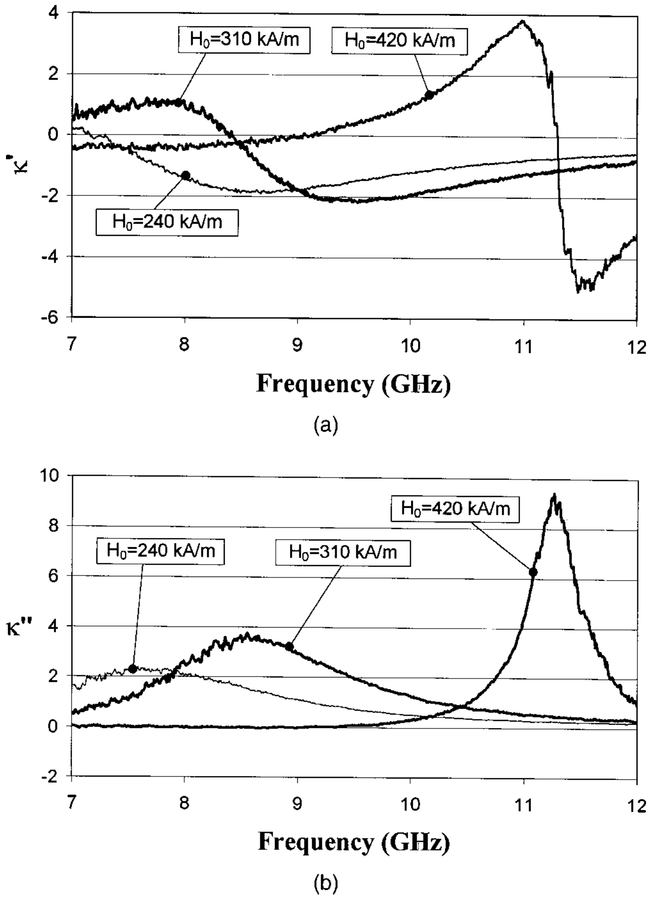

FIG. 4. Measured κ data vs frequency for the hexaferrite loaded composite material ($p = 0.60$). (a) Measured κ' data and (b) measured κ'' data.

V. CONCLUSION

Now, two stages must be reached to obtain results of a technological interest. We will first have to show that composites made up of ferromagnetic powders can be at the same time nonreciprocal and low loss. Then, we will have to reproduce this demonstration in the case of thick ferromagnetic powder loaded layers deposited by silkscreen printing.

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