Scanning microwave microscopy technique for nanoscale characterization of magnetic materials

C.H. Joseph a,b,*, G.M. Sardi a, S.S. Tuca c, G. Gramse c, A. Lucibello a, E. Proietti a, F. Kienberger d, R. Marcelli a

a National Research Council, Institute for Microelectronics and Microsystems (CNR-IMM), Via del Fosso del Cavaliere 100, 00133 Rome, Italy
b Department of Electronics Engineering, University of Rome “Tor Vergata”, Via del Politecnico 1, 00133 Rome, Italy
c Johannes Kepler University, Institute for Biophysics, Gruberstrasse 40, A-4020 Linz, Austria
d Keysight Technologies Austria GmbH, Keysight Laboratories, Gruberstrasse 40, A-4020 Linz, Austria

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In this work, microwave characterization of magnetic materials using the scanning microwave microscopy (SMM) technique is presented. The capabilities of the SMM are employed for analyzing and imaging local magnetic properties of the materials under test at the nanoscale. The analyses are performed by acquiring both amplitude and phase of the reflected microwave signal. The changes in the reflection coefficient $S_{11}$ are related to the local properties of the material under investigation, and the changes in its magnetic properties have been studied as a function of an external DC magnetic bias. Yttrium iron garnet (YIG) films deposited by RF sputtering and grown by liquid phase epitaxial (LPE) on gadolinium gallium garnet (GGG) substrates and permalloy samples have been characterized. An equivalent electromagnetic transmission line model is discussed for the quantitative analysis of the local magnetic properties. We also observed the hysteretic behavior of the reflection coefficient $S_{11}$ with an external bias field. The imaging and spectroscopy analysis on the experimental results are evidently indicating the possibilities of measuring local changes in the intrinsic magnetic properties on the surface of the material.

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1. Introduction

Scanning microwave microscopy (SMM) is a novel tool providing the ability to perform microwave frequency material characterization and imaging at the nanoscale level [1]; it belongs to the wider group of near field probing techniques [2]. SMM combines an atomic force microscopy (AFM) with a vector network analyzer (VNA); by this way highly sensitive measurements can be achieved with high spatial resolution. Imaging as well as quantitative characterization of materials and devices at the nanoscale has become a capital concern in recent years; SMM technique has been proven to be useful in many research fields [3]. This technique proffers localized non-destructive characterization of materials ranging from semiconductors to biological samples in a wide frequency band of operation (typically 1–20 GHz). Significant results are reported for the electrical characterization of nano-devices [4]; with more emphasis on the material under analysis, few works are about graphene testing [5,6]. In this specific case, measurement of the calibrated complex surface impedance at microwave frequencies is crucial for proper designing of devices. Contact and non-contact modes of operation are possible in SMM, but the most promising asset is the sub-surface imaging and non-destructive capabilities of the instrument [7].

At present, SMM technique is widely applicable and well established mainly for dielectric materials [8], for calibrated doping profile measurements of semiconductor materials [9,10]. In addition to that, promising results have been reported also for biological applications [11], for the characterization of the electric permittivity and complex impedance of bacteria and cells [12,13]. Concerning magnetic materials, microwave microscopy is not yet a consolidated technique in the literature. This is in spite of the importance of such kind of materials in micro- and nano-electronics devices: tunable filters [14], magnonic crystals [15], magnetostatic spin waves and tunable resonators [16]. However, the necessity of an accurate and localized characterization of these materials in terms of electric and magnetic properties such as the permeability is still an actual need. The application of near-field microwave microscopy for magnetic materials has been briefly demonstrated for the analysis of the magnetic properties of metal...
sheets and permalloy thin films \[17,18\]. A similar technique was also used for imaging magnetic artificial domains of a hard disk \[19\]; in this case magnetic bits (or hard disk sectors) having micrometric size are quite clearly recognized with respect to the non-magnetized surface. An interesting challenge is the possibilities of imaging and of electromagnetically characterizing the native magnetic properties variations in magnetic thin films. Here, we performed SMM characterization focused on yttrium iron garnet (YIG) films deposited by RF sputtering and grown by liquid phase epitaxy (LPE) on gadolinium gallium garnet (GGG) substrates; we also studied bulk permalloy as well as permalloy thin films on silicon substrate. The analysis is performed by acquiring both amplitude and phase of the reflected microwave signal. The SMM probe acts as a local antenna, able to detect very small changes of the phase and amplitude of the $S_{11}$ reflected signal. These changes are induced by variations in the measured local impedance of the sample, which are subsequently related to both the intrinsic magnetic properties of the material under analysis and their modification, stimulated by an external DC magnetic bias field.

2. Experiment

2.1. Experimental setup

The commercial SMM (Keysight Technology) composed of a standard 5600 atomic force microscopy (AFM) interfaced with a vector network analyzer (VNA) is used for the experiments. In reflection mode SMM, a microwave signal from the VNA fed to a specially designed conductive AFM tip through a $\lambda/2$ coaxial line resonator. Its length is 90 mm along with a 50 $\Omega$ shunt resistor to match the high impedance of tip to the characteristic impedance of the VNA (standard value of 50 $\Omega$); Fig. 1 shows a sketch of the experimental setup. Depending on the impedance at the tip/sample interface, part of the microwave signal is reflected back and measured by the VNA as the $S_{11}$ reflection signal. Using an AFM tip as a probe, SMM can offer simultaneous topography and microwave measurements ($S_{11}$ amplitude and phase) with the spatial resolution of an AFM, on the same order of magnitude of the tip apex size. A commercial solid platinum AFM tip (RMN 25pt 300B) with spring constant \(18 \text{ N m}^{-1}\) and nominal apex radius of \(<20 \text{ nm}\) with a tip height of \(\sim 100 \mu\text{m}\), from Rocky Mountain Technology is used in our experiment.

A permanent magnet (Sm-Co) is placed under the sample and by this way a DC magnetic field is applied perpendicularly with respect to the sample surface; the variation of the magnetic bias is obtained by moving the magnet up and down under the sample plate using a manually adjustable micromanipulator. Fig. 2 shows the photograph of this experimental arrangement. With this arrangement, we tuned the DC magnetic field values, from 200 Oe to almost 1700 Oe. We measured the magnetic field values at each position with a Hall probe. All the measurements were performed in contact mode AFM. The reflection coefficient $S_{11}$ has been measured for different external magnetic bias values. All the measurements were finally performed in the frequency range of 15–20 GHz, where best sensitivity was observed, after acquiring the full frequency sweep between 1 GHz and 20 GHz.

2.2. Preparation of samples under test

Two different kinds of samples have been considered for the characterization:

1. Low permeability lossy sample (YIG thin films);
2. High permeability conductive sample (permalloy thin films).

Then, two differently grown YIG film samples have been considered and both samples were grown on commercially available 500 $\mu\text{m}$ thick gadolinium gallium garnet (GGG) wafers. One sample was grown by RF sputtering technique, initially the substrate heated up to 400 °C, and the deposition performed at a working pressure of $5 \times 10^{-2}$ mbar with RF powers between 200 and
300 W. The grown film then crystallized by a post-thermal annealing at 850 °C. The other sample was grown by liquid phase epitaxy (LPE) technique from the PbO/B2O3 fluxed melt. We obtained a 2.5 μm thick (RF sputtered) and an 87 μm thick (LPE) YIG films.

Subsequently, thermally evaporated permalloy thin films on 500 μm silicon substrate with film thicknesses of 30 nm, 50 nm and 70 nm were also prepared and measured.

3. Theory

3.1. Transmission line modeling

Observing the geometry of the set-up, Fig. 1, and recalling that our analysis employs contact mode SMM, the local phenomenon, in terms of electromagnetic modeling, can be analyzed by an equivalent transmission line [20], as schematized in Fig. 3. The sample under test can be described, in a generic way, as a bilayer composite, thus a substrate covered by a deposited magnetic film.

When performing the measurements, the sample is set on top of a sample holder, in our case a glass support. Each material is electrically defined by the two complex quantities.

Dielectric permittivity \( \varepsilon_r = \varepsilon' - j\varepsilon'' = \varepsilon' (1 - j\tan \delta_r) = \varepsilon' (1 - j\tan \delta_r) \).

Magnetic permeability \( \mu_r = \mu' - j\mu'' = \mu' (1 - j\tan \delta_r) = \mu' (1 - j\tan \delta_r) \).

Each section \( i \) of the line is described by the complex parameters:

Impedance, neglecting losses \( Z_i = \sqrt{\frac{\mu_r}{\varepsilon_r \omega}} \).

Propagation constant (phase constant \( \beta \), attenuation constant \( \alpha \)) \( k_i = \omega \sqrt{\mu_r \varepsilon_r} = \beta_i - j\alpha_i \).

Length (real quantity) \( t_i \).

In the above model, we assume that the only unknown parameter is the permeability \( \mu_{mag} \) of the magnetic film.

The whole transmission line can be studied either numerically by means of a commercial software, like Microwave Office (National Instruments) in our case, or either modeled analytically and then manipulated in a broader way. The former model is useful for a fast preparatory analysis to check the expected results. For this analytical model, we use the cascade of \( T \) (or \( ABCD \)) matrices. In fact, the whole model is the product of seven matrices. However, we shall consider negligible the losses induced by the various layers; that hypothesis is consistent to the very low thickness of the magnetic film and the supporting substrate. Each section of Transmission Line is described by the \( 2 \times 2 \) matrix:

\[
T_i^{(i)} = \begin{bmatrix}
\cos(\beta_i t_i) & jZ_i \sin(\beta_i t_i) \\
\frac{j \sin(\beta_i t_i)}{Z_i} & \cos(\beta_i t_i)
\end{bmatrix}
\]

(1)

Any shunt load is modeled by:

\[
T_i^{(sh)} = \begin{bmatrix} 1 & 0 \\ 0 & 1 \end{bmatrix}
\]

(2)

Any series load is:

\[
T_i^{(sl)} = \begin{bmatrix} 1 & Z_i \\ 0 & 1 \end{bmatrix}
\]

(3)

The above model relates accurately the reflection coefficient \( S_{11} \) to the terminal impedance. By working close/or at the resonance frequencies of the coaxial resonator one can obtain impedance matching between the tip/sample system and VNA; only in this...
particular condition, $S_{11}$ can be approximated as [21]:

$$S_{11} \approx \frac{Z_{\text{total}} - Z_{0}}{Z_{\text{total}} + Z_{0}}$$

(4)

$Z_{0}$ is the characteristic impedance of the system which is 50 Ω, and $Z_{\text{total}} = Z_{\text{res}} + Z_{\text{sample}}$; $Z_{\text{res}}$ is the equivalent impedance of the $\lambda/2$ coaxial line resonator section, defined as [22]:

$$\frac{1}{Z_{\text{res}}} = \frac{1}{Z_{\text{shunt}}} + \frac{1}{R_{\text{res}}} + \frac{1}{j\omega L_{\text{res}}} + j\omega C_{\text{res}}$$

(5)

By considering magnetic film and substrate $Z_{\text{sample}}$ is a bi-layer system, defined as:

$$Z_{\text{sample}} = \frac{Z_{\text{sub}} + j\mu_{\text{mf}} \tan(k_{\text{mf}} \delta_{\text{mf}})}{Z_{\text{mf}} + j\mu_{\text{sub}} \tan(k_{\text{mf}} \delta_{\text{mf}})}$$

(6)

where $Z_{\text{sub}}$ is the impedance of the substrate and $Z_{\text{mf}}$, $k_{\text{mf}}$ and $\delta_{\text{mf}}$ are the impedance, wave vector and thickness of the magnetic film respectively. In our case, with two different kinds of samples (lossy dielectric and metallic), the impedance and wave vector of the magnetic film defined as:

$$Z_{\text{mf}} = \frac{\mu_{\text{mf}}}{\epsilon_{\text{mf}} \left(1 - j \tan(\delta_{\text{mf}})\right)}$$

and $k_{\text{mf}}$

$$= \frac{\mu_{\text{mf}} \epsilon_{\text{mf}}}{2 \left[1 + (\tan(\delta_{\text{mf}}))^2 - 1\right]}$$

for lossy dielectrics (YIG)

$$Z_{\text{mf}} = (1 + j)\sqrt{\sigma_{\text{mf}} \mu_{\text{mf}}}$$

and $k_{\text{mf}} = (1 + j)\sqrt{\sigma_{\text{mf}} \mu_{\text{mf}}}$, for metals (Permalloy)

3.2. Numerical analysis

Here, we analyze the case of a GGG substrate with a deposited YIG magnetic film (LPE grown YIG with thickness 87 μm). The length of the standard coaxial cable connecting the sensing part (tip plus resonator) to the VNA is 105 cm. As the coaxial cable length is proportional to the loss introduced in the system, we included this value in the modeling to obtain the best matching conditions with respect to the experimental results. The sensing part is described by the resonator (a coaxial cable of fixed length, with an effective length of 9 cm, and an inner dielectric permittivity of 3.72) and a shunt matching load [23], plus the tip equivalent impedance [24] and the cantilever contribution [25].

In the studied case, we vary the permeability $\mu_{\text{mf}}$ of the magnetic film, keeping geometrical and electromagnetic parameters unchanged. The changing of $\mu_{\text{mf}}$ is expected by means of the variation of the intensity of the external magnetic biasing field. In

![Fig. 6](image_url) The measured $S_{11}$ amplitude for the LPE-YIG with respect to different external magnetic fields. Inset shows the measured $S_{11}$ as a function of applied external DC magnetic field for the RF sputtered and LPE-YIG samples at the resonance frequencies.

![Fig. 7](image_url) Measured $S_{11}$ response as a function of magnetic field at 17.45 GHz. The arrows indicate the direction of magnetic field sweep. The inset shows the hysteresis curve obtained from vibrating sample magnetometer measurement.

![Fig. 8](image_url) (a) Measured reflection coefficient for air (empty sample holder), silicon substrate and permalloy films with thicknesses 30 nm, 50 nm and 70 nm at zero bias condition. (b) Measured $S_{11}$ variations and resonance frequency as a function of thickness of the permalloy film.

![Fig. 8](image_url)
Fig. 4, the inset (1) shows the expected broadband $S_{11}$ based on our model and in comparison with experimentally obtained $S_{11}$ response for the case of air (without sample). The resonator behaves as a band pass filter and shows resonance notches at each 1 GHz, while in Fig. 4 and its inset (2) shows closer look in lower frequency region in comparison with experiment data.

It can be observed from Fig. 4 that the experimental results cannot be completely modeled due to losses and artefacts coming from imperfect interconnections in the real setup. Fig. 5 and its inset show the modeling results of reflection coefficient changes as a function of permeability of the magnetic film. The theory above supports the statement that the reflection coefficient $S_{11}$ decreases with increasing permeability. In the practical case, the changes in permeability will come from the external magnetic bias field variations.

4. Results and discussion

4.1. Spectroscopy

In the experiments, all the measurements were performed in contact mode SMM. As a starting point of the SMM measurement, the frequency sweep from 1 to 20 GHz has been obtained; in a second step we decided to reduce the frequency bandwidth between 15 GHz and 20 GHz with a step width of 250 kHz, to procure even the smallest variations in the resonance frequency shift. The incident microwave power level was set to 0 dBm and kept the same for all the measurements. This power level is referred at the output port of the VNA, and, due to the losses and mismatches, the actual power incident on the sample, can be evaluated, roughly, around 50% of the nominal value, considering the power dissipation of the shunt matching resistor.
The measured changes in the amplitude of the reflection coefficient $S_{11}$ with respect to external fields for the RF sputtered and LPE-YIG samples as a function of frequency is shown in Fig. 6. The external magnetic field was applied perpendicular to the sample surface. The inset of Fig. 6 shows the comparison between the LPE and RF sputtered YIG film samples and $S_{11}$ values as a function of magnetic field at each resonance frequencies ($S_{11}$ minimum). The LPE sample is more absorptive, as the magnetic field vs amplitude diagram shows a difference of about 1 dB compared to the RF sputtered film, because of its large thickness and crystalline nature.

The important point to note here is that $S_{11}$ changes do not follow a linear variation with the external magnetic bias field, instead it shows a hysteretic behavior which can be related to the magnetization of the sample. Fig. 7 shows the hysteresis behavior of $S_{11}$ with respect to the external magnetic field and the inset shows the hysteresis curve obtained from vibrating sample magnetometer (VSM) data. The measured in-plane coercivity for LPE and RF sputtered YIG films from the VSM measurements are 253.29 Oe and 404.13 Oe and the magnetization values are 0.168 emu and $22.66 \times 10^{-3}$ emu respectively.

Similarly, the reflection coefficient changes have been analyzed for the permalloy thin films having different thicknesses. Fig. 8 (a) shows the resonance notches for the cases of air (empty sample holder), pure silicon substrate and permalloy films with the thicknesses of 30 nm, 50 nm and 70 nm. From Eq. (6), we know that the changes in the thickness of the film will change the total impedance of the system, which results in observing changes in $S_{11}$. Fig. 8(b) shows the changes in $S_{11}$ and the shift in the resonance frequency with respect to the thickness of the permalloy film. It also shows that the 70 nm thick film has large change in $S_{11}$ compared to 30 and 50 nm and this change also related to the surface quality of the film with the increase in thickness.

### 4.2. Imaging

Fig. 9 shows the obtained SMM image of bulk permalloy sample surface. The image has been acquired after the selection of single frequency at 19.21 GHz close to a resonance notch from the frequency sweep of 15–20 GHz. The IF band width was kept at...
500 Hz for all the images and the scan speed was 21.54 μm/s and also the image obtained without external magnetic bias condition. Fig. 9(a) shows the topography image and Fig. 9(b) shows the schematic of the bulk sample and the arrangement. Fig. 9(c) and (d) show the amplitude and the phase of the recorded $S_{11}$ signal. The topography shows locally a smooth surface but the amplitude and phase images are showing two distinct regions with large changes in $S_{11}$ signal between them of about 2 dB in amplitude and more than 10° changes in the phase signal. The profiles are showing the variations along the solid line in the images which goes through the two regions. The variations are clearly visible in $S_{11}$ signal when comparing these results to the topographical variations. This is a visible evidence that those two regions exhibit two different magnetization properties on the surface of the sample. In order to see the variations with respect to the external bias field $H$, different fields of values 225 Oe (Fig. 10(b)) and 330 Oe (Fig. 10(c)) have been applied perpendicularly to the sample surface.

Fig. 10(a) shows the image of bulk permalloy sample surface without any bias field. Fig. 10(b) as well as (c) shows the images with external field values of 225 Oe and 330 Oe, respectively. Fig. 10(e) shows the topography and its profile, which clearly indicates that there is no topographic variation and no distinct regions like in the case of $S_{11}$ signal. The images are showing that the variations with respect to external field are not high, but a very small change of about 0.08 dB from 0 Oe to 225 Oe and about 0.28 dB when changing the magnetic field from 225 Oe to 330 Oe. Fig. 10(d) shows the comparison of the profiles of zero bias condition and 225 Oe bias field condition and it shows the improvement in differentiating the two regions when applying external bias field. As the changes observed in the images are extremely sensible when applying external fields, we have also performed single point spectroscopy measurements on the two regions. To improve the signal to noise ratio, 30 dB amplification has been provided using the Dopant Profiling Measurement Module (DPMM, Keysight Technology). Fig. 10(f) shows the frequency sweep response of the two regions with respect to different bias fields. The inset shows a clear picture of the variations in both regions with respect to the bias magnetic field. This is an indication that the magnetic properties on the two regions are changing with the external field.

Fig. 11 shows the images obtained for the LPE-YIG film sample. Fig. 11(a) shows the topography containing some grains of average diameter of 1.5 μm. In order to demonstrate the magnetic properties changes on the surface of the sample, we swept the magnetic bias field: (b) 0 Oe, (c) 500 Oe, (d) 1000 Oe and (e) 1500 Oe. It is clearly visible, when increasing the magnetic bias value from zero, the surface becomes more absorptive and this effect results in a decrease of $S_{11}$ amplitude with an increase in magnetic bias. Fig. 11(f) shows the profiles of the single grain absorption with magnetic bias field. This is evidently an indication that the changes in the impedance of the tip/sample system come only from the changes in magnetic properties, i.e. from the inductive part of the impedance, but not from any capacitive changes. Actually, the magnetic bias will not produce any alterations in the capacitance part, which is related to the dielectric constant of the material and
not to the magnetic permeability.

5. Conclusions

We have demonstrated the capability of a scanning microwave microscope (SMM) system of characterizing thin and bulk magnetic materials. Specifically, we tested the response of magnetic garnets and permalloy samples looking for imaging and microwave spectroscopy in terms of reflection measurements. Qualitative and quantitative results have been discussed, with evidence for the change of the microwave absorption of the material under test when biased by means of a DC magnetic field orthogonal to the surface. The versatility proper of the SMM allows for both a fast, large area, characterization of the sample's magnetic properties in imaging mode, and also for point-wise spectroscopy mode, which provides higher accuracy and sensitivity of the minute changes in the measured parameters. In fact, the SMM tip acts as a local antenna, able to detect very small changes of the phase and amplitude of the reflected signal. An equivalent electromagnetic transmission line model of the interaction between the tip and the sample has also been proposed, to be evolved for calibration purposes and for quantitative analysis of the local permeability. We also observed the hysteretic behavior of the reflection coefficient $S_{11}$ with the external bias field. In comparison with vibrating sample magnetometry (VSM), which is a non-local measurement, SMM can provide a frequency dependent local hysteresis plot. In conclusion, exploiting the nanoscale potentiality of the measurement system, highly localized measurements at microwave frequencies can be performed, very useful in the characterization of materials and devices exhibiting respectively magnetic nanoscale response and details.

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