Near-Field Microwave Techniques for Micro- and Nano-Scale Characterization in Materials Science

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***Abstract*—In this paper, the basic principles of Near-Field Microscopy will be reviewed with focus on the micro- and nano-scale resolution configurations for material science measurements. Results on doping profile, dielectric and magnetic properties will be presented, with details on the calibration protocols needed for quantitative estimation of the dielectric constant and of the permeability.**

***Keywords*—Near-Field, Microwave Microscopy, Materials Science, Dielectric Materials, Magnetism.**

1. Introduction

Microwave Near-Field measurements gained attention during the last decade for characterizing micro- and nano-structured samples [1][2][3][4]with several possible implications for Materials Science [5][6] and even for Biology [7][8][9] or Cultural Heritage, where also THz radiation has been successfully proposed [10][11].

The measurement technique is based on the utilization of a probe for scanning a surface in contact or non-contact mode, depending on the setup, on the required response and on the sensitivity.

Two main aspects favored the development of Near-Field analysis for local measurements at micro- and nano-size level: (i) the penetration of microwaves, thus allowing the possibility for surface and sub-surface characterization [12], and (ii) the high lateral resolution, dominated by the probe size and not by the wavelength [13].

The possibility for obtaining the above information opened unprecedented possibilities in studying frequency dependent local properties of materials and for imaging the surface or buried details.

Historically, small-size directive antennas as well as purposely-machined tips have been used as pioneering tools [14]. Recently, micro-machined tips have been furtherly developed for homemade setups with micrometric resolution [15], and nano-size tips are commercially available and electrically matched for microwave signal processing [16][17].

Presently, imaging and spectroscopy are both possible, making use of typical microwave engineering setups coupled to purposely-designed tips connected to an Atomic Force Microscope (AFM), measuring microwave scattering parameters [18]. Actually, amplitude and phase of reflected and transmitted signals can be converted in the topography of the inspected surface or give local information on dielectric, magnetic and conducting properties of the investigated sample. Sub-micron depths are currently possible in commercial installations, with perspectives in deeper probing by means of different tips or setup arrangements.

In this paper, State-of-Art for the Near-Field Microwave Measurement Techniques will be briefly reviewed, with advances in the study of dielectric and magnetic properties, and with focus on quantitative analysis and on the necessary calibration protocols based on measurements as well as on modelling approaches.

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2. Basic Principles of Microwave Microscopy

A. General setup

Near-Field Microscopy at microwave frequencies is based on measurements of amplitude and phase recorded by means of a Vector Network Analyzer (VNA) System, or its modern version, the Precision Network Analyzer (PNA) currently commercialized by Keysight Technologies, even if there are in principle no restrictions on the kind of instrument to be chosen. The interaction with the sample under investigation is provided by a probe, which is a tip with sub-micron to nano-sensitivity, able to detect surface and sub-surface details related not only to morphology but also to the material properties. A signal due to the coupling with the sample is re-elaborated to get information about the material (doping profile, dielectric constant, magnetic permeability, ...). In between the tip and the Network a properly designed system with precise mechanical control and high frequency circuits is used for guiding the microwave signal to be processed. An example of the full system is shown in Fig. 1.

The characterization technique, originally designed for reflection type measurements, has been recently evaluated for transmission purposes [19]. Other groups studied the same problem by using a modified scanning tunneling microscopy (STM) configuration [20]. The natural evolution of approaches based on both reflection and transmission will be the development for tomography and 3D imaging, with perspective nano-meter resolution [21].

Fig. 1. Setup of the Scanning Microwave Microscopy technique commercialized by Keysight Technologies. A PNA is connected to the SMM and a nose-cone is used for hosting the cantilever interacting with the sample and the microwave circuitry.

B. Probes and Probe-Sample Interaction

Several probes have been experienced in the past (aperture coupled and tips). For a review on this topic see [1]. Actually, the main differences among the two kind of probes are in resolution and power handling. In fact, an aperture coupled probe can handle in principle more power, but the microwave beam emerging from the end of the waveguide will suffer because of lower definition due to leakage. On the other hand, a tip guarantees a higher resolution, determined roughly by the size of its end, but the transmitted power is lower, limiting the maximum depth of the radiation inside the measured sample. In the case of tips, the coupling is measured by means of the impedance (real and imaginary part), from which the post-processing will give back the material quantity to be determined. In the case of a purely dielectric specimen, a capacitive contribution is expected, which has to be corrected with the losses. Using equivalent circuit approaches (valid within the microwave range) mixed with electromagnetic evaluations, the dielectric constant ε and the microwave losses tanδ can be inferred. They will be related to an equivalent capacitor and resistor respectively.

One more advantage in using a Network Analyzer is the possibility to obtain a spectroscopic information, i.e. to measure the frequency dependence of the material characteristics. To understand better the modeling problems, in Fig. 2 the schematic diagram of the tip interfacing the surface of the sample is shown, together with the relevant geometrical quantities to be used in the modeling.

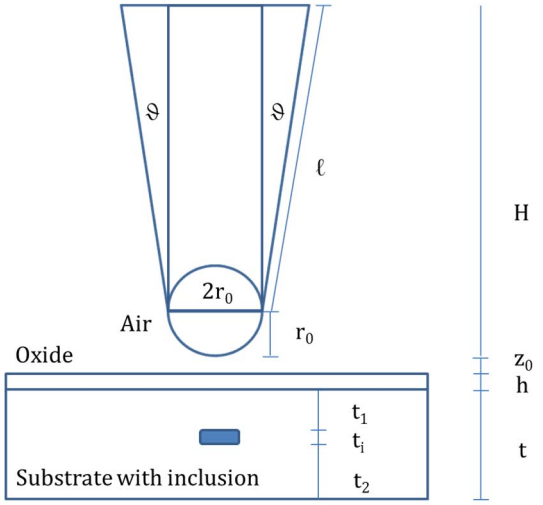


Fig. 2. Schematic diagram for modelling the interaction between a metallic tip and an oxidized substrate with a buried inclusion.

By assuming a capacitive contribution only, the tip and its shape as well as the cantilever hosting the tip are responsible for the lumped capacitors useful for defining the equivalent circuit. It is worth noting that the tip, being a radiating element, will contribute with leakage too, and sometimes first order approximation show a lack of accuracy in quantitative results. The justification for using lumped is that the involved wavelengths are longer than the typical size of the probes. So far, formally the measured impedance is simply $Z=1/\omega C$, but several efforts are needed to evaluate C with high precision. In particular, according to fig. 2, the tip is usually composed by a half-sphere and a metallic cone. Therefore, in the equivalent circuit of the tip the contribution of the existing capacitance between the lateral surface of the cone and the ground plane should be included. An electrostatic model based on an analytic approach has been recently presented for the evaluation of this capacitance [22].

To enhance the sensitivity of the measurement, often resonating elements are integrated with the setup. In this case, the frequency of resonance will be chosen according to the optimum electrical matching, and amplitude or phase can be selected for the best imaging results [23][24].

Alternatively, phase matching components can provide an increased sensitivity by means of an external tuning and/or using an interferometric setup [25][26].

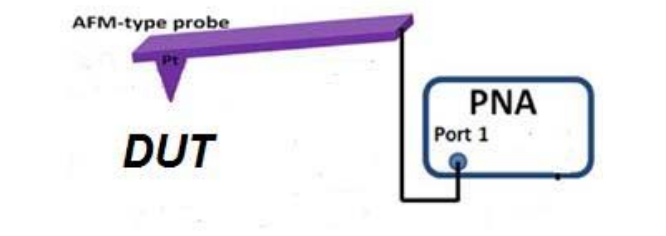
C. Calibration Issues and Doping Profile

Calibration is an essential procedure in microwave engineering measurements. Owing to the frequency dependence of material and device characteristics, the calibration is based on hardware standards and on a software protocol, to normalize the recorded measurement with respect to the plane used as a reference. In this way, the measurement is de-embedded from the un-wanted contributions between the Network port and the device (cables, interconnections and other paths and discontinuities).

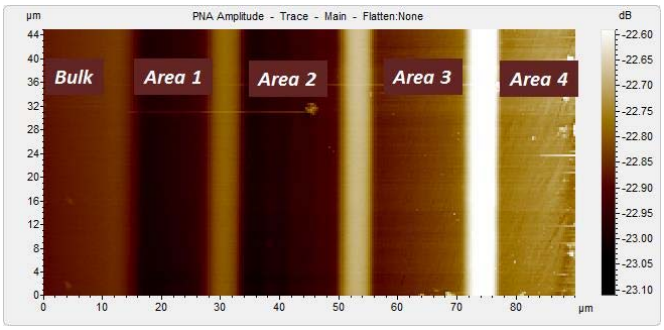
The above procedure is well established when on-wafer or connectorized devices are measured. In particular, broadband standards emulating an ideal short, open, load and a throw are used, accounting for their frequency dependence, to obtain a so-called SOLT calibration. Alternatively, transmission lines purposely designed onto the same wafer where the devices are manufactured can be realized, with Throw, Reflect and Line configurations to get the obtain the TRL calibration.

For microwave microscopy, a completely different perspective is needed, because the measurement is more close to an “antenna” characterization, with a micro-tip emitting microwave radiation. For this reason, only under specific circumstances hardware standards can be utilized [27][20], whereas often software de-embedding and post-processing are vital tools for obtaining a calibrated measurement [28][29][22]. The cantilever hosting the tip is itself a source of impedance (stray capacitance), and it is not negligible because of its wide area with respect to the small size of the tip. This is an additional reason for combining hardware and software tools to obtain a de-embedded measurement. Moreover, if a contact measurement is performed, the tip interacting with the sample will be subjected to wearing during the characterization, with a consequent dynamic modification in the size of the tip end. Such a situation will contribute to change quantitative results, as they are tightly related to the interaction. Imaging will not suffer so much, but spectroscopy will be altered in a sensitive way. For instance, one of the first and important applications for the microwave microscopy developed during the past few years has been the doping profile. In this case, following the first attempts to get quantitative information on the local capacitance of semiconductor samples [30], further studies demonstrated the possibility to account for the tip wearing for obtaining a correct evaluation of the doping level [31]. This method was successful even in comparison with previous attempts, focused on calibrating the measurement by using approach curves and interpolating data at different quotes with respect to the surface of the sample [5]. In fact, the measurement of doping profile is a very good example of a procedure which needs: (i) a known value of doping to be used as a reference in the area of measurement, (ii) a theoretical approach giving the basic formalism for individuating the quantities to be evaluated, (iii) a criterion accounting for the change of the properties of the tip during the scanning.

Following the above approach, nano-scale evaluation of the doping profile was possible in a range between 10^{14} and 10^{19} cm^{-3}. A full example of the method and of the results obtained by means of the tip wearing calculation is shown in the following Fig. 3.



(a)



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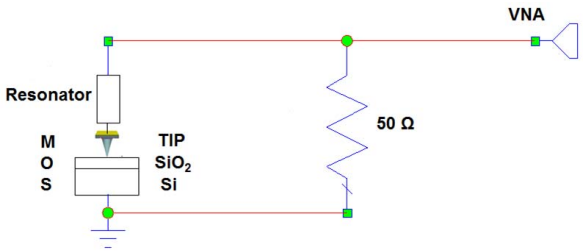
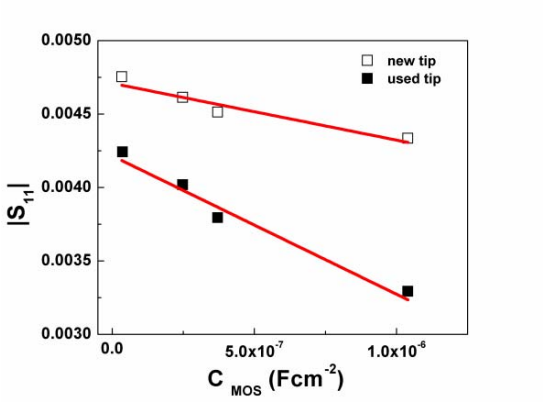


(c)

$$|S_{11}| = \frac{\omega_{RF} Z_0 (\pi R_{eff}^2)}{2} C_{MOS} + \frac{\omega_{RF} Z_0 C_{stray}}{2}$$

(d)



(e)

Fig. 3. Evaluation of the tip wearing for calibration purposes in doping profile measurements. In (a) the schematic of the interaction is given for reflection type measurements of a device under test (DUT). In (b) it is shown the amplitude of the signal experimentally recorded and the image of the scanned surface. Different colors indicate different surface properties of the sample. In (c) the experiment is schematized with an equivalent circuit. In (d) the equation governing the interaction is given, with evidence for the MOS and for stray capacitance contrinutions, which can be de-embedded thanks to the expected linear dependence, to get results shown in (e), where a fresh and a used tip are compared, leading to a radius of 65 nm for the new tip and 100 nm for the used one.

3. Characterization of dielectric materials

Spectroscopic analysis of dielectric materials is always necessary when the frequency dependence of the dielectric constant is required for a correct design of broadband high frequency devices. Substrates classically used for microelectronic applications do not present a significant change in the frequency response up to 100 GHz (like alumina or silicon and gallium arsenide). On the other hand, local technological developments to get capacitors or transistors with a distribution of doping and ε-values could be better controlled by means of local measurement techniques, to determine not only the topography, but also the high frequency response. In this case, phase and amplitude help both for obtaining the required contrast for imaging purposes and the value of ε as a function of frequency. For micro-scale characterization of dielectric materials and buried metallization or inclusions, the measurement can be local, with no scanning, as it is shown in the setup of Fig. 4. In this case, two commercial flange connectors interface a multilayer composed of SU-8 polymer spinned onto a metallized or not metallized silicon wafer. Such a transmission measurement, with an exact knowledge of the geometry and of the known materials, is useful for determining the dielectric constant of the unknown material. Commercial pins, chemically etched to obtain resolutions from few microns down to one micron can give a more precise local spectroscopic information.

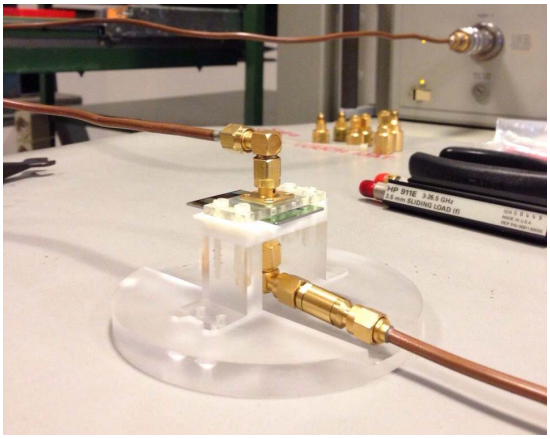
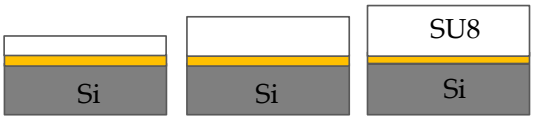


Fig. 4. Setup for Near-Field microwave measurements of planar samples, to investigate dielectric constant properties and buried metallizations without scanning. Flange connectors are used for areas as wide as 500x500 μm^2, down to few μm^2. A two-port arrangement has been utilized to improve sensitivity for buried details. In principle, even asymmetric solutions can be proposed, with a radiating element illuminating the sample from the back side, and the receiving antenna with smaller size on the top. The resolution, as well as the depth for the microwave radiation, is dominated by the tip size.

No.	Material	Thickness [μm]	Metallization (1000 Å Cr)
1	SU-8 2002 on Si	530 + 3.2	NO
2	SU-8 2002 on Si	530 + 3.2	YES
3	SU-8 2007 on Si	530 + 7	NO
4	SU-8 2007 on Si	530 + 7	YES
5	SU-8 2007 on Si	530 + 15	NO
6	SU-8 2007 on Si	530 + 15	YES
7	Si only	530	NO
8	Si only	530	YES

(a)



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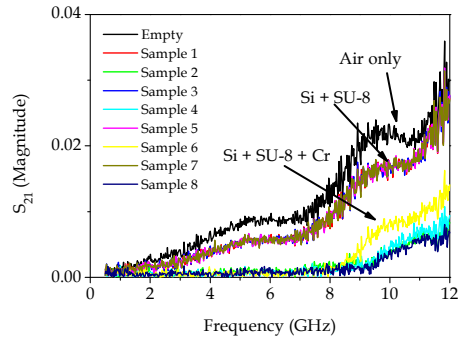

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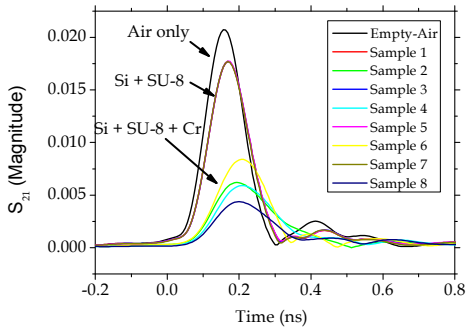

(d)

Fig. 5. (a) Table with material and geometry characteristics of the measured layered samples. SU-8 2002 and 2007 are the two commercial names of polymeric material spinned on silicon. (b) Schematic of the samples, with different thickness of polymeric material on the top of a silicon wafer and Cr in between optional for some of them. (c) Transmission of the microwave signal through the samples prepared for the experiment, in linear scale, as a function of the frequency. (d) Transmission of the signal through the samples, in linear scale, as a function of time, using the time domain option of the Network Analyzer. The time-peak is an indication of the discontinuity position.

Using the results in Fig. 5 and the formulation given in [32], it was possible to get to distinguish metal from dielectric materials and to obtain the ε-values of the measured materials.

A first important indication coming out from Fig. 5 is that a 100 nm metallization can be clearly detected by means of a transmission setup making use of both amplitude and time domain measurements. Secondly, different materials can be grouped to get a «family» answer depending on the nature (metal or dielectric) of the inclusion. Even phase, sometimes more sensitive than amplitude, can be used for the characterization, leading to the results presented in Fig. 6, where the phase as a function of time is shown, and the same consideration about the possibility to group materials of the same kind is valid.

Distinguishing materials very different between them, like paper, alumina, silicon, … is quite easy looking to the amplitude as a function of frequency, but materials belonging more or less to the same class (ceramics for instance) and with the same thickness need to be analyzed carefully because they are characterized by having comparable losses as well as phase response. For this reason, a method based on an analytical

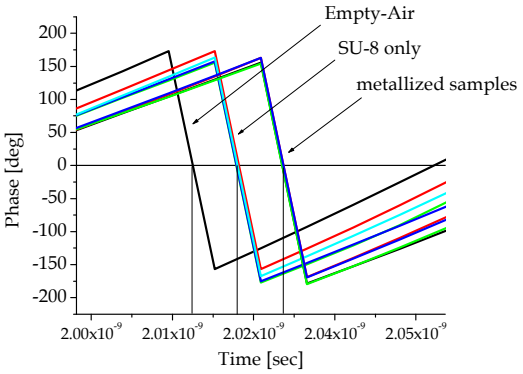

Fig. 6. Plot with the phase change in time domain for SU-8 samples and for the metallized ones covered by SU-8. The arrows give evidence for three distinguished groups of data: (i) a curve typical of the setup without sample (first trace evidenced on the left), (ii) a second family of curves related to the samples where SU-8 only covers the silicon wafer, and (iii) a third group of curves having a more pronounced shift, typical of the metallized samples.

approach has been developed, which utilizes a concept of "characteristic frequency", useful for de-embedding the selected layer and to evaluate the dielectric constant [33].

4. Measuring magnetic materials

The measurement of magnetic materials introduces a further parameter to be accounted, which can be represented by the inductive response of the sample. In fact, using a lumped element approach, always valid up to the beginning of the millimeter wave range, i.e. close to 30 GHz, the equivalent circuit of a purely

magnetic material can be represented by and inductance, and a resistor will be placed in parallel to account for the losses. In this framework, in analogy with the dielectric materials, the permeability μ and the loss tangent $\tan(\delta_\mu)$ can be inferred, relating them to an equivalent inductor and to a resistor for the magnetic loss contribution. It has to be stressed that the formalism for magnetism is coherent with that for dielectric materials, where $\mu=\mu'+i\mu''$ substitutes $\varepsilon=\varepsilon'+i\varepsilon''$ and $\delta_\mu=\mu''/\mu'$ is conceptually equivalent to $\delta_\varepsilon=\varepsilon''/\varepsilon'$. When contact measurements are performed, an inductor only can be used for the equivalent circuit. The situation is a bit more complicated when non-contact measurements are considered, and the air capacitance between the surface of the sample and the tip end must be included. Moreover, magnetic samples are sensitive to an external DC magnetic field, contributing to the formation of domains and domain walls, and to local ferromagnetic resonance effects. To account for all of these contributions is not a trivial task, and while imaging is always related to local adjustments for increasing the sensitivity of the measurement, a quantitative result is currently not obvious. On the other hand, local magnetic properties are very important not only for basic research, to measure and model static and dynamic magnetic properties (remanent magnetization, anisotropy, resonance, …) but also for getting information related to magnetic spin control under proper DC biasing conditions. This opens a new window on the spintronic applications, where spin dynamics is essential for magnetic information processing at the nano-scale.

In our group, preliminary results on magnetic properties have been obtained by measuring the domains of yttrium iron garnet (YIG) magnetic films when biased by a DC magnetic field and the response for permalloy thin films. The capabilities of the SMM have been tested for analyzing and imaging local magnetic properties of the materials under test at the nanoscale. The analyses have been performed by measuring both amplitude and phase of the reflected microwave signal. The changes in the reflection coefficient S_{11} have been 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. In particular, 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 as thick as tens of nanometers have been characterized. An equivalent electromagnetic transmission line model has been used to for the quantitative analysis of the local magnetic properties. Hysteretic behavior of the reflection coefficient S_{11} with an external bias field has been also observed. 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.

An example of the spectroscopic sensitivity of the SMM when measuring permalloy thin films is shown in Fig. 7 [33].

5. Conclusions

In this paper, we reviewed the general performances of Near Field Microwave Measurements by using scanning probe setups. Specifically, a micro-scale characterization technique was used for checking dielectric

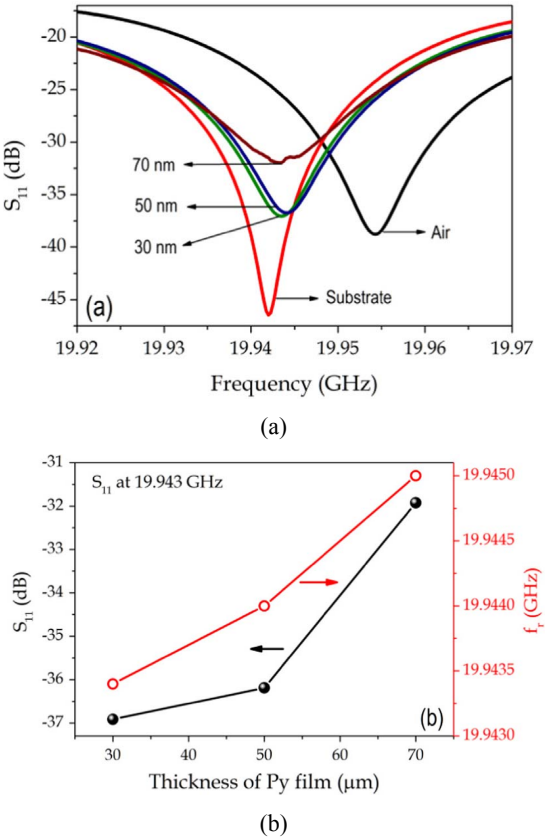

Fig. 7. (a) Experimental reflection coefficient for air (empty sample holder), silicon substrate and permalloy films with thicknesses 30 nm, 50nm and 70 nm at zero bias condition. (b) Measured S_{11} variations and resonance frequency as a function of thickness of the permalloy film [33].

properties of planar samples, including those having buried metallization. Nano-scale measurements have been performed by means of a Scanning Microwave Microscope (SMM) to get information on the semiconductor and magnetic properties of different materials. In particular, doping concentrations have been measured in the range between 10^{14} and 10^{19} cm^{-3}. Moreover, preliminary measurements on magnetic samples demonstrated the possibility for determining local changes of the properties when subjected to a DC magnetic bias. In conclusion, Near Field Microwave Measurements are now demonstrated to be a powerful tool for obtaining complementary information with respect to classical microscopy techniques, with appealing perspectives in measuring different material properties from micro- to nano-scale, and for determining the presence and nature of buried structures.

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