

Sub-surface Imaging of Electromagnetic Properties of Materials Using Near-Field Microwave Microscopy

María F. Córdoba-Erao, *Student Member, IEEE*, and Thomas M. Weller, *Senior Member, IEEE*

Abstract—This report summarizes the main outcomes of the research project awarded with the 2015 MTT-S Graduate Fellowship, General Category. The research objective of this project is to design and build a coaxial transmission line resonator-based near-field microwave microscope (NFMM) able to characterize with spatial resolution in the order of micrometers flexible materials coated with insulating films. The microscope is equipped with a quartz tuning fork-based distance-following feedback system in order to perform non-contact and non-destructive imaging of the samples. Additionally, we present the NFMM imaging of flexible PDMS-Ba_{0.55}Sr_{0.45}TiO₃ composite coated with a 10 μm thick parylene-C layer which reveals concentration of low permittivity regions on one side of the sample.

Index Terms—Microwave microscopy, near-field, permittivity, sub-surface imaging.

I. INTRODUCTION

NFMM is a technique that has been employed to measure the permittivity of insulating materials, sheet resistance of metallic thin films and dopant concentrations in semiconductors with sub-wavelength resolution. Changes in the resonant properties of the probe can be correlated with the material properties using semi-empirical methods and/or theoretical models based on resonant perturbation theory. NFMM has also been used to detect the topography of metallic features coated by a conductive layer [1] and insulating layer [2]. Feature identification has been carried out by detecting changes in the output signals of NFMM (mag(S11), phase(S11), voltage) as the NFMM scans over a homogenous, uniform and known film containing the buried features; any detected change in the output reading represents a change in the buried material. In [2], a coaxial resonator-based NFMM operating in non-contact mode at a frequency of 763 MHz is used to image MMIC elements embedded at 0.8 μm and 8.6 μm below the surface using probe tips of radius of 10 μm and 50 μm. Recently, the subsurface imaging characteristics of a NFMM operating in transmission mode were presented in [3].

In the aforementioned works, detection of metallic features

embedded in metallic and insulating films using NFMM has been discussed. However, there is a lack of information on the study of the dielectric constant of coated insulating samples which can be of interest for the characterization of ceramic composites. Polydimethylsiloxane (PDMS) can be loaded with ceramic powders such as Ba_{0.55}Sr_{0.45}TiO₃ and MgCaTiO₂ in order to develop high-k and low-loss flexible polymer-ceramic substrates suitable for microwave applications such as antennas, filters and baluns. To the best of the authors' knowledge, this is the first work reporting the non-contact imaging of flexible composites through NFMM. This application presents a major challenge since the contact point with the sample cannot be clearly determined, due to the lack of a well-defined boundary on the region where the tuning fork interacts with the surface; the absence of a well-defined boundary subsequently reduces the effectiveness of distance following feedback system of the NFMM. In contrast, it has been observed that a parylene-C layer deposited on the flexible composite allows the distance-following feedback system to determine the contact point with the sample within a distance of 100 nm [4]. In this work we present the imaging of PDMS-Ba_{0.55}Sr_{0.45}TiO₃ 49% coated with a 10 μm thick parylene-C layer. Additionally, we briefly discuss the design of the coaxial resonator-based NFMM used for the characterization.

II. DESCRIPTION OF THE PROJECT

The schematic diagram of the 5.0 GHz coaxial transmission line resonator-based probe using in the NFMM is presented in Fig. 1. The resonant microwave probe is a half-wavelength coaxial transmission line resonator operating at 5 GHz that is critically coupled to the VNA through an adjustable gap and a feed line. The resonator and feed line are fabricated using semi-rigid coaxial cable. From the inset, it can be seen that a short section of the copper inner conductor of the resonator is removed and replaced with a stainless steel tube with length $L_s = 3$ mm, in which a tungsten wire with length of 4.5 mm and radius of 100 μm is inserted. The end of the wire is sharpened down to a few microns using electrochemical etching prior inserting it in the stainless steel tube. In order to operate the NFMM in non-contact mode and to map simultaneously F_r , Q , and surface topography, the NFMM employs a quartz tuning fork-based distance following feedback system. NFMM measurements were performed at a

M. F. Córdoba-Erao and T. M. Weller are with the Department of Electrical Engineering, University of South Florida, Tampa, FL, 33620 USA. (e-mail: maria15@mail.usf.edu).

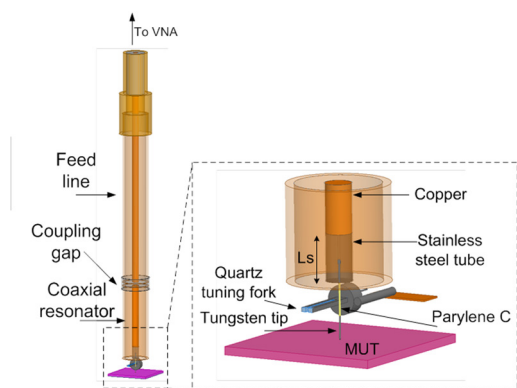


Fig. 1. Schematic of the coaxial-based probe. The inset shows a close-up view of the probe tip and the quartz tuning fork used as a distance sensor in the distance following feedback system.

tip-sample distance of 100 nm. The tungsten probe tip used has a radius of 10 μm . Details on the NFMM experimental setup and simulations are presented in [4].

NFMM imaging of the polymer-ceramic composite made of PDMS-Ba_{0.55}Sr_{0.45}TiO₃ 49% coated with a 10 μm thick parylene-C layer was carried out over an area of 50 μm x 50 μm in steps of 2 μm at a constant tip-sample distance of 100 nm. The dielectric constant and loss tangent of the composite were separately measured using a cavity resonator and found to be 22.7 and 0.032 at 4.26 GHz, respectively. During the scan, F_r and surface topography images of the sample were acquired simultaneously. From Fig. 2(a) and 2(b), it can be observed that there is no direct correlation between topography (surface height) and F_r which indicates that measured variations in F_r are a consequence of localized changes in the permittivity of the composite. Surface topography image 2(a) reveals patterns of parallel lines which are marks from the mold used for sample preparation. Microscope images of the sample and mold confirm this observation as shown in 2(c). From 2(b) it is clear that F_r , and therefore the permittivity distribution is not constant over the scan area and that a concentration of lower permittivity (higher F_r) is observed on the left side of the sample. This region could represent PDMS that was not effectively mixed with the Ba_{0.55}Sr_{0.45}TiO₃ ceramic particles as can be observed in the SEM micrograph shown in 2(d). It can be noted that a \sim 50 μm thick PDMS region is formed on top of the sample and that PDMS-ceramic agglomerates are distributed across the sample. The average, minimum and maximum frequency observed in 2(b) are 4.9889 GHz, 4.9867 GHz and 4.9904 GHz respectively. These values translated to permittivity after NFMM calibration are $\epsilon'_r = 8.37$, $\epsilon'_r = 11.78$ and $\epsilon'_r = 6.63$, respectively.

The discrepancy can be explained by the fact that the cavity resonator technique extracts an effective permittivity over a volume of about 5 cm x 5 cm x 0.5 mm whereas the NFMM study volume is determined by the scan area 50 μm x 50 μm and the estimated penetration depth 30 μm .

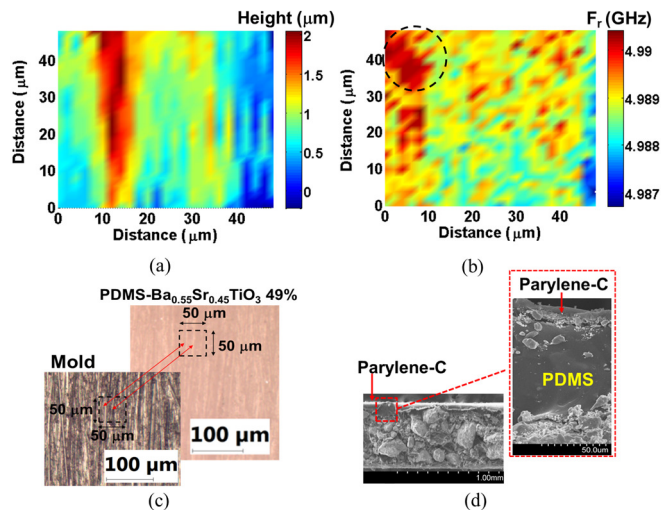


Fig. 2. (a) Surface topography and (b) F_r image of PDMS-Ba_{0.55}Sr_{0.45}TiO₃ 49% coated with a 10 μm thick parylene-C layer. (c) Microscope image of PDMS-Ba_{0.55}Sr_{0.45}TiO₃ 49% and stainless steel mold. (d) SEM micrograph of the cross section of PDMS-Ba_{0.55}Sr_{0.45}TiO₃ 49% coated with a 10 μm thick parylene-C layer. Tip-sample distance is 100 nm for (a) and (b).

III. CAREER PLANS, PUBLICATIONS, FELLOWSHIP IMPACT AND IMS IMPRESSIONS

My career plan is to gain experience in the RF industry and after a couple of years return to academia. Two journal papers describing the presented project have been submitted to Review of Scientific Instruments and Journal of the American Chemical Society.

I want to express my sincere gratitude to MTT-S for the economic support and for sponsoring my attendance to IMS 2015 in Phoenix, AR. In addition to attending the technical sessions in IMS-2014 and IMS-2015, I had the opportunity of networking with colleges and learning from their experiences working in industry and academia. Additionally, I participated in the Project Connect initiative where I interacted with undergraduate students and shared my experiences as graduate student.

REFERENCES

- [1] C. Plassard, E. Bourillot, J. Rossignol, Y. Lacroute, E. Lepleux, L. Pacheco, *et al.*, "Detection of defects buried in metallic samples by scanning microwave microscopy," *Physical Review B*, vol. 83, p. 121409, 2011.
- [2] J. D. Chisum and Z. Popovic, "Performance Limitations and Measurement Analysis of a Near-Field Microwave Microscope for Nondestructive and Subsurface Detection," *IEEE Transactions on Microwave Theory and Techniques*, vol. 60, pp. 2605-2615, 2012.
- [3] A. O. Oladipo, A. Lucibello, M. Kasper, S. Lavdas, G. M. Sardi, E. Proietti, *et al.*, "Analysis of a transmission mode scanning microwave microscope for subsurface imaging at the nanoscale," *Applied Physics Letters*, vol. 105, p. 133112, 2014.
- [4] M. F. Cordoba-Erao and T. Weller, "Near-Field Microwave Microscopy for Non-Destructive Characterization of Polymer-Coated Samples " Submitted to *Review of Scientific Instruments*.