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Microwave Characterization of Dielectric Material at 2.45GHz

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ABSTRACT

We describe a near field microwave microscopy method for characterization and rapid non-destructive imaging of dielectric materials samples at 2.45 GHz. This system is composed of a probe coupled to a dielectric resonator. It is used for two purposes: first to characterize dielectric samples with accuracy and high spatial resolution, knowing that they do not have predetermined forms but a small plane surface; and second, to draw dielectric cartographies for flat samples, with a 100 µm resolution.

Key words: matrial characterization, near field microscopy, dielectric resonator, dielectric material, dielectric constant.

1. INTRODUCTION

Several methods of characterization exist, depending on the nature or the geometrical form of the material to be analyzed [1]. Most of these techniques need homogeneous samples, with particular geometrical forms and dimensions; they give then only an average permittivity of the whole sample, making it impossible to know if the substrate shows an inhomogeneous distribution of permittivity [2]. This includes the parallel-plate resonator technique. Methods based on dielectric resonators also allow the characterization of dielectric layers, even if the obtained values are averaged over an area of the order of the wavelength linked to the resonator resonance [3]. They measure a weighted average over large areas of the sample in centimeters scale. In the case of inkjet technology, the used technology for printing patterns is of the order of a millimeter, the usage of this kind of characterization technique is not relevant.

To overcome these limitations, several methods in near field microscopy have been developed for non-destructive characterization of non-homogenous dielectric samples that do not have a well-defined geometrical form. Near field microscopy was suggested by Synge [4] and demonstrated by Sooho for microwave frequencies [5].

Several research groups have developed near-field scanning microwave microscopy with different designs and for different applications [6, 7]. However, the study of precision and sensitivity in these methods for small variations in dielectric constant was not accurate and complete. C.Gao presented in his article [7] some measured values of permittivity for different samples. The error rate between measured values and reported values was significantly high (10% in average).

In this article, we propose a specific calibration technique and an optimization method in order to increase the sensitivity of the proposed characterization device towards any variation in the physical characteristics of samples. We also present complex permittivity measurements of different samples of small sizes at 2.45 GHz and particularly a dielectric material printed on Alumina substrate by inkjet technology.

2. THEORY

When an object (sample) is placed near the probe coupled to a dielectric resonator, the resonance frequency f_r and the quality factor Q_0 of the resonator are affected by the presence of this sample. These variations (Δf_r and ΔQ_0) depend primarily on the physical properties of the sample, the size and geometry of the probe as well as the distance between the probe and the sample [7].

Various analytical models have been used to describe the near field microscopy response. The image charge model suggested by C.Gao for a coaxial resonator [7] remains the most widely used as an analytical model. The frequency and quality factor shifts, as predicted by the model when the probe tip is in contact with a bulk dielectric sample, are linked to permittivity and loss tangent by equation 1(a,b) :

$$\frac{\Delta f_r}{f_r} = \frac{f_r - f'_r}{f_r} = A\left(\frac{\ln(1-b)}{b} + 1\right) \text{ and } b = \frac{\varepsilon_r - 1}{\varepsilon_r + 1} \quad (1a)$$

$$\Delta\left(\frac{1}{Q_0}\right) = \frac{1}{Q_0} - \frac{1}{Q'_0} = -(B + \tan\delta)\frac{\Delta f_r}{f_r} \quad (1b)$$

where f_r and f'_r are the resonance frequencies with and without sample respectively, ε_r is the relative permittivity of the sample, Q_0 and Q'_0 are the unloaded quality factors in the presence and absence of sample respectively. A and B are calibration constants determined by measuring standard samples, with well-known dielectric constant and loss tangent at the used frequency.

In this article, we use the analytic model suggested by C.Gao and apply it on a device that uses a dielectric resonator. Our device is shown in Figure 1. It is composed of a dielectric resonator (ε_r = 78, tan δ = 3.27 10^{-4} at 2.5 GHz) having a diameter of 15 mm and a height of 7 mm. It is placed on a Teflon support having a height of 15 mm. The resonator and the support are placed in a cylindrical copper cavity having a diameter of 53 mm and a height of 35 mm. A 50 ohm

impedance coaxial cable is opened at its both ends: the first end represents the probe with a 70 µm diameter tip and the other end has a loop form that magnetically couples the probe with the dielectric resonator. This coupling is optimized for more sensitivity towards any variation in the physical characteristic of the sample.

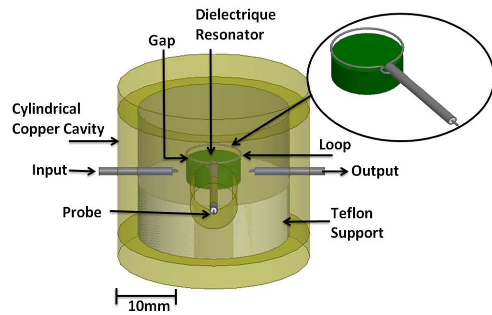

Figure 1: Schematic structure of the device.

2.447 GHz is the frequency measured by a vectorial network analyzer of the $TE_{01\delta}$ mode of the resonator when no dielectric samples are close to the measurement tip. 2.444 GHz and 2.44 GHz are the frequencies measured when a sample of FERRO A6M ($\varepsilon_r = 5.6$) and Alumina 97.9% ($\varepsilon_r = 9.7$) are close to the tip as shown in Figure 2. At this frequency, traditional methods need large samples (>100cm^2) with arbitrary form (substrate, cylinder, resonator...).However, the proposed solution only needs small size elements (in our case 1mm^2 is sufficient) with non-uniform geometry.

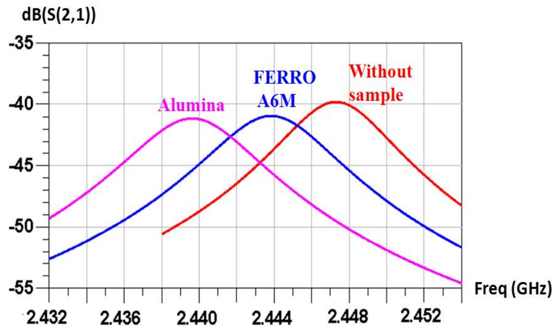

Figure 2: Measured transmission coefficient S_{21} of the dielectric resonator without sample and with FERRO A6M and Alumina samples.

3. CALIBRATION TECHNIQUE

Our measuring system is composed of a vectorial network analyzer and a controller connected to a computer for the monitoring of micrometric displacement of the sample and the probe; this is done by means of three motion devices (Figure 3): two to control the displacement of the sample on X-Y axis and the third to control the displacement of the probe on the Z axis.

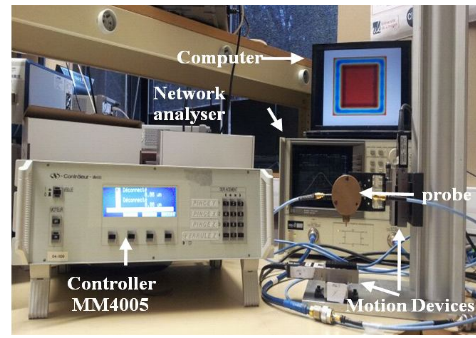

Figure 3: Measuring system

The control of the probe-sample distance can be achieved via several means such as shear force, capacitance, photon tunneling and electron tunneling [8]. In our measurement system, we use a different technique for probe-sample distance control. This technique offers several advantages: it's a low-cost technique and it is simple to manipulate. This technique is based on current control. The first step is to use an Al_2O_3 substrate (surface=4 mm^2, thickness=0.4 mm) with a 100 nm thick chromium layer on his top surface. Then a multimeter is connected between the coaxial probe tip and the chromium layer as shown in Figure 4.

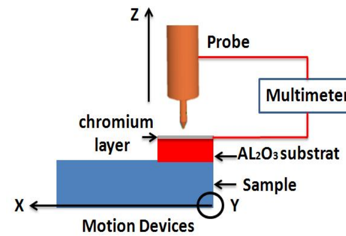

Figure 4: Current control.

The probe comes down in the direction of the sample with a 1 µm step until there is a short circuit, meaning there is contact between the probe and the chromium layer. At this moment, the gap between the tip and the sample to be characterized is known to be 400 µm (thickness of the Al_2O_3 substrate). After this we pick up the tip, the sample is moved by the motion devices along the axis X and Y and the probe is back down by around 400 µm to be just in contact with the sample. The movement of the tip, on the last microns, is controlled by monitoring the resonant frequency shift.

4. OPTIMIZATION OF THE COUPLING BETWEEN THE DR AND THE PROBE

If we consider equation 1 (a,b) we can clearly see that we have to calculate the constants A and B in order to determine the complex permittivity of a sample. These constants are determined by measuring the resonant frequency of a standard sample with a well-known complex permittivity at the used frequency, we take an Alumina substrate Al_2O_3 as a standard sample. We obtain by the resonant cavity method (SCR): $\varepsilon r = 9.7 \pm 0.08$ and $\tan\delta = 7x10^{-4} \pm 0.4x10^{-4}$. Having A and B, we are now able to determine the permittivity from a frequency

and quality factor shift obtained when a sample is placed in front of the probe.

A study is carried out on the coupling between the dielectric resonator and the coaxial cable loop. We can see that constant A in equation (1a) depends on the permittivity of the standard material and frequency offset in the presence of this material. The frequency shift increases as the value of A increases. For the same standard material, the value of A can increase if we have a stronger coupling between the probe and the dielectric resonator. In our case the coupling depends of the gap between the coaxial cable loop and the dielectric resonator. The closer is the loop to the dielectric resonator, the greater is the coupling. This relation is given by the equation 2.

$$A= (-0.38G+2.7)*10^{-3} \quad (2)$$

where G is the gap (mm) between the loop and dielectric resonator. We have a linear relation between A and the gap.

For each value of A we can plot the curve representing the frequency shift as a function of permittivity based on equation (1a). As shown in Figure 5, we can clearly see that the greater is A (smaller gap between the DR and loop), better the sensitivity over small variations in dielectric constants is.

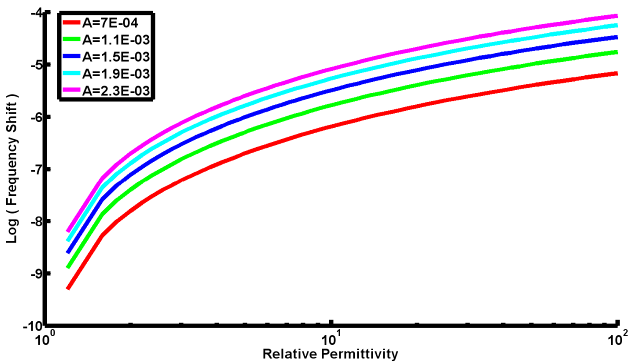

Figure 5: Variation of frequency Δf_r (GHz) as a function of the permittivity for different value of A.

With a strong coupling (gap<1 mm) between the metal loop and dielectric resonator, a strong degradation of quality factor and a saturation of the offset frequency Δf_r are observed. Indeed the experimentally extracted relation between the quality factor and the gap is a linear function (equation 3). The closer is the loop to the dielectric resonator, the lower is the quality factor.

$$Q_0=315G+231 \quad (3)$$

As a result, we decide to choose two configurations of coupling: the first configuration is a strong coupling dedicated to the measurements with high sensitivity of the samples permittivity (high Δf_r). This configuration improves the accuracy of the characterization technique even with small variations of permittivities. The second configuration is a weak coupling, and is best suited for the characterization of the samples loss tangent. This configuration can maximize the unloaded quality factor of the resonator and consequently the sensitivity of the probe when measuring loss tangent.

5. CHARACTERIZATION OF MATERIALS

After optimizing the gap in order to increase the sensitivity of the system to complex permittivity measurement, we characterized several dielectric samples having non-homogeneous forms and different dimensions as shown in Figure 6. The table 1 shows the permittivity and the loss tangent (with uncertainties) at 2.45 GHz for different samples. The uncertainties on ε_r are calculated from the uncertainty of VNA and measurement devices: (1%), and the uncertainty on previous measurement of our Alumina sample used to calculate constant A (eq: 1a: ~0.5%). The uncertainty on the measurement of the reference Alumina sample leading to the determination of B (eq: 1b) leads to an estimated error of 5% on tan δ.

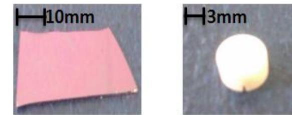

Figure 6: Example of Dielectric samples: (a) Silicon, (b) BMT

Material	ε_r (measured)	ε_r (reported)	$\frac{\Delta\varepsilon r}{\varepsilon r}$	tanδ (measured)	tanδ (reported)	$\frac{\Delta tan\delta}{tan\delta}$
Fused silica	3.77	3.78 at 10 GHz [9]	±1.3%	———	$1.4x10^{-4}$ at 10 GHz [9]	———
Ferro A6M	5.5	5.6 at 10 GHz*	±1.4%	$0.85x10^{-3}$	$1.05x10^{-3}$ at 10 GHz*	±4.8%
Silicon	11.5	11.69 at 8 GHz [10]	±1.5%	$4.5x10^{-3}$	$5x10^{-3}$ at 8 GHz [10]	±4.8%
BMT	25.3	24.7 at 16 GHz*	±1.5%	———	$9.8x10^{-5}$ at 16 GHz*	———
Zircon	32.7	33.5 at 7 GHz*	±1.5%	———	$9x10^{-4}$ at 5 GHz*	———
Epoxy HT	3.72	3.61 at 16GHz*	±1.3%	$4.9x10^{-3}$	$5.39x10^{-3}$ at 16 GHz*	±5.1%
Arlon 45N	4.15	4.3 at 2.4 GHz*	±1.4%	$1.1x10^{-2}$	$1.2x10^{-2}$ at 2.4 GHz*	±4.9%

Table 1: Determination and comparison of complex permittivity. (*characterized with SCR method).

We can also plot dielectric cartographies that are rapid, non-destructive, on contact and non-contact mode, for samples having no dielectric or surface homogeneity with a spatial resolution of 100 µm.

6. CHARACTERIZATION OF PRINTED DIELECTRIC LAYERS

Inkjet technology is based on the superposition of layers of diverse materials in order to build a 2D or 3D component, using multi nozzle piezoelectric print heads delivering precise volume of ink droplets (few pL) on a substrate. In order to study the effect of the thickness of printed layer by an inkjet technology on the characterization, an electromagnetic model was designed by electromagnetic analysis software, based on finite elements method, developed at the XLIM laboratory by Dr. Michel AUBOURG. The Figure 7 shows the 3D meshed version of our model, ie the probe in contact with the dielectric sample.

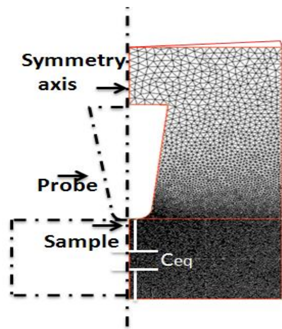

Figure 7: 3D meshed Electromagnetic model of the probe and dielectric sample

A numerical simulation allows us to extract the value of equivalent capacity C_{eq} which models the dielectric sample. Two parameters are studied: the thickness of the sample, and its permittivity, the tip diameter is fixed at 70µm. Figure 8 shows the variation of C_{eq} when we increase the thickness of the sample. For a permittivity of 64.2 (corresponds to the permittivity at 4 GHz of a bulk resonator made of LTCC 51K65 material made by Heraeus) the value of C_{eq} decreases with the increase of the sample thickness. But starting from 0.3 mm, the increase of the thickness does not have any influence on the value of C_{eq}. Therefore, if a sample of height 0.3 mm or more is printed on a substrate, this latter does not have any effect on the results of characterization, and equations 1 and 2 can be directly applied. However, if the thickness of the sample is less than 0.3 mm, the substrate modifies the characterization results, hence, new equations must be established, taking into account the height of the printed sample.

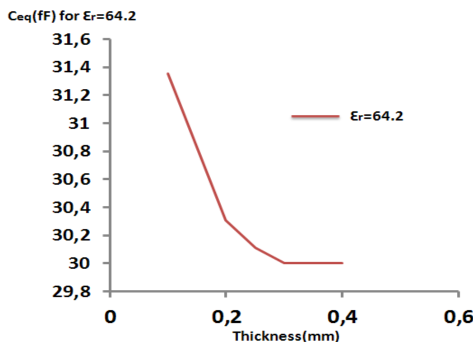

Figure 8: Variation of C_{eq} as a function of the thickness and the permittivity

A layer of LTCC 51K65 material made by Heraeus (thickness 300 µm, surface=2.25 mm^2) has been printed over an Alumina substrate by inkjet technology as shown in Figure 9. This layer is a typical example of dielectric material having a size that we cannot use traditional characterization techniques with, and thus requiring such probe type characterization. We have measured the variation of f_r and Q_0 provided when our probe is in contact with the sample. We therefore find complex permittivity of this layer equal to ε_r= 67.5 and tan δ = 1.28 10^{-3} at 2.5 GHz. The obtained results are in good agreement with those obtained with standard method with a dielectric resonator which gives the following values: ε_r= 64.2 and loss tangent 1.8 10^{-3} at 4 GHz. A very good accuracy of 5% on the relative permittivity of this material is obtained with the probe method.

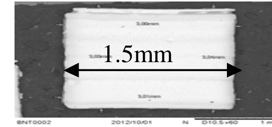

Figure 9: LTCC 51K65 layer

7. CONCLUSION

In conclusion, we have proposed a specific technique and optimization method in order to increase the sensitivity of near-field microscopy technique. This nondestructive method allows the characterization (ε_r and tan δ at 2.45 GHz) of small samples having a non-arbitrary form, such as dielectric layers printed on substrate by inkjet technology. A forthcoming work will be done to extend the procedure of characterization to a frequency band and to improve our modeling approach to extract ε_r and tan δ for layers having a lower thickness (less than 0.3 mm).

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