Reflection ellipsometry for in-situ measurements of complex permittivity and thickness of a single-layer material at microwave frequencies : theory and experiments

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For in-situ measurement of the complex permittivity of planar materials, we have developed a free-space experimental setup based on reflection ellipsometry and extended to microwave frequencies. Different angles of incidence were studied in the range [35 ; 50°]. Original numerical methods, derived from analytical relations based on contour line charts and least-square optimization, allow to highlight the influence of measurement uncertainties. Novel developments concerning the simultaneous determination of both complex permittivity and thickness of a single-layer sample are shown.

INTRODUCTION

Reflection ellipsometry, an optical technique recently adapted to microwave frequencies, has been proved to be an efficient approach for nondestructive in-situ characterization of dielectric materials [1,2]. The reflection configuration appears relevant for non destructive evaluation of materials such as construction materials, which cannot be moved to suit the positioning of the experimental system.

Reflection ellipsometry allows to experimentally determine, from analytical relations and scalar measurements, the complex ratio $\tilde{r} = \tilde{r}_p / \tilde{r}_s$ of the reflection coefficients of a sample. This technique offers the two following advantages : it requires a low-cost detection system based on a powermeter, an alternative choice to a vector network analyzer (VNA) ; moreover the angle of incidence is fixed during the measurement process and thus can be chosen optimally so as to minimize the direct coupling between the two antennas.

METHODS FOR DETERMINING THE COMPLEX PERMITTIVITY ONLY, WHEN THE THICKNESS IS KNOWN

The principle of the method is as follows : once the angle of incidence is fixed, the reflected power P_d of the electric field is recorded as a function $P_d = f(A)$ of the polarization angle A of the receiving antenna (see figures 1 and 3). Fixing the polarization of the transmitting antenna to an angle $P = \pm p/4$, the power detected by the powermeter versus the analysis angle A has a raised-sine shape which writes as follows [1] :

$$P_d = P_0[1 - \cos(2A)\cos(2y_r) \pm \sin(2A)\sin(2y_r)\cos(\Delta_r)] \quad (1)$$

where P_0 is a normalizing parameter that depends on the power of the incident wave and on the characteristics of the detector.

To determine the ratio $\tilde{r} = \tilde{r}_p / \tilde{r}_s$ from the experimental curve $P_d = f(A)$ and then to deduce the complex permittivity $\tilde{e}$ using Fresnel formulas, we have developed a multisteps statistical data analysis method associated with each of the two different approaches so-called, the "min-max" and the "three points" methods. These approaches use different parameters extracted from the experimental curve to determine afterwards y_r and Δ_r, which are linked to the modulus and the phase of the ratio $\tilde{r}$ as follows : $\tilde{r} = \tan(y_r)e^{j\Delta_r}$. These parameters, y_r and Δ_r, allow in turn to estimate both real and imaginary parts of the complex permittivity using contour line charts. These charts allow to plot a parallelogram of uncertainties centered on the experimental values, thus allowing to determine the influence of measurement uncertainties.

Studies made by the authors lead to the achievement of an automated experimental setup [2]. The interaction in the reflection configuration between a plane wave at microwave frequencies (X-band and Ku-band) and usual materials (fibreboad, PVC, plasterboard) with different thicknesses leads to estimations of complex permittivities which agree with those obtained in the literature.

METHOD FOR DETERMINING THE COMPLEX PERMITTIVITY AND THE THICKNESS

For the case where the thickness of the material is not available, we have developed a novel approach to determine jointly the complex permittivity and the thickness of a single-layer sample when considering ellipsometry measurements made at angles of incidence other than the normal one [3,4]. This approach differs from previous developments by the association of three peculiar features : 1) no initial guess value of the complex permittivity is required, 2) the angles of incidence differ from the normal incidence, 3) the numerical approach is innovative and it involves automatically the measurement uncertainties. The principle consists in finding an equation which does not involve the thickness to first determine the relative complex permittivity

$\tilde{e} = e' - je''$; in a second step, the thickness ΔZ of the sample is evaluated. To solve the problem, more than two measurements of the complex ratio $(\tilde{r}_1, \tilde{r}_2)$ at the associated angles of incidence (Θ_1, Θ_2) are required.

The equation to be first solved, obtained by eliminating the thickness, is the following :

$$C(\Theta_1, \Theta_2, \tilde{r}_1, \tilde{r}_2, \tilde{e}) = \frac{\ln|\tilde{A}_1|}{\mathrm{Im}(\tilde{q}_1)} - \frac{\ln|\tilde{A}_2|}{\mathrm{Im}(\tilde{q}_2)} = 0 \qquad (2)$$

where :

$$\tilde{A}_{(1,2)} = \frac{\dfrac{1}{\tilde{s}_{(1,2)}} - \dfrac{\tilde{R}_{(1,2)}}{\tilde{s}_{(1,2)}}}{\tilde{s}_{(1,2)} - \tilde{R}_{(1,2)}\,\tilde{p}_{(1,2)}}$$

$$\begin{cases} \tilde{s}_{(1,2)} = \dfrac{q_0 - \tilde{q}_{(1,2)}}{q_0 + \tilde{q}_{(1,2)}} ; \; p_{(1,2)} = \dfrac{\dfrac{\tilde{q}_{(1,2)}}{\tilde{e}} - q_0}{\dfrac{\tilde{q}_{(1,2)}}{\tilde{e}} + q_0} \\ q_0 = \dfrac{2p}{l_0}\cos\Theta_{(1,2)} ; \; \tilde{q}_{(1,2)} = \dfrac{2p}{l_0}\sqrt{\tilde{e} - \sin^2\Theta_{(1,2)}} \end{cases} \qquad (3)$$

l_0 is the wavelength in air.

We propose to numerically solve this equation in $\tilde{e}$ by minimizing the cost function $\ln|C(\Theta_1, \Theta_2, \tilde{r}_1, \tilde{r}_2, e)|$ in the sample space (e', e'') (figure 2a). The optimum value retained for e' and e'' corresponds theoretically to a minimum of the cost function. In fact, supposing that the angles (Θ_1, Θ_2) are measured with enough precision (which is a valuable approximation), the determination of $(\tilde{r}_1, \tilde{r}_2)$ is influenced only by ellipsometric measurement uncertainties. Statistically, better results are obtained if several couples from M angles of incidence ($M > 2$) are considered (figure 2b) ; this is why we consider a global cost function C_g of the following form:

$$C_g(\tilde{e}) = \sum_{i=1}^{M-1} \sum_{j=i+1}^{M} C(\Theta_i, \Theta_j, \tilde{r}_i, \tilde{r}_j, \tilde{e}) \qquad (4)$$

Then, the thickness ΔZ can be deduced by the following relations :

$$\frac{\ln|\tilde{A}_1|}{\mathrm{Im}(\tilde{q}_1)} = 2\Delta Z \quad or \quad \frac{\ln|\tilde{A}_2|}{\mathrm{Im}(\tilde{q}_2)} = 2\Delta Z \qquad (5)$$

A mean value of different estimations of ΔZ is adopted. Experimental results yield satisfactory values of the thickness, with an estimated relative uncertainty lower than 10%.

EXPERIMENTAL SETUP

The experimental measurement system, named COTREMO, depicted on figure 1, is built on two semi-circular horizontal plane supports made up of plexiglass and placed in the front and the back side of the material under test. This setup is based on a bistatic configuration, which consists of transmitting and receiving antennas. The reflected power is detected by a powermeter. The sample is hanging from the ceiling by four fixed pulleys in the middle of the room, far from any surrounding objects. The distance R between the antenna and the spot of reflection on the sample surface is $R = 0.6\ m$. Each antenna is set on a column fixed on an arm, allowing the adjustment of the radius R of the support which marks out the angle of incidence. The antennas are mounted on their own column by a rectangular mechanical adapter which allows to consider different waveguides (X and Ku-bands at present) ; this adapter fills the rotating system to allow the rotation of the antenna around its horizontal axis. A pair of pyramidal horns with a particularly low phase deviation at their aperture have been chosen To cancel scattering phenomena from the sample, its size (1m× 2 m) has been chosen greater than the first Fresnel zone at the grazing incidence.

EXPERIMENTAL RESULTS

Several samples like fibreboard ($e = 10\ mm$ and $16\ mm$), plasterboard ($e = 12\ mm$) and PVC slabs ($e = 10.4\ mm$ and $15.5\ mm$) have been measured in reflection at different frequencies included in the X- and Ku-bands. Materials are supposed to be homogenizable and their relative complex permittivity $\tilde{e}_r$ is estimated at different angles of incidence Θ_i and frequencies F in the range [35°;50°] and [8.5 GHz;17 GHz], respectively. The reflected powers have been normalized to the power measured when the two antennas face each other at a distance equal to the value of the ray path ($2R$). The maximal uncertainties associated with the measured reflected power P_d and the analyzer angle A are respectively estimated to the following average values $\pm 0.2dB$ (relative) and $\pm 0.3°$ (absolute).

Considering the PVC slab with thickness $15.5mm$, the mean value of the complex permittivity at frequency 12 GHz which have been estimated using the "min-max" approach, at the angles of incidence {35°,40°,45°,50°}, is $\tilde{e}_r = 2.19 - 1.13j$; the corresponding experimental ellipsometric curves are plotted on figure 3. Curves of figure 3(a) show that the angular position of the maximum and the corresponding amplitude increase with the angle of incidence (except for 35°), as predicted by theory and shown on figure 4. Fixing the angle of incidence at 45°, experimental ellipsometric curves have been plotted as a function of frequency [8.5;12;14;17] GHz, as shown on figure 3(b) ; it can be observed that the maximum amplitude decreases significantly with frequency, but that the corresponding angular position is almost constant. It must be underlined that different complex permittivity values are associated to each curve, because of the measurement uncertainties (see table 1). Considering the mean value of the complex permittivity ($\tilde{e}_r = 2.19 - 1.13j$), the theoretical ellipsometric curves are plotted on figure 5 for each frequency in the set {8.5;12;14;17} GHz.

The mean values of the complex permittivity of the thicker materials studied have been collected in table 2. The uncertainty on the real part of the permittivity is estimated to 15%. Concerning the imaginary part, we have observed that when its value is lower than 0.5, the estimation is not valuable ; this difficulty characterizes free-space methods.

CONCLUSION

In-situ characterization of dielectric materials at microwave frequencies using ellipsometry appears as a new low cost experimental approach. The reflection configuration is well adapted to nondestructive evaluation. The main objective is to determine complex permittivities of different slabs at different angles of incidence. When the thickness is available, the estimates of the complex permittivity for the two types of materials studied with different thicknesses appear close as a function of the angle of incidence. When the thickness is not available, a novel method has been developed by the authors, which allows to simultaneously determine the real and imaginary parts of the complex permittivity and the thickness of the sample. Such a development will help the full characterization of a single-layer dielectric sample. Analysis of experimental data for this dual determination are in progress.
Future trends focus on in-situ dielectric characterization of materials at other frequency bands, and the measurement and modeling by numerical methods of heterogeneous materials. Results on the modeling of wave-matter interaction at an oblique angle of incidence have already been obtained.

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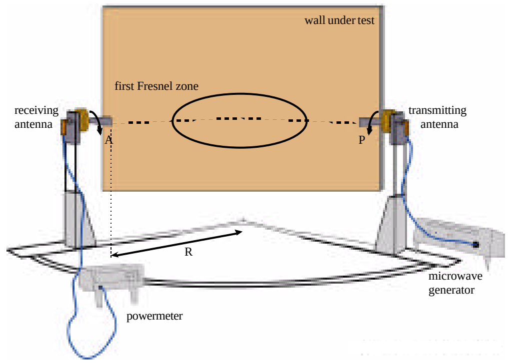

Figure 1 : Setup for the measurement of reflected waves

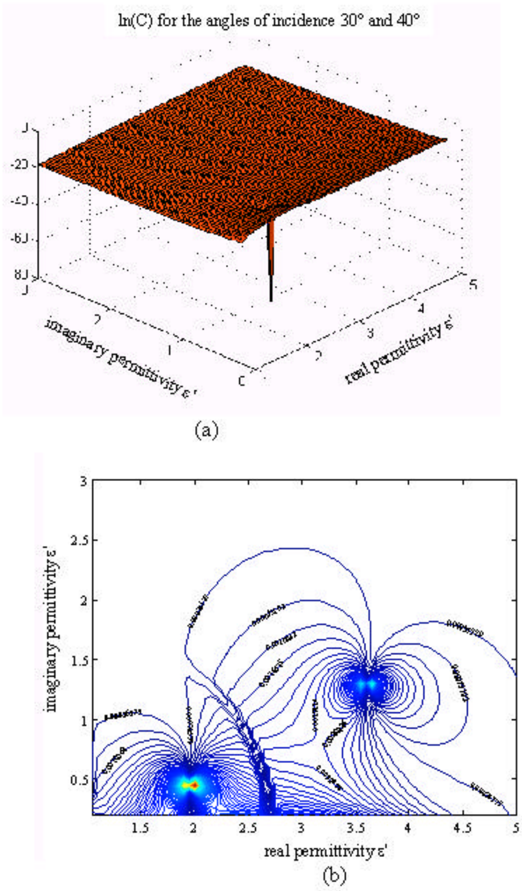

Figure 2 : Charts of cost functions for the theoretical estimation of ε=2-0.5j without (a) and with measurement uncertainties (b)

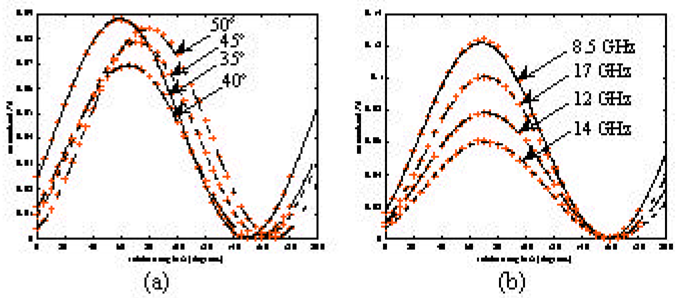

Figure 3 : Experimental ellipsometric curves in the case of a PVC slab (15.5 mm) at different angles of incidence [35°;50°] (F=12 GHz) (a) and different frequencies ($q_i = 45°$) (b)

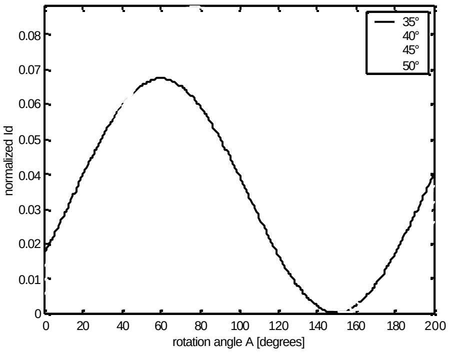

Figure 4 : Theoretical ellipsometric curves in the case of a PVC slab (15.5 mm) with a mean value the complex permittivity equal to 2.19-1.13j at the frequency 12 GHz and different angles of incidence [35°;50°]

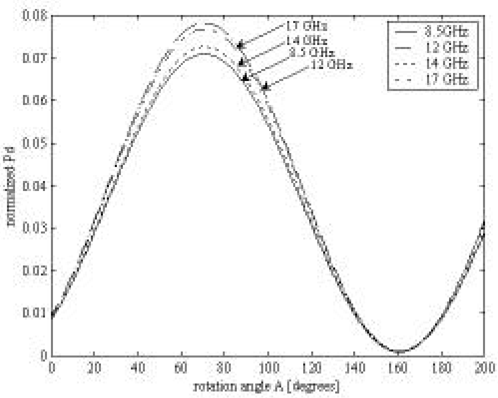

Figure 5 : Theoretical ellipsometric curves in the case of a PVC slab (15.5 mm) with a mean value the complex permittivity equal to 2.19-1.13j at the angle of incidence 45° and different frequencies [8.5;17] GHz

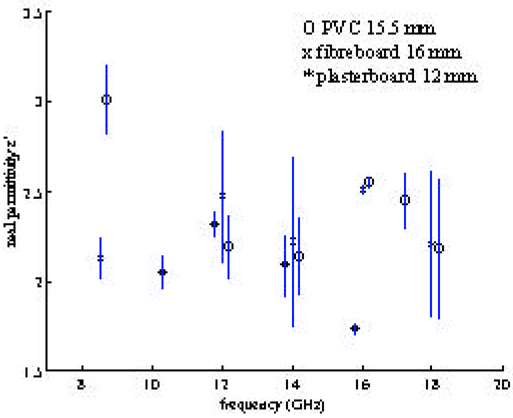

Figure 6 : Estimations of the real part of the complex permittivity and their uncertainties of common materials versus frequency

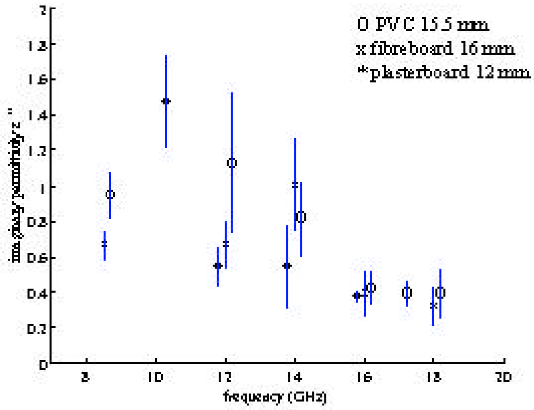

Figure 7 : Estimations of the imaginary part of the complex permittivity and their uncertainties for common materials versus frequency

	Angles of incidence q_i ($f = 12\ GHz$ fixed)			
samples	$q_i = 35°$	$q_i = 40°$	$q_i = 45°$	$q_i = 50°$
PVC 15.5mm	1.98-1.63j	2.22-1.05j	2.14-1.16j	2.41-0.67j
fibreboard 16mm	2.02-0.84j	2.32-0.69j	2.74-0.52j	2.8-0.63j
plasterboard 12mm	2.20-0.71j	2.37-0.56j	2.35-0.48j	2.34-0.46j

Table 1 : Complex permittivities values obtained at the frequency 12 GHz and at angles of incidence in the range [35°;50°] of common samples

	Frequency F ($q_i = 45°$ fixed)			
samples	8.5 GHz	12 GHz	14 GHz	17 GHz
PVC 15.5mm	3.01-0.95j	2.19-1.13j	2.14-0.82j	2.45-0.4j
fibreboard 16mm	2.13-0.65j	2.47-0.67j	2.22-1.01j	2.21-0.4j
plasterboard 12mm		2.32-0.55j	2.01-0.55j	1.76-0.38j

Table 2 : Mean values of complex permittivities of common samples