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Characterization of the refractive index of isotropic materials by three-detector microwave ellipsometry

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ABSTRACT: A three-detector microwave ellipsometer is an experimental free-space bench for characterization of non-transparent materials. It is a non-destructive characterization technic working in oblique transmission in the frequencies range of 26 to 30 GHz. A vector network analyzer (VNA) is used as microwave source. The method is based on the determination of complex diagonal tensor which requires the measurement of the sample transmission coefficients. Calibration of the network vector analyzer is needed in order to correct the values of this coefficients due to the measurement errors. The aim of this paper is to show that One Path Two Ports calibration method is convenient for this technic.

KEYWORDS: Microwave, ellipsometry, free space method, isotropic materials, calibration.

1 INTRODUCTION

Natural isotropy is present in many materials at different levels and is directly related to their dielectric properties. As non-destructive methods, microwave techniques are efficient to characterize it. These techniques are based on measurement of transmitted or reflected wave send through the sample in free space [1], [2]. For better accuracy of the measurement, a vector network analyzer (VNA) [3] is used in some cases to automate the process.

In this paper, we use a polarimetric transmission measurement techniques with three fixed detectors to characterize isotropy of materials in free space as described in some previous works [4], [5]. So called microwave ellipsometry method, the technique requires the determination of the angle between the elliptical transmitted polarization and the linear incident waves. This is quite possible by the knowledge of the reflection and transmission coefficients (S_{11}, S_{21}) of the sample under testing. The incident wave is provided by a VNA through coaxial cables that have to be calibrated. We adopt the One Path Two Port Calibration method for the errors determination in order to correct these reflections and transmissions coefficients. The measured and then, adjusted transmission coefficient S_{21}, leads to the three detectors given intensities. These intensities allow the calculation of the rotation (angle between elliptical polarization axes). The theoretical calculation model is based on the Jones' matrix formalism representing each element in the measurement system. In this model, we determine the three intensities with respect to the angle. First, we calculate the transmission coefficients of the considered sample which are related to its dielectric properties in given experimental conditions (frequency, incidence angle and angular position of the sample). Then, the best matching between measurement and simulation of these coefficients is met by inverse problem resolution based on classical Levenberg–Marquardt optimization on complex refractive indices [2].

In this paper, we deal with the calibration procedure to be performed for measuring the transmission coefficient in the case of the three-detector microwave ellipsometer. After all, we will present some results yielded from it.

2 Principle Of The Measurement

An isotropic media can be described by its thickness d and its three complex optic indexes N_x, N_y and N_z. Generally, when the material is biaxial, these indexes form an ellipsoid.

$$N_i = n_i - jk_i \text{, with } i = \{x, y, z\} \quad (1)$$

Each index is related to a complex permittivity given by:

$$\varepsilon_i = \varepsilon_i^{'} - j\varepsilon_i^{''} \text{, with } i = \{x, y, z\} \quad (2)$$

Generally, the associated tensor ε is symmetric and is given by the following matrix:

$$\varepsilon = \begin{bmatrix} \varepsilon_{xx} & 0 & 0 \\ 0 & \varepsilon_{yy} & 0 \\ 0 & 0 & \varepsilon_{zz} \end{bmatrix} \quad (3)$$

Similarly to the case of isotropy, $n_i = \sqrt{\varepsilon_i}$, the elements of this tensor enter into the calculation of the following matrix [6], [7] Δ of the equation (4).

$$\Delta = \begin{bmatrix} -k_{xx}\dfrac{\varepsilon_{zx}}{\varepsilon_{zz}} & -k_{xx}\dfrac{\varepsilon_{zy}}{\varepsilon_{zz}} & 0 & 1-\dfrac{k_{xx}^2}{\varepsilon_{zz}} \\ 0 & 0 & -1 & 0 \\ \varepsilon_{yx} - \varepsilon_{yz}\dfrac{\varepsilon_{zx}}{\varepsilon_{zz}} & k_{xx}^2 - \varepsilon_{yy} + \varepsilon_{yz}\dfrac{\varepsilon_{zy}}{\varepsilon_{zz}} & 0 & k_{xx}\dfrac{\varepsilon_{yz}}{\varepsilon_{zz}} \\ \varepsilon_{xx} - \varepsilon_{xz}\dfrac{\varepsilon_{zx}}{\varepsilon_{zz}} & \varepsilon_{xy} - \varepsilon_{xz}\dfrac{\varepsilon_{zy}}{\varepsilon_{zz}} & 0 & k_{xx}\dfrac{\varepsilon_{xz}}{\varepsilon_{zz}} \end{bmatrix} \quad (4)$$

k_{xx} is related to the component of the propagation vector k_x, and ε_{ij} are the elements of the tensor.

The component of the propagation vector through x-axis is given by:

$$k_x = \frac{\omega}{c} n_0 \sin\varphi \quad (5)$$

where n_0 is the ambient index and φ the angle of incidence angle.

So the expression of k_{xx} is given by:

$$k_{xx} = \frac{c}{\omega} k_x = n_0 \sin\varphi \quad (6)$$

The matrix Δ of equation (4) makes it possible to determine the partial transfer matrix T_p established by Schubert from the Berremen equation [6], [7]:

$$T_P = \exp\left(i\frac{\omega}{c}\Delta(-d)\right) \quad (7)$$

where ω is the angular frequency, c the light speed, and d the thickness of the material.

Schubert defined two matrixes: an input matrix L_i (incident matrix) and an output matrix L_t (matrix of transmission).

The input matrix L_i is obtained from the tangential components of the electric field and the magnetic field through the ambient-material interface at $z=0$. Then, its inverse matrix L_i^{-1} is given by the following expression:

$$L_i^{-1} = \frac{1}{2}\begin{bmatrix} 0 & 1 & -1/n_0\cos\varphi & 0 \\ 0 & 1 & 1/n_0\cos\varphi & 0 \\ 1/\cos\varphi & 0 & 0 & 1/n_0 \\ -1/\cos\varphi & 0 & 0 & 1/n_0 \end{bmatrix} \quad (8)$$

The output matrix L_t, is expressed according to the variation of the electric fields and the magnetic fields transverse to the material-ambient interface at $z=d$ and is given by the following expression :

$$L_t = \begin{bmatrix} 0 & 0 & \cos\varphi_t & 0 \\ 1 & 0 & 0 & 0 \\ -n_2\cos\varphi_t & 0 & 0 & 0 \\ 0 & 0 & n_2 & 0 \end{bmatrix} \quad (9)$$

In our case $\varphi_t = \varphi$ and $n_2 = n_0$.

Given the transfer matrix T_p, the inverse input matrix L_i^{-1} and the output matrix L_t, a new matrix called the transfer matrix T is defined and given by the following expression [8], [12], [9], [6] :

$$T = L_i^{-1}T_pL_t \quad (10)$$

This transfer matrix T is a 4 × 4 matrix and expressed as follow:

$$T = \begin{bmatrix} T_{11} & T_{12} & T_{13} & T_{14} \\ T_{21} & T_{22} & T_{23} & T_{24} \\ T_{31} & T_{32} & T_{33} & T_{34} \\ T_{41} & T_{42} & T_{43} & T_{44} \end{bmatrix} \quad (11)$$

The elements of this matrix are used to determine the transmission coefficients which constitute the Jones matrix of the equation (21). These coefficients are described by the following equations [8]:

$$t_{pp} = \frac{T_{11}}{T_{11}T_{33} - T_{13}T_{31}} \quad (12)$$

$$t_{sp} = \frac{-T_{13}}{T_{11}T_{33} - T_{13}T_{31}} \quad (13)$$

$$t_{ss} = \frac{T_{33}}{T_{11}T_{33} - T_{13}T_{31}} \quad (14)$$

$$t_{ps} = \frac{-T_{31}}{T_{11}T_{33} - T_{13}T_{31}} \quad (15)$$

The three detectors ellipsometer can be configured for measurement in transmission and either oblique or normal incidence. In our case, we process in oblique incidence φ ($\varphi = 36^0$). During the measurement process, the sample rotates around the direction of propagation with fixed step (2^0) in order to obtain a set of measurements. Angular position θ is measured from the direction of the incident wave [5] Fig. 1.

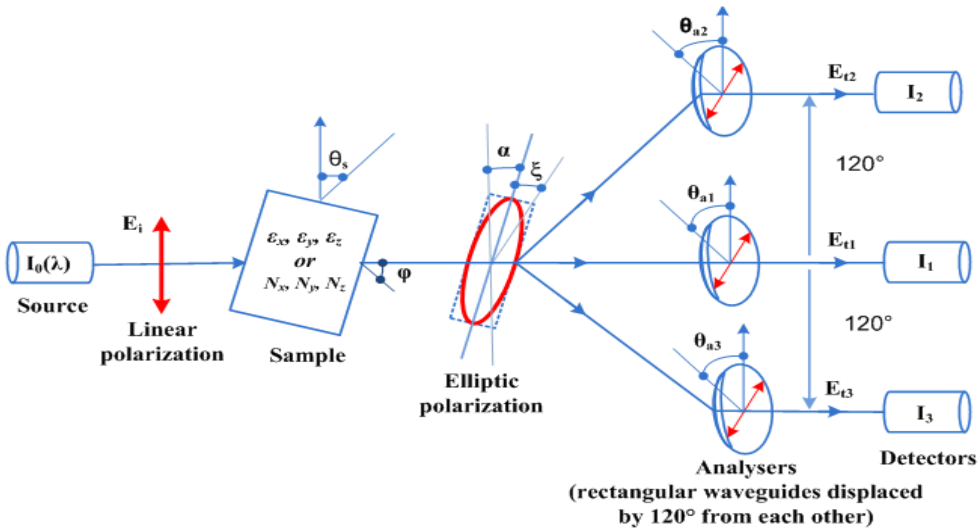

Fig. 1.   Principle of the measurement method

Transmitted and incident waves fields are theoretically bound by the formalism of Jones matrix [8], [10] given by:

$$E_t = R(\theta_s) M_s R(-\theta_s) M_a R(\theta_a) E_i \quad (16)$$

where $R(\theta_s)$, $R(-\theta_s)$ are the Jones's matrixes of the rotation of the sample and M_s is Jones matrix of the sample. M_a is the Jones matrix of the analyzer and $R(\theta_a)$ is the Jones matrix of its rotation. During measurement, only the sample rotates whereas the analyzer is motionless ($\theta_a = 0$).

The linear incident polarization is along the x-axis. Three branches represent the three analyzers which are rectangular waveguides placed at 120° from each other. As for the three directions, the transmitted electric fields are given by the following equations:

$$E_{t_1} = R(\theta_s) M_s R(-\theta_s) M_a R(\theta_a) E_i \quad (17)$$

$$E_{t_2} = R(\theta_s) M_s R(-\theta_s) M_a R(\theta_a + 120^0) E_i \quad (18)$$

$$E_{t_3} = R(\theta_s) M_s R(-\theta_s) M_a R(\theta_a - 120^0) E_i \quad (19)$$

The corresponding theoretical electromagnetic intensities are given by the dot product:

$$I_i = E_{t_i}^{\ *} E_{t_i} \quad (20)$$

where $i = 1,2,3$ referring to branch directions.

Theses intensities depend on the angular rotation of the sample $I_i(\theta_s)$.

For the isotropic case, M_s Jones matrix is

$$M_s = \begin{bmatrix} t_{pp} & 0 \\ 0 & t_{ss} \end{bmatrix} \quad (21)$$

Where t_{pp} , t_{ss} are transmission coefficients depending on parallel and perpendicular axes [11], [12], [13].

They are function of sample dielectric properties, thickness and incident wave frequency.

The rotation α measured by our ellipsometer [5], i.e. the angle between the large radius of the transmitted elliptical wave and the direction of the linear emitted wave Fig. 4, is calculated as a function of three theoretical intensities :

$$\alpha = \frac{1}{2}\arctan\left[\frac{\sqrt{3}\left(I_3 - I_2\right)}{2I_1 - I_2 - I_3}\right] \quad (22)$$

The three calculated intensities depend on the starting value of the diagonal tensor.

To compare the theoretical value of the rotation α given in equation (22) with its measured value, we need to measure transmission coefficients through the three detectors. To do so, we use a 2-port calibration procedure to correct them.

3 VNA Calibration

Before measuring S_{11} and S_{21} parameters of the device under test (DUT), VNA must be calibrated to calculate system errors. Depending on the desired correction, different 2 ports calibration types can be performed. The 2-portcalibration method determines the best uncertainty [14], [15]. The most common used methods consist in calculating 12 error terms, 6 forward and 6 reverse error terms [16] as depicted in Fig. 2.

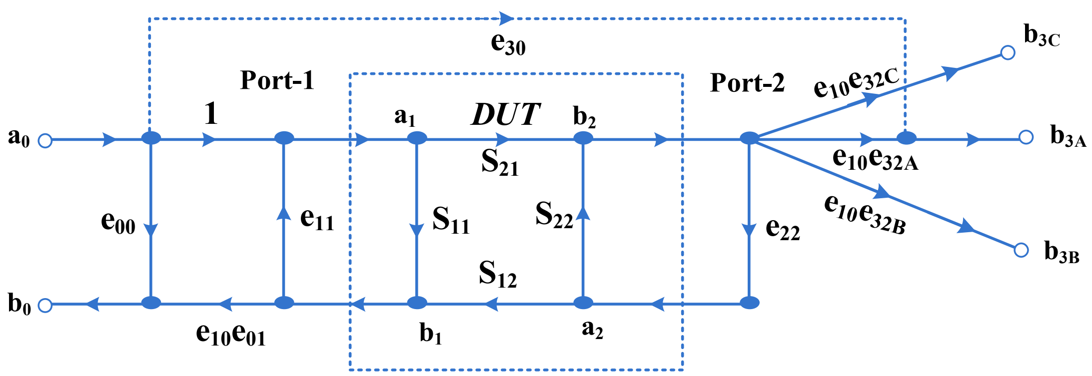

Fig. 2. Graph of influence model 12 terms

e_{00} : Directivity

e_{11} : Port-1 (Source) Match

$e_{10}e_{01}$: Reflection Tracking

$e_{10}e_{32}$: Transmission Tracking

e_{30} : Leakage (crosstalk)

e_{22} : Port-2 (Load) Match

The One Path Two ports calibration method we use consists of calculating 3 error terms (3 related to port 1 and 2 associated to Port 2). In our case, we do not take into account isolation error.

3.1 Port 1 calibration

TSO (Thru, Short and Offset-Short) tests are required. Then, error terms of port 1 are calculated from Fig. 2:

$$e_{00} = S_{11m}(Thru) \quad (23)$$

$$e_{11} = \frac{\left(\left(S_{11m}(OffsetShort) - e_{00}\right)^{e^{-2\Gamma(OffsetShort)}} - S_{11m}(Short) - e_{00}\right)}{S_{11m}(Short) - S_{11m}(OffsetShort)} \quad (24)$$

$$e_{10}e_{01} = -(S_{11m}(Short) - e_{00})(1 - e_{11}) \quad (25)$$

Where $\Gamma(OffsetShort) = j\omega c * d$ and d is the distance of the Offset Short

3.2 Port 2 Calibration

Thru calibration calculates the port 2 error terms. In this procedure $e_{22} = 0$.

$$e_{10}e_{32(A,B,C)} = S_{21m}(Thru_{(A,B,C)}) \quad (26)$$

3.3 S_{11} and S_{21} Correction

To calculate the Device Under Test (DUT) corrected S_{11}, it is necessary to measure S_{11m} and apply the appropriated mathematical correction [17] using equations (23), (24), (25), which give

$$S_{11(A,B,C)} = \frac{S_{11m(A,B,C)}(DUT) - e_{00}}{e_{10}e_{01} + e_{11}\left(S_{11m(A,B,C)}(DUT) - e_{00}\right)} \quad (27)$$

To calculate DUT adjusted S_{21} parameter, it is also necessary to measure the S_{21m} parameter and apply proper mathematical correction16 using equations (24), (26), (27) which give

$$S_{21(A,B,C)} = \frac{S_{21m(A,B,C)}(DUT)\left(1 - e_{11}S_{11(A,B,C)}\right)}{e_{10}e_{32(A,B,C)}} \quad (28)$$

Measurements are done in transmission with three detectors 120° shifted each from other and in oblique incidence as shown in Fig. 3. Three transmission coefficients corresponding to the three intensities are determined.

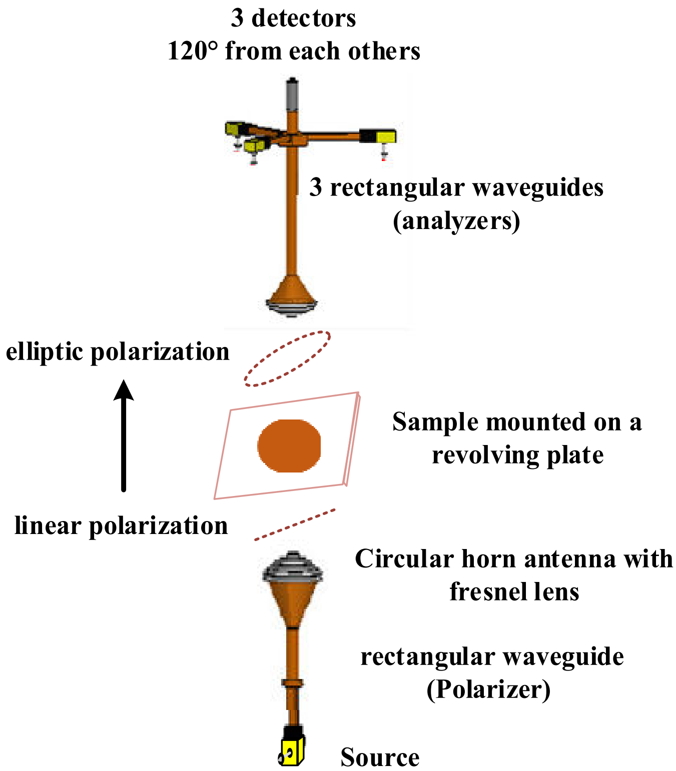

Fig. 3. Configuration of the experimental measurement bench

The three intensities measured are deduced from corrected S_{21} (eq. 28).

$I_1 = S_{21A}$ for detector 1 (29)

$I_2 = S_{21B}$ for detector 2 (30)

$I_3 = S_{21C}$ for detector 3 (31)

One detector is placed in the direction of the incident polarization. The maximum energy is recovered by this one. The two other detectors recover quite the same energy is which equal to the quarter of that of the first one.

The Intensities are measured for different angular positions of the sample with a frequency sweep. It is possible to calculate the rotation α of the equation (22) Fig. 4.

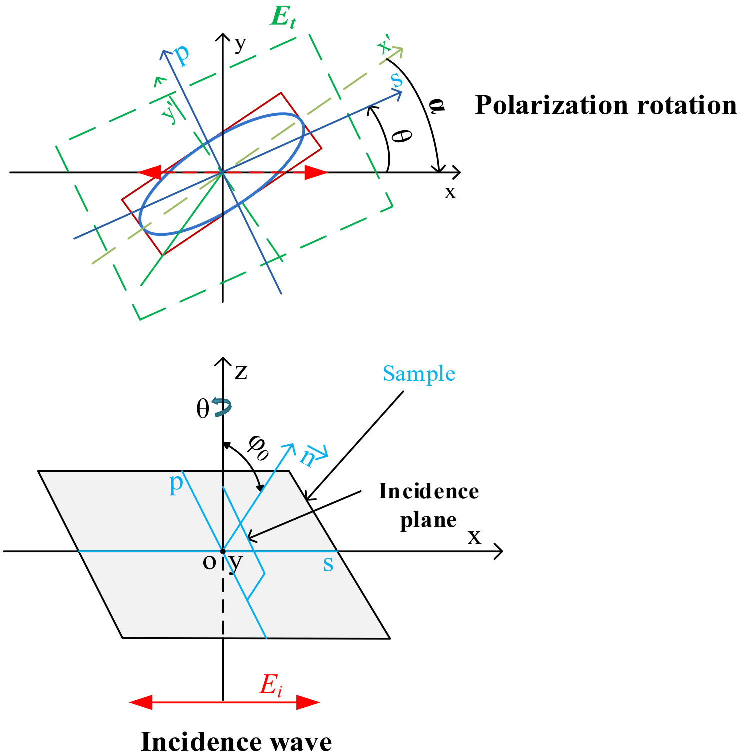

Fig. 4. ***Polarization state change of the polarized wave propagating in isotropic media***

4 RESULTS

In Fig. 5, we present one example of characterization of a polytetrafluoroethylene (PTFE) sample.

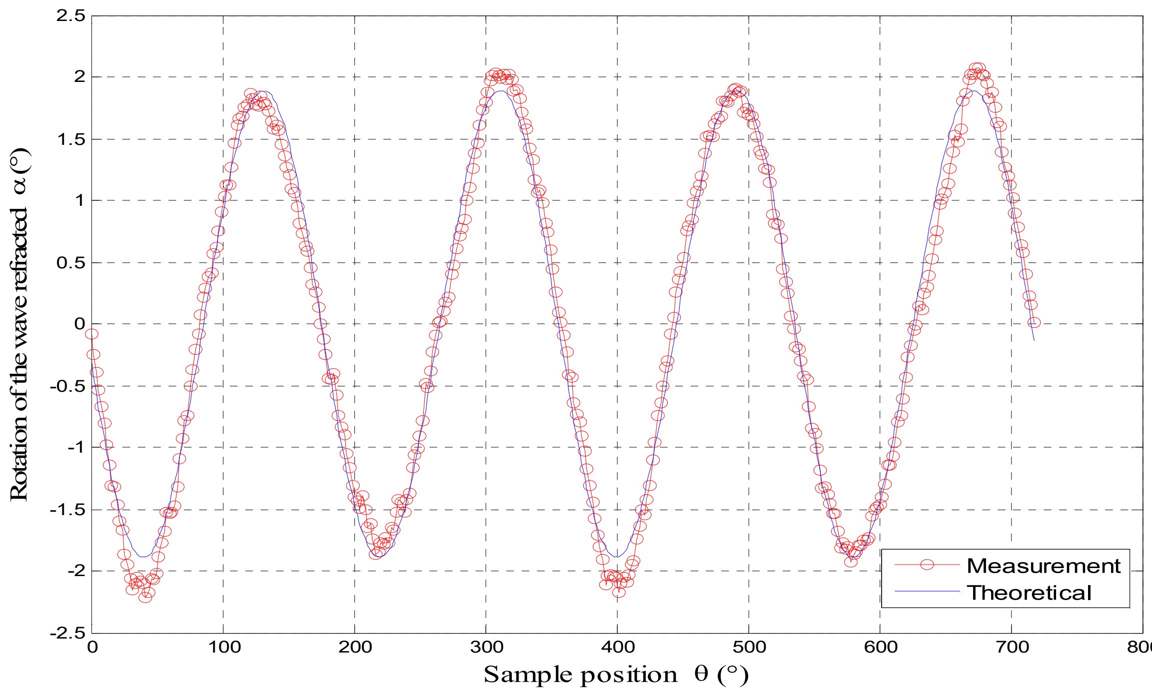

Fig. 5. ***Characterization curve of a PTFE sample having 2 mm of thickness at f = 26 GHz.***

The results of the 2 mm thick refractive indices and absorption indices obtained from the tensor results of complex permittivities after resolution of the inverse problem for a frequency of 26 GHz of the incidence wave are summarized in Table 1.

Table 1. Characterization results

Thickness 2 mm , f = 26 GHz					
$\varepsilon_x^{'}$	$\varepsilon_x^{''}$	$\varepsilon_y^{'}$	$\varepsilon_y^{''}$	$\varepsilon_z^{'}$	$\varepsilon_z^{''}$
2.0921	0.0366	2.1017	0.4091	2.0298	0.0544
n_x	k_x	n_y	k_y	n_z	k_z
1.4465	0.0126	1.4565	0.1404	1.4249	0.0191

he following example shows the correlation between the model of interaction and the measurement. The PTFE refraction indices obtained when solving the inverse problem are almost equal to their values given in the several references [18], [19], [20], [5], where $N=\sqrt{\varepsilon}$ and ε is the permittivity of the sample. As the PTFE is known as non-absorbent, it is quite normal that the absorption coefficients be low.

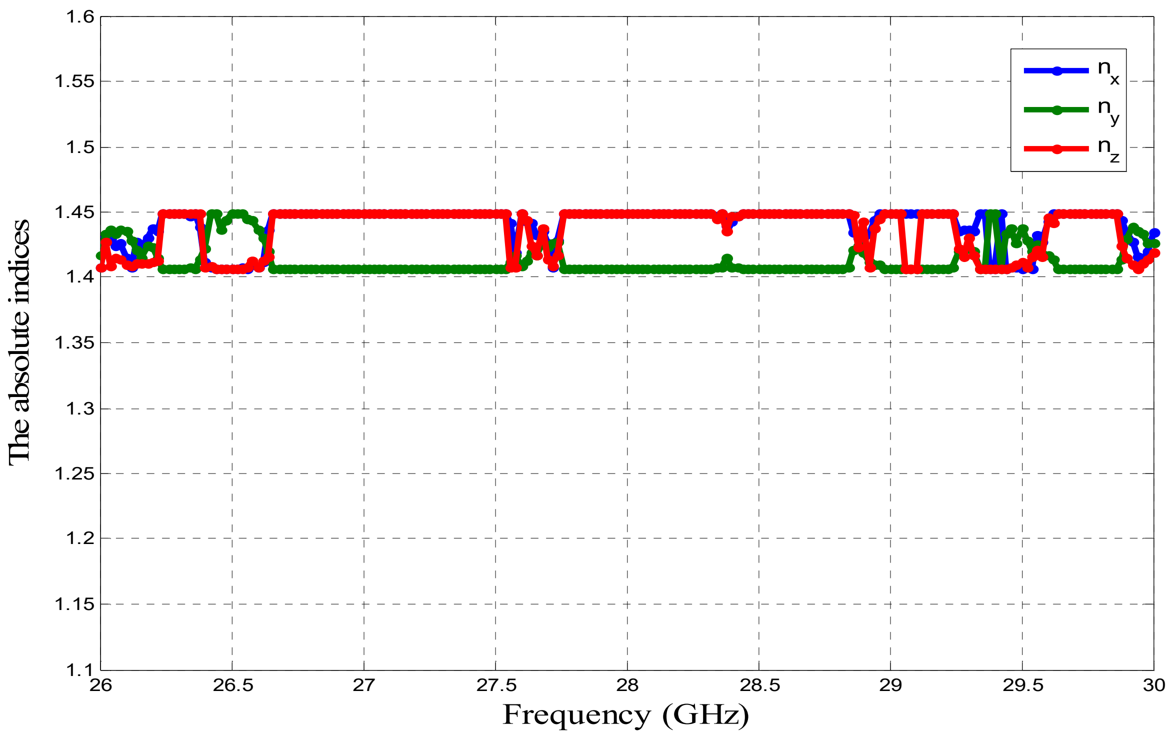

Fig. 6. Variation of absolute indices as a function of frequency

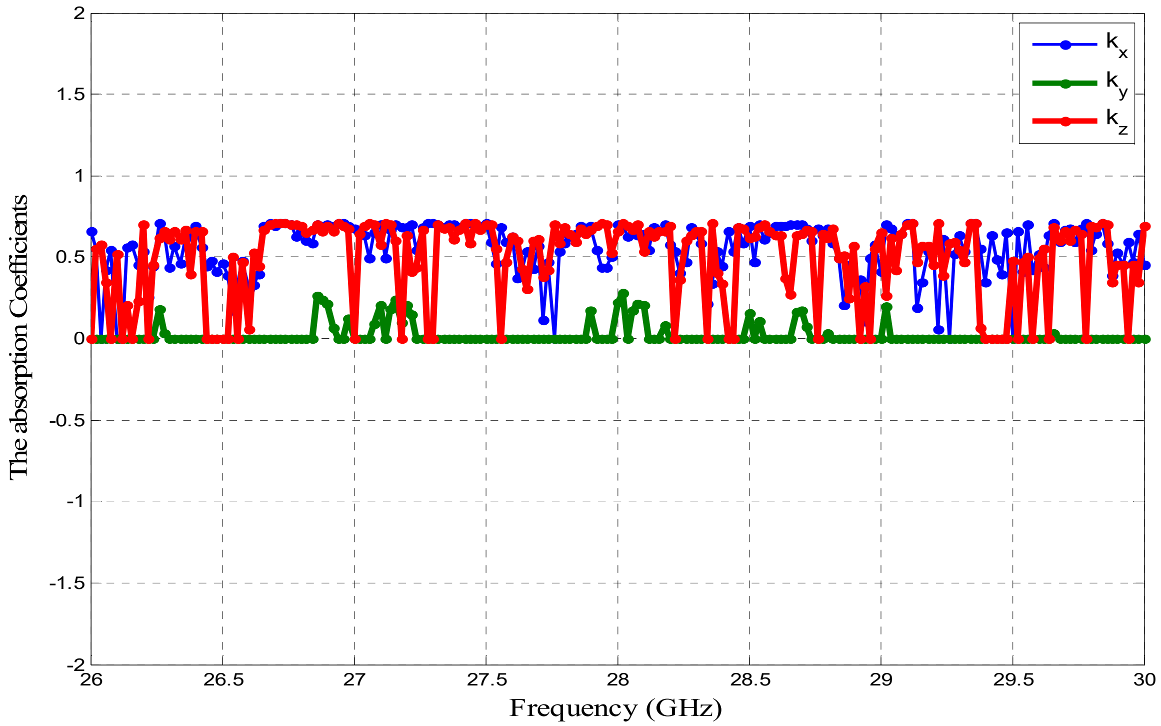

Fig. 7. Variation of absorption coefficients as a function of frequency

The curve of Fig. 6 shows the evolution of the absolute indices as a function of the frequency and that of Fig. 7 also shows the evolution of the absorption coefficients as a function of frequency. For the case of the absolute index curve, we note that the values of the indices are between 1.41 and 1.45. These values are around the values of the absolute index of PTFE which is of the order of 1.449. The absorption coefficients are close to 0 because the PTFE is known as an isotropic material not very absorbent. Therefore, we can say that the method of characterization we proposed makes it possible allows to determine the absolute index of PTFE along three directions.

5 CONCLUSION

In this paper, we presented the principles of a three-detector free-space polarimetric measurement method for determining the refractive indices of an isotropic material using the ellipsometric principle model of an anisotropic medium. We studied its feasibility with PTFE samples whose the results were compared to those which are expected. To do so, we introduced the One Path Two Ports calibration method, a method among two-port calibration methods used in the microwave domain to determine measurement errors. The applications of this characterization method can be extended to a wide range for non-transparent materials such as epoxy, extruded plastics, etc.

REFERENCES

[1] S. Biju Kumar, U. Raveendranath, P. Mohanan, K.T. Mathew, M. Hajian, and L.P. Ligthart, *A simple free-space methode for mearsuring the complex permittivity of single and compound dielectric materials*, Microwave Opt Technol Lett 26, 117–119, 2000.

[2] F. Sagnard, F. Bentabet, and C. Vignat. *In situ measurements of the complex permittivity of materials using reflection ellipsometry in the microwave band* : Experiments (part II). IEEE Trans Instrum Meas 54, 1274–1282, 2005.

[3] A.M. Nicolson and G.F. Ross, *Measurement of the intrinsic properties of materials by time-domain techniques*, IEEE Trans Instrum Meas 19, 377–382, 1970.

[4] F. GAMBOU, B. Bayard and G. Noyel, *Characterization of materiel anisotropy using microwave ellipsometry*, Micorwave and Optical Technology Letters 53, pp 1996-1998, 2011.

[5] A. Moungache, B. Bayard, Abakar Mahamat Tahir, S. Robert, D. Jamon and F. Gambou, *Measurement of refraction index of thick and non-transparent isotropic material using transmission microwave ellipsometry*, Micorwave and Optical Technology Letters 57, Issu 4, pp 1006–1013, 2015.

[6] P. Yeh. *Optics of anisotropic layered media: a new 4x4 matrix algebra*, Surf. Sci, 96, 41-53, 1980.

[7] Mathias Schubert. *Infrared Ellipsometry on Semiconductor Layer Structures*, volume 209 of Springer tracts in Modern Physics. Springer, 2004.

[8] Hiroyuki Fujiwara. *Spectroscopic Ellipsometry.* National Institute of Advanced Industrial Science and Technology, Ibaraki, Japan, 2007.

[9] P. Yeh, *Electromagnetic propagation in birefringent layered media* : J. Optic. Soc. Am, 69, 742-756, 1979.

[10] R.C Jones, *A new calculus for the treatment of optical systems*, *I. Description and discussion of the calculus*, J.opt. SOC, AM. 31, 488-493, 1941.

[11] R. M. A. A. N. M. Bashara, *Ellipsometry and polarized light, Amsterdam*: North Holland: Elsevier Science B. V, 1996.

[12] M. Schubert, *Polarization-dependent optical parameters of arbitrarily anisotropic homogeneous layered systems*, Phys. Rev. B, 53, 4265–4274, 1996.

[13] M. Schubert, *Theory and application of generalized ellipsometry, in Handbook of Ellipsometry*, edited by H. G. Tompkins and E. A. Irene, Chapter 9, 637–717, William Andrew, New York 2005.

[14] B. Hall, *VNA error models: Comments on EURAMET/cg-2/v.01*, ANAMET Report 051, Measurement Standards Laboratory of New Zealand Lower Hutt, New Zealand, 2010.

[15] S. K. a. K. K. Masanobu Hirose, «*Masanobu Hirose, SatAntenna Measurements by One-Path Two-Port Calibration Using Radio-on-Fiber ExtendedPort Without Power Supply*» IEEE TRANSACTIONS ON INSTRUMENTATION AND MEASUREMENT, vol 56, N° 2,pp. 397-400, 2007.

[16] D. Rytting, "*Network analyzer error models and calibration methods*", Palo Alto, CA, Hewlett Packard Inc., 1998.

[17] A. Henze, "*Incomplete 2-Port VNA calibration methods*", INTI, (unpublished) 2014.

[18] K. P. M. H. J. R. R. S. Rajesh, "*The effect of filler on the temperature coefficient of the relative permittivity of PTFE/ceramic composites*", Physica B : Condensed Matter, vol. 406, no. 22, pp. 4312-4316, 2011.

[19] H.-C. L. a. Y.-D. L. Y.-C. Chen, "*The Effects of Filler Content and Size on the Properties of PTFE/SiO2, Composites*," Journal of Polymer Research, vol. 10, issu 4, pp. 247-258, 2003.

[20] M. J. P. M. a. M. S. G. Subodh, *PTFE/Sr2Ce2Ti5O16 polymer ceramic composites for electronic packaging applications*", Journal of the European Ceramic Society, vol. 27, no. 8, pp. 3039-3044, 2007.