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DIELECTRIC MEASUREMENTS FOR LOW-LOSS MATERIALS USING TRANSMISSION PHASE-SHIFT METHOD

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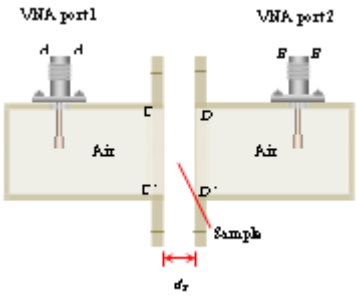
Graphical abstract



Abstract

This paper presents a calculation of the dielectric properties of low-loss materials using the transmission phase-shift method. This method can provide calibration-independent and position-insensitive features, and it was verified experimentally by measuring several well-known samples using X-band rectangular waveguides.

Keywords: Rectangular waveguides, dielectric constant, loss factor, transmission phase-shift, sample thickness

Abstrak

Kertas kerja ini membentangkan pengiraan sifat dielektrik untuk bahan lengai dengan menggunakan kaedah penghantaran cara anjakan-fasa. Kaedah ini bebas daripada penentukuran dan tidak bersandar kepada kedudukan bahan yang diletakkan dalam pandu gelombang. Kaedah ini telah disahkan secara eksperimen dengan mengukur beberapa sampel yang terkenal dengan menggunakan X-band pandu gelombang yang berbentuk segi empat.

Kata kunci: Pandu gelombang berbentuk segi empat, pemalar dielektrik, faktor susutan, penghantaran cara anjakan-fasa, ketebalan sampel

1.0 INTRODUCTION

Conventionally, the relative permittivity, ε_r, and relative permeability, μ_r, of a sample filled in a waveguide are obtained by measuring and converting the complex reflection coefficient, S_{11}, and the complex transmission coefficient, S_{21}, by using Nicholson-Ross-Weir (NRW)[1-2] or improved NRW routines.[3-5, 8-9] Some methods for matching the propagation boundaries have been proposed that require the measurement of only one of these properties for non-magnetic, thin samples. However, the phase of the measured S_{11} and S_{21} depends on the position of the calibration reference plane and on consistent positioning of the sample in the waveguide. The uncertainty of phase shift in the measured S_{11} and S_{21} can result in an incorrect prediction of the permittivity of the sample. Thus, normally, the calibration plane is referred to the air-material interface, and the thickness of the sample is essentially constant. Another problem that can occur in the broadband measurements of S_{11} and S_{21} is phase-shift ambiguity. Although, the thickness, d, of the material should be less than 25% of the operational wavelength, i.e., $\lambda/4$, this condition does not guarantee the elimination of phase ambiguity over a range of operational frequencies. In earlier work, the ambiguity problem was solved by using group-delay analysis[1-2] and iterative/non-iterative methods.[3-4] Recently, several new calibration-independent and material position-invariant techniques [10-12] have been used to reduce the complexity of the de-embedding procedures and to provide techniques that are applicable for different thicknesses of samples when using this measurement method. In their calibration procedures, most of these new techniques require obtaining the solution of a matrix or the solution of matrix determinants.[10, 12] In addition, the prediction of the tangential loss, tan δ, for low-loss samples from measured S_{11} and S_{21} lacks sufficient resolution.[9] It is well known that resonance measurement techniques are good choices for determining low tan δ values, but such techniques cannot be used for the measurement of swept frequency. This paper presents the simple calculation of dielectric properties based on the difference in the phase shifts for the measured S_{21} between the air and the sample, which was capable of handling the above issues. In this work, formulations were explicitly expressed for the dielectric constant, ε_r', and the loss factor, ε_r''. This method does not involve having to make an initial estimate of the anticipated value or solving a matrix. However, to make these calculations, the samples were assumed to be homogeneous, isotropic, and non-magnetic isotropic, and non-magnetic (μ_r=1).

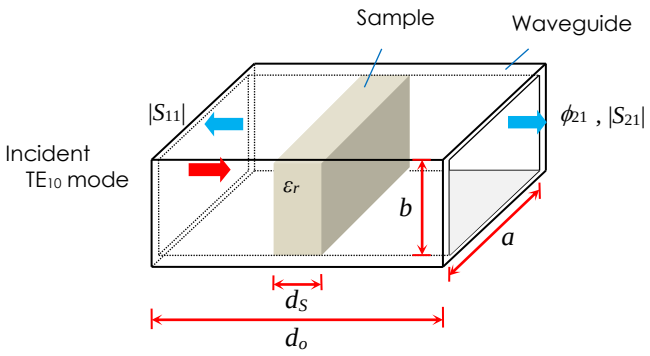


Figure 1 Rectangular waveguide with a sample of d_S thickness.

2.0 PRINCIPLE OF THE TRANSMISSION LINE

The transmission (S_{21}) /reflection (S_{11}) measurement using rectangular waveguide is shown in Fig. 1, in which a homogeneous and isotropic sample slab with thickness d_S is partly placed in the rectangular waveguide.

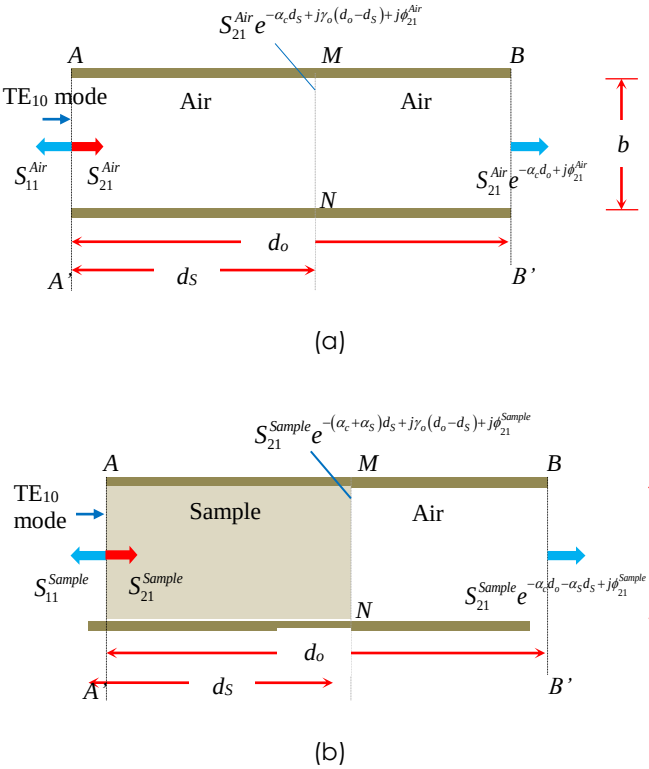


Figure 2 (a) Model of a transmission line: propagating TE_{10} wave, attenuation and phase shifted inside waveguide. (b) A sample of d_S thickness filled in waveguide holder with length of d_S.

For the TE_{10} propagation mode, the relationship between the transmission phase shift, ϕ_{21}^{Air} (in radians), and the propagation constant, γ_o, in an air-filled rectangular waveguide with the length of d_o, can be represented by a model (cross-sectional view of rectangular waveguide) as shown in Fig. 2 (a). The shifted phase, ϕ_{21}^{Air} (at reference *BB'*), can be embedded to surface, *MN*, which has a length of d_S from *AA'*:[2]

$$\begin{aligned} &-\alpha_C d_S + j\left[\phi_{21}^{Air} + \gamma_o\left(d_o - d_S\right)\right] \\ &= -j\gamma_o d_S \\ &= -jd_S\sqrt{k_o^2 - \left(\frac{\pi}{a}\right)^2} \end{aligned} \quad (1)$$

where α_C is the conductor attenuation constant (in nepers/meter). When the waveguide is filled with a sample that has a thickness of d_S (in meter), as shown in Fig. 2 (b), the phase shift, ϕ_{21}^{Sample} (in radians) from reference surface, *AA'* to surface *MN*, will be changed based on its new propagation constant, γ_S, as shown:

$$\begin{aligned} &-\left(\alpha_C + \alpha_S\right)d_S + j\left[\phi_{21}^{Sample} + \gamma_o\left(d_o - d_S\right)\right] \\ &= -j\gamma_S d_S \\ &= -jd_S\sqrt{k_o^2\left(\varepsilon_r' - j\varepsilon_r''\right) - \left(\frac{\pi}{a}\right)^2} \end{aligned} \quad (2)$$

where $k_o = 2\pi f/c$ is the propagation constant of free space (c = 2.99792458 ms^{-1}); a (in meter) are the width of the aperture of the waveguide, respectively (X-band: a = 0.02286 m, b = 0.01016 m); $\varepsilon_r = \varepsilon_r' - j\,\varepsilon_r''$ and α_S (in nepers/meter) are the relative permittivity and the dielectric attenuation constant for the sample, respectively. The α_S is calculated from:

$$\alpha_S \approx -1.15129254\left[\begin{array}{l} \log_{10}\left(\left|S_{11}^{Sample}\right|^2 + \left|S_{21}^{Sample}\right|^2\right) \\ -\log_{10}\left(\left|S_{11}^{Air}\right|^2 + \left|S_{21}^{Air}\right|^2\right) \end{array}\right] \quad (3)$$

where $|S_{11}^{Sample}|$ and $|S_{21}^{Sample}|$ are the measured linear magnitudes of the reflection coefficient and the transmission coefficient for the sample, respectively.

The difference between the phase shift of (1) and the phase shift of (2) can be written as:

$$\alpha_S d_S + j\left(\phi_{21}^{Air} - \phi_{21}^{Sample}\right) = -j\left(\gamma_o - \gamma_S\right)d_S \quad (4)$$

From (4), the dielectric constant, ε_r' and the loss factor, ε_r'' of the sample can be expressed as:

$$\varepsilon_r' = \frac{1}{k_o^2}\left\{\left(\sqrt{k_o^2 - \left(\frac{\pi}{a}\right)^2} + \frac{\phi_{21}^{Air} - \phi_{21}^{Sample}}{d_S}\right)^2 + \left(\frac{\pi}{a}\right)^2 - \alpha_S^2\right\} \quad (5)$$

$$\varepsilon_r'' = \frac{2\alpha_S}{k_o^2}\left(\sqrt{k_o^2 - \left(\frac{\pi}{a}\right)^2} + \frac{\phi_{21}^{Air} - \phi_{21}^{Sample}}{d_S}\right) \quad (6)$$

However, (5) and (6) are only applicable for the ideal case. Normally, the dielectric constant, ε_r' of the material can be accurately predicted using (5) and (6) at the center operating frequency, f_{center} of the waveguide (X-band: f_{center}=10.3 GHz). For broadband measurements, (5) and (6) should be modified by multiplying (5) with *A* term as:

$$\varepsilon_r' = \frac{A}{k_o^2}\left\{\left(\sqrt{k_o^2 - \left(\frac{\pi}{a}\right)^2} + \frac{\phi_{21}^{Air} - \phi_{21}^{Sample}}{d_S}\right)^2 + \left(\frac{\pi}{a}\right)^2 - \alpha_S^2\right\} \quad (7)$$

$$\varepsilon_r'' = \frac{2A\alpha_S}{k_o^2}\left(\sqrt{k_o^2 - \left(\frac{\pi}{a}\right)^2} + \frac{\phi_{21}^{Air} - \phi_{21}^{Sample}}{d_S}\right) \quad (8)$$

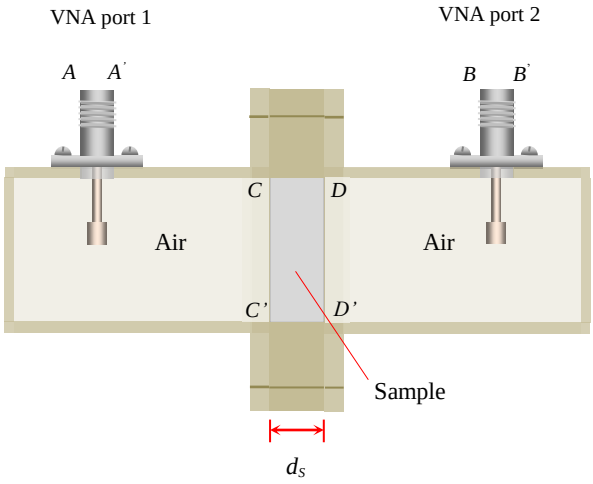
where

$$A = \left(\frac{f}{f_{center}}\right)^{\beta}$$

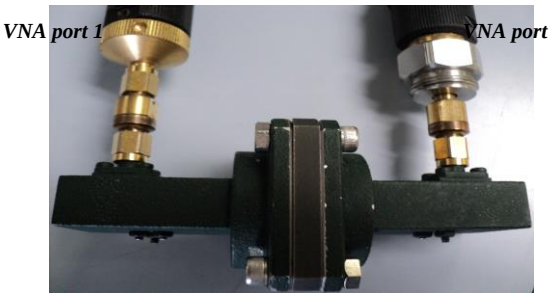
Symbol f is the operating frequencies. The β is a constant value depended on the model and the quality of the rectangular waveguide. In this study, the β is equal to 0.3. In fact, the *A* coefficient is used to correct the imperfection of propagation wave (such as VSWR effects) in the rectangular waveguide at lower or upper limit of the operating frequencies.

3.0 MEASUREMENT SETUP

The linear magnitude, $|S_{11}|$, $|S_{21}|$, and the phase shift, ϕ_{21}, for air and the sample were measured with an Agilent E5071C vector network analyzer (VNA) using two X-band rectangular waveguides (Flann 16094-SF40 Model waveguide adaptors) from 8.2 GHz to 12.4 GHz. For the conventional measurement techniques (such as NWR method or Keysight 85071E software), the thru-reflect-line (TRL) calibration should be done on the surface of *CC'* and *DD'* which are the exact surface between the samples and the waveguide as shown in Fig. 3 (a). Thus, the position of the sample in the waveguide is dependent on the calibration plane. However, in this study, either open-short-load-thru (OSLT) or TRL calibration is required. The OSLT calibration is normally applied to VNA-ports on *AA'* and *BB'* planes, respectively. If there is existing waveguide calibration tool kits, thus, the calibration process can be performed on plane *CC'* and *DD'*, respectively. The sample with thickness d_S is permitted to place in any position in the rectangular waveguide, which does not affect the accuracy of the measurement.

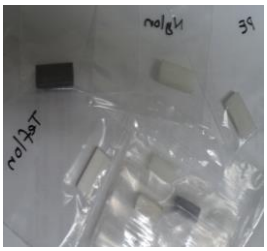


(a)



(b)

(c)



(d)

Figure 3 (a) Cross-sectional view of experimental setup. (b) Actual measurement setup. (c) Customized sample holders. (d) Well known dielectric samples in plastic cover.

The actual measurement setup is shown in Fig. 3 (b). The nylon 6 (d = 5 mm), Teflon (d = 5 mm), PVC (d = 5 mm), and FR4 (d = 4.74 mm) samples, respectively, were placed in the waveguides and measured for

Table 1 Absolute errors due to ±0.1 radian deviation in $\left(\phi_{21}^{Air} - \phi_{21}^{Sample}\right)$

d_S (m)	± Δ ε_r'		± ($\Delta\varepsilon_r''/\Delta\varepsilon_r'$)	
	10 GHz	15 GHz	10 GHz	15 GHz
0.01	0.07	0.05	0.015	0.010
0.005	0.15	0.10	0.015	0.010
0.001	0.80	0.56	0.014	0.009

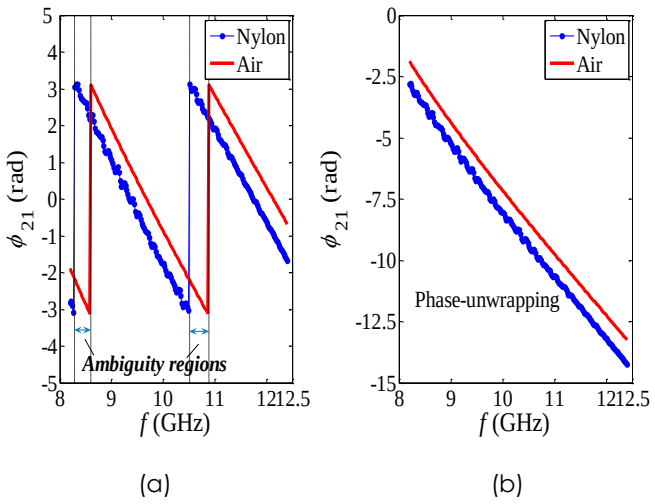
Table 2 Dielectric constant and loss tangents

Material	ε_r'		
	1-10 GHz	12.4 GHz	This Study 10.3 GHz
Teflon	2.2 – 1.9[15]	2.11 ± 0.05[16]	2.07
Nylon	3.5 – 2.5[15]	–	2.99
PVC	2.6 – 2.5[11]	2.88 ± 0.04[16]	2.76
FR4	4.2 – 3.9[17]	–	3.82
Material	**Loss tangent, tan δ**		
	1-10 GHz	12.4 GHz	This Study 10.3 GHz
Teflon	0.0001 – 0.0002[15]	–	0.0002
Nylon	0.01 – 0.05[15]	–	0.022
PVC	0.0016 – 0.0003[11]	0.0079[16]	0.007
FR4	0.018 – 0.022[17]	–	0.056

validation. The dimensions of the samples were precisely machined by using a computer numerical control (CNC) cutter in order to fit the sample into the waveguide holder.

4.0 RESULTS AND ANALYSIS

The uncalibrated measurements took into account the total propagation wavelength (>λ/4) within the length of the waveguide and the length of the sample, which caused phase-ambiguity regions due to the variation of phase shift between air and the sample over a given frequency range, as shown in Fig. 4(a). Thus, the predicted values of ε_r' and ε_r'' would be incorrect. In this study, the phase ambiguity was solved by using MATLAB's 'unwrap' command[13], as shown in Fig. 4(b).



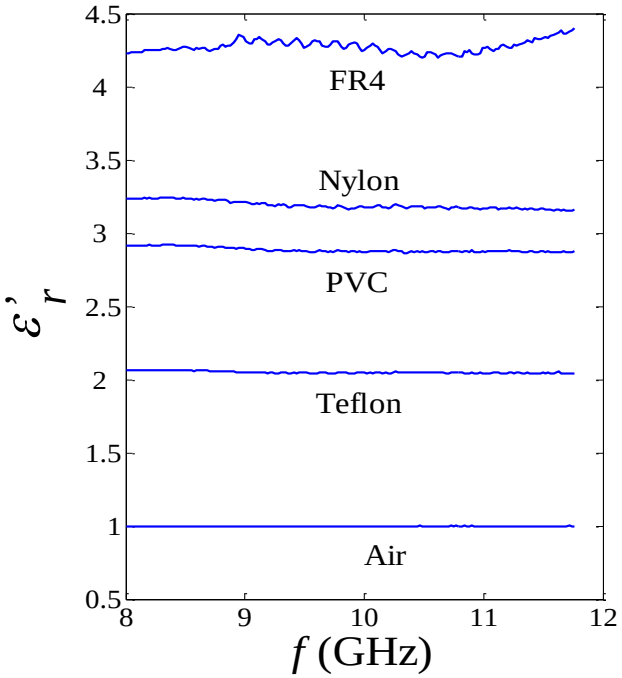
(a) (b)

Figure 4 (a) Phase ambiguity in uncalibrated measurements between air and nylon; (b) Phase ambiguity solved by using the phase-unwrapping method

As known that the conventional NRW method requires sample thickness must be less than λ/4 in order to avoid phase ambiguity in the dielectric measurements. However, for this study method, the minimum thickness of the sample was limited to λ/8. The uncertainty measurement is high for a thin sample due to the decreasing of the sensitivity for the transmitted wave through the sample, especially for transmitted waves that have longer wavelengths. For instance, a deviation of ±0.1 radian in the measurement of $(\phi_{21}^{Air} - \phi_{21}^{Sample})$ for nylon, may cause different absolute errors, i.e., Δ ε_r' and Δ ε_r'', in the prediction of the dielectric properties depending on the thickness, d_s, of the nylon, as shown in Table I. The ε_r' results at 10.3 GHz using this study method were in good agreement with the measurements obtained from literatures,[15-17] as tabulated in Table II.

Fig. 5 (a) and (b) show the measured dielectric constant, ε_r' and loss tangent, tan δ (=$\varepsilon_r''/\varepsilon_r'$), for four types of low loss samples using NWR method (See Appendix) from 8.2 GHz to 11.75 GHz. The ambiguity phase exists when the operating frequency exceeds 11.75 GHz for the 0.5 mm thickness of the samples. The dielectric constant, ε_r' of the same samples were also calculated using Keysight 85071E software for comparison and validation, as illustrated in Fig 6. The predicted values of ε_r' and $\varepsilon_r''/\varepsilon_r'$ versus operating frequencies for the four samples by using (7) and (8), respectively, are shown in Figs. 7 (a), (b) and 8. Clearly, the ε_r' results were in good agreement with the measurements obtained using Agilent 85071E software.[14] It should be noted that the calibration planes for Figs. 5, 6 and 7 cases are refer to plane *CC'* and *DD'*, which the waveguide thru-reflect-line (TRL) calibration was applied to the planes.

On the other hand, for the case of Fig. 8, the calibration is only done at plane *AA'* and *BB'* using coaxial open-short-load-thru (OSLT) technique (Keysight 85052D calibration kits). In this case, the mismatch at the surface of the sample, the waveguide and imperfections in the waveguide introduced noise in the calculations of ε_r' and $\varepsilon_r''/\varepsilon_r'$. In addition, errors in the measurements can also be caused by the air gap that existed between the sample and the metal walls of the waveguide. Moreover, the flatness of the sample surfaces can also be one of the factors too. It was very challenging to get accurate results of the broadband loss tangent ($\varepsilon_r''/\varepsilon_r'$) calculations in the case of Fig. 8 because the values of $\varepsilon_r''/\varepsilon_r'$were too small and they were very sensitive to measurement errors. In fact, the fluctuation of the random noise is larger than the values of $\varepsilon_r''/\varepsilon_r'$. Despite that, the random noise in the broadband dielectric constant measurements can be smoothed using filter techniques[18].



(a) Dielectric constant, ε_r'

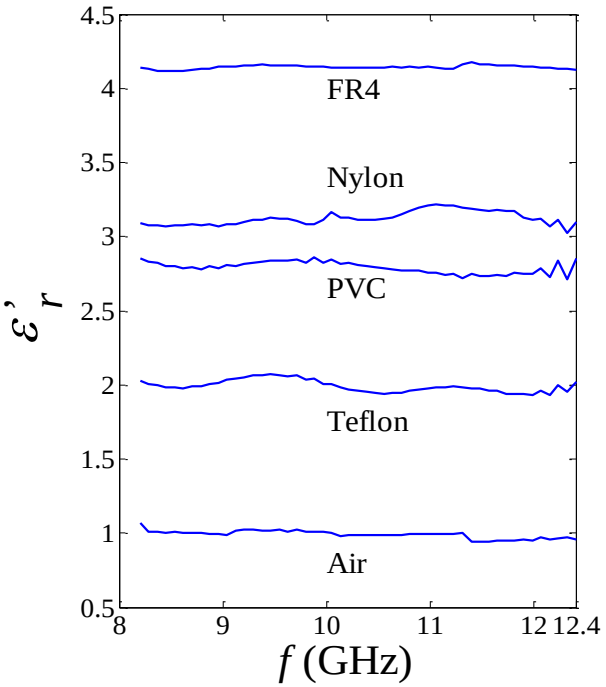
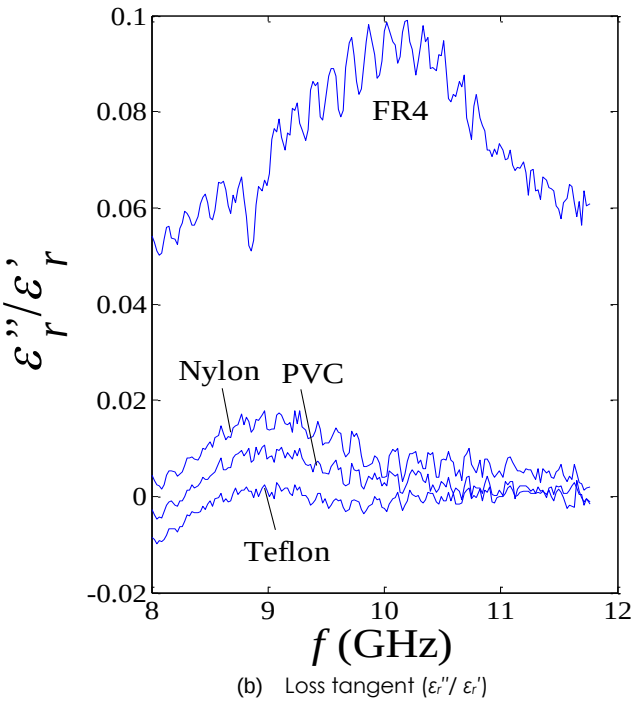
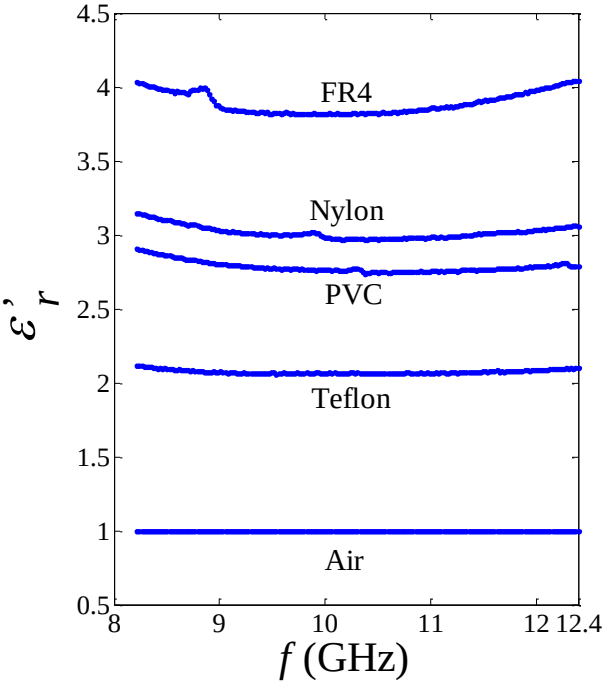


Figure 6 Comparison of measured ε_r' (Keysight 85071E software with waveguide from Flann Inc.).

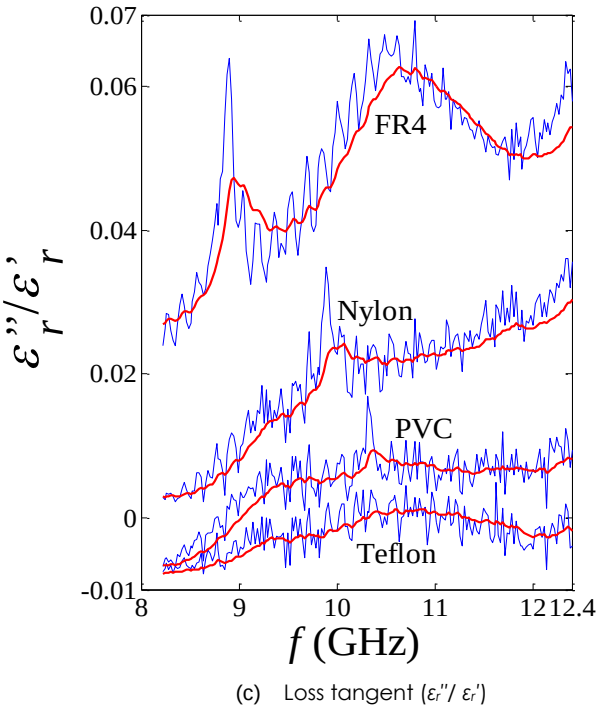


(b) Loss tangent ($\varepsilon_r''/\varepsilon_r'$)

Figure 5 Comparison of (a) measured ε_r' and (b) measured $\varepsilon_r''/\varepsilon_r'$. (NRW Method with waveguide from Flann Inc.).



(a) Dielectric constant, ε_r'



(c) Loss tangent ($\varepsilon_r''/\varepsilon_r'$)

Figure 7 Comparison of (a) measured ε_r' and (b) measured $\varepsilon_r''/\varepsilon_r'$. (with thru-reflect-line (TRL) calibration at plane *CC'* and *DD'*). The red solid lines are the smooth filter lines.

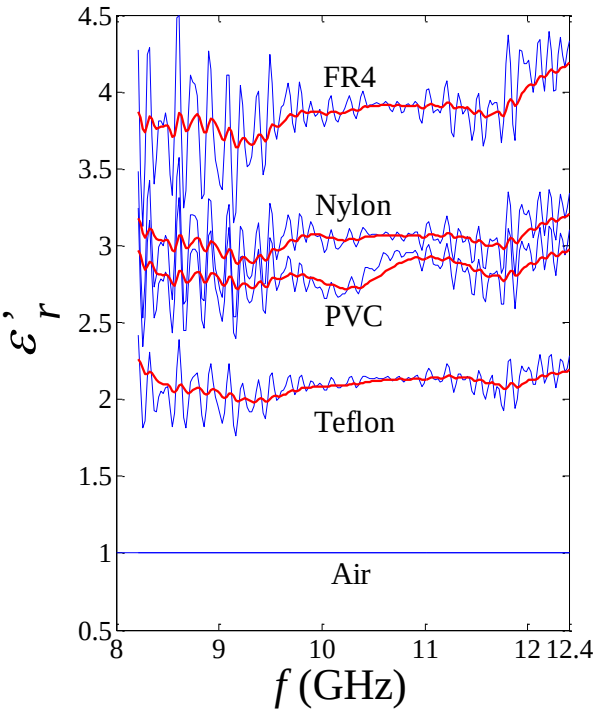


Figure 8 Comparison of measured ε_r'. (with open-short-load-thru (OSLT) calibration at plane *AA'* and *BB'*). The red solid lines are the smooth filter lines.

5.0 CONCLUSION

In this work, equations (7) and (8) were derived to provide broadband calibration-independent and sample position-insensitive techniques for measuring the dielectric properties of low-loss materials.

Acknowledgement

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Appendix

A. Nicolson-Ross-Weir (NRW) Method

The *S*-parameters (S_{11} and S_{21}) at calibrated plane *CC'* and *DD'* are measured by vector network analyzer. The S_{11} is the complex reflection coefficient of the d_s thickness of the sample at plane *CC'*. On the other hand, the S_{21} is the complex transmission coefficient of the d_s thickness of the sample at plane *DD'*.

The actual reflection coefficient, Γ and transmission coefficient, *T* of the infinite sample can be calculated using measured *S*-parameters[1-5]:

$$\Gamma = K \pm \sqrt{K^2 - 1} \quad \text{(A1)} \qquad \text{and} \qquad T = \frac{S_{11} + S_{21} - \Gamma}{1 - \Gamma\left(S_{11} + S_{21}\right)} \quad \text{(A2)}$$

where

$$K = \frac{S_{11}^2 - S_{21}^2 + 1}{2S_{11}} \quad \text{(A3)}$$

The $|\Gamma| < 1$ condition is required in order to find the correct root value using (A1). From Γ and *T*, the relative complex permeability, μ_r of the sample can be predicted as:

$$\mu_r = \frac{\lambda_o}{\sqrt{1-\left(\lambda_o/\lambda_c\right)^2}}\left(\frac{1+\Gamma}{1-\Gamma}\right)\left[\frac{j}{2\pi d_S}\ln\left(\frac{1}{T}\right)\right] \quad \text{(A4)}$$

And the relative complex permittivity, ε_r of the sample is given as:

$$\varepsilon_r = \frac{\lambda_o}{\mu_r}\left\{\frac{1}{\lambda_c^2} - \left[\frac{1}{2\pi d_S}\ln\left(\frac{1}{T}\right)\right]^2\right\} \quad \text{(A5)}$$

The phase ambiguity may lead to ambiguities in retrieving the values of ε_r and μ_r. Thus, the ln(1/*T*) term in (A4) and (A5) is modified as:

$$\ln\left(\frac{1}{T}\right) = \ln\left(\frac{1}{|T|}\right) + j\left(\phi + 2n\pi\right) \quad \text{(A6)}$$

where *n* is the integer value (*n* = 0, 1, 2, …). From the cutoff frequency to the first resonance frequency, *n* is set to zero. On the other hand, from first resonance to second resonance frequency, *n* is set to 1.

B. Alternative Method

For nonmagnetic sample (μ_r =1), the actual reflection coefficient, Γ at plane *CC'*of an infinite dielectric sample ($\varepsilon_r = \varepsilon_r' - j\,\varepsilon_r''$) filled in the waveguide can be simplified as:

$$\Gamma = \frac{1-\sqrt{\varepsilon_r}}{1+\sqrt{\varepsilon_r}} \quad \text{(B1)}$$

On the other hand, the transmission coefficient, *T* at plane *DD'* can be reduced as:

$$T = \exp\left(-\gamma_S d_S\right) \quad \text{(B2)}$$

where γ_S is the propagation constant of the waveguide filled with d_S thickness of sample and given as:

$$\gamma_S = j\sqrt{k_o^2\left(\varepsilon_r' - j\varepsilon_r''\right) - \left(\frac{\pi}{a}\right)^2} \quad \text{(B3)}$$

The measured complex value of S_{21} at plane *DD'* is written as[1-5]:

$$S_{21} = \frac{T\left(1-\Gamma^2\right)}{1-\Gamma^2 T^2} \quad \text{(B4)}$$

Instead (B1), (B2) and (B3) into (B4), yields

$$S_{21} - \frac{\left[1-\left(\frac{1-\sqrt{\varepsilon_r}}{1+\sqrt{\varepsilon_r}}\right)^2\right]\exp\left(-j\sqrt{k_o^2\varepsilon_r - \left(\frac{\pi}{a}\right)^2}\,d_S\right)}{1-\left(\frac{1-\sqrt{\varepsilon_r}}{1+\sqrt{\varepsilon_r}}\right)^2\exp\left(-2j\sqrt{k_o^2\varepsilon_r - \left(\frac{\pi}{a}\right)^2}\,d_S\right)} = 0 \quad \text{(B5)}$$

Find the value of ε_r in (B5) using MATLAB's 'fsolve' command. The value of ε_r is optimized in order to find a root (or zero) of (B5). For Fig. B1 case, (B5) is required to be modified as (B6).

$$S_{21} - R\frac{\left[1-\left(\frac{1-\sqrt{\varepsilon_r}}{1+\sqrt{\varepsilon_r}}\right)^2\right]\exp\left(-j\sqrt{k_o^2\varepsilon_r-\left(\frac{\pi}{a}\right)^2}\,d_S\right)}{1-\left(\frac{1-\sqrt{\varepsilon_r}}{1+\sqrt{\varepsilon_r}}\right)^2\exp\left(-2j\sqrt{k_o^2\varepsilon_r-\left(\frac{\pi}{a}\right)^2}\,d_S\right)} = 0 \quad \text{(B6)}$$

where R is reference plane (at plane DD') transformation as:

$$R = \exp\left[j\sqrt{k_o^2-\left(\frac{\pi}{a}\right)^2}\left(d-d_S\right)\right] \quad \text{(B7)}$$

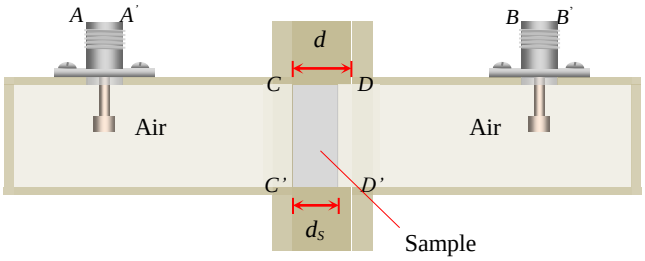

Figure B1 Thickness of sample, d_S less than length of sample holder, d (Distance from plane CC' to plane DD').