PERMITTIVITY MEASUREMENT OF THIN DIELECTRIC MATERIALS FROM REFLECTION-ONLY MEASUREMENTS USING ONE-PORT VECTOR NETWORK ANALYZERS

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Abstract—We have proposed a simple waveguide method for complex permittivity determination of dielectric materials which are not completely filling the entire sample holder. The method reconstructs the permittivity from measured reflection-only scattering parameters by a one-port vector network analyzer of two configurations of the sample holder. It not only eliminates the necessity of any knowledge of the location of the shifted sample inside its holder but also decreases measurement errors occurring with the presence of undesired air gaps, which seriously affect the measurement accuracy of transmission-only measurements, present between the sample and holder walls. Furthermore, the reconstruction of permittivity can be realized by any one-port vector network analyzer, which is less expensive than their two-port counterparts. Therefore, the proposed method is cost-effective. We have analyzed the accuracy of the proposed method and noted a good compromise between the reference data and measured values of permittivities of low-loss polyvinyl-chloride and polytetrafluoro.ethylene samples (less than 8 percent for dielectric constant and less than 15 percent for loss tangent values).

1. INTRODUCTION

Material characterization is an important issue in many material production, processing, and management applications in agriculture, food engineering, medical treatments, bioengineering, and the concrete industry [1]. In addition, microwave engineering requires precise

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knowledge of electromagnetic properties of materials at microwave frequencies since microwave communications are playing more and more important roles in military, industrial, and civilian life [1]. For these reasons, various microwave techniques, each with its unique advantages and constraints [2–29], have been introduced to characterize the electrical properties of materials. These methods can roughly be divided into resonant and non-resonant methods [1].

Resonant methods have much better accuracy and sensitivity than nonresonant methods [1, 13, 14]. They are generally applied to characterization of low-loss materials. In a recent study, it has been shown that they are also applicable to high-loss materials provided that very small samples are prepared or higher volume cavities are constructed [13]. Though, a meticulous sample preparation is needed before measurements. In addition, for an analysis over a broad frequency band, a new measurement set-up (a cavity) must be made. This is not feasible from a practical point of view. Tunable resonators can be used for a wider frequency band analysis; nonetheless, they are expensive and an increase in the frequency bandwidth accompanies a decrease in the accuracy.

Non-resonant methods have relatively higher accuracy over a broad frequency band and necessitate less sample preparation compared to resonant methods [1, 15]. Due to their relative simplicity, non-resonant waveguide (or coaxial) transmission/reflection methods are presently the most widely used broadband measurement techniques [1]. These methods have effectively been applied to determine the relative complex permittivity (ε) of thin materials [9, 12, 16–18]. It is not generally possible to locate thin samples to completely fill a coaxial line or rectangular waveguide section. In this circumstance, the transformation of scattering (S-) parameters from the calibration (reference) plane to the sample end surfaces (measurement plane) has to be done [19]. Such a transformation may result in enormous errors for phase measurements of reflection S-parameters. On the other hand, transmission S-parameter measurements are not affected if the sample length and the sample holder length both are precisely known [12, 16, 17, 20]. This is because transmission measurements take longitudinal averaging of variations in sample properties [21]. To overcome the problems arising from reflection S-parameters, transmission-only measurements can be employed [9, 18]. Although the method in [18] does not require complete filling of the cross section of a waveguide section, it requires adequate sample thickness in order to obtain accurate measurement result. As a solution to this problem, a transmission-only waveguide method can be utilized [9]. Although the derivations are independent upon any offset of the sample inside its holder, it is sensitive to sample

thickness. As another solution to the measurement errors arising from reflection S-parameters, we have lately proposed various amplitude-only methods [22–27]. While the methods in [22, 23, 25] are proposed for ε determination of medium- and low-loss materials with substantiate lengths, those in [24, 26] are for ε determination of high-loss materials which possess at least 10 dB attenuation. Although that in [27] is effective in eliminating the phase-shift problems for ε measurement of thin materials, it is not much applicable for dispersive materials since it assumes that electrical properties of thin materials do not considerably change with frequency.

Another problem in ε measurement of thin materials is the measurement errors arising from the presence of undesired air gaps between the sample transverse surfaces and inner waveguide walls [28]. This circumstance seriously affects the accuracy of transmission S-parameter measurements more than that of reflection S-parameters. This is because, for transmission measurements, the electromagnetic waves must pass through the sample as well as air regions between the sample transverse surfaces and inner waveguide walls while reflection measurements are mainly depended on surface properties. Despite their advantages, reflection measurements suffer from phase measurements. Direct location of the sample into waveguide or coaxial section away from the reference plane cannot solve this problem since exact location of the shifted sample must be known [9]. We have recently proposed a method to eliminate any errors arising from inaccurate knowledge of sample location inside the waveguide [29]. Although it is attractive and feasible, it experiences problems arising from imprecise information about both electrical properties and length of sample holders. A promising solution to the aforementioned problems is to employ phase-shift-invariant reflection-only measurements for ε determination of thin materials.

For ε determination of materials, two-port vector network analyzers (VNAs) are generally employed since they are highly accurate and perform spectral forward and inverse S-parameter measurements in a short time. The aforementioned phase-shift-invariant reflection-only measurements can easily be obtained using a two-port VNA such as HP8720C [15]. However, such measurements are not easy to conduct using one-port VNAs, which are widely employed for measurements below 6 GHz (e.g., HP 8752C and HP 8714B) or at millimeter waves (e.g., Anritsu 37000 series with 3740/3741 modules) [30], since they are capable of only forward S-parameter measurements. The advantage of one-port VNAs over their two-port counterparts is its cost. The motivation of this study is to demonstrate that the abovementioned phase-shift-invariant reflection-

only measurements can also be attained using any one-port VNA. The proposed method eliminates the measurement errors arising from inaccurate knowledge of the location of the sample inside its holder in a simple fashion.

2. THE METHOD

2.1. Background

The problem for ε measurement of a thin material with length L inside a waveguide is shown in Fig. 1. The sample is arbitrarily located between reference planes (P1 and P2). In the analysis, it is assumed that the sample is isotropic, symmetric and homogenous.

The expressions of electric and magnetic fields can be derived from their vector potentials (or Hertzian vectors), $\vec{A}$ and $\vec{F}$, such as [31]

$$\vec{E} = -j\omega\vec{A} - j\frac{1}{\omega\mu_0\varepsilon_0\varepsilon}\nabla\left(\nabla\cdot\vec{A}\right) - \frac{1}{\varepsilon_0\varepsilon}\nabla\times\vec{F} \tag{1}$$

$$\vec{H} = \frac{1}{\mu_0}\nabla\times\vec{A} - j\omega\vec{F} - j\frac{1}{\omega\mu_0\varepsilon_0\varepsilon}\nabla\left(\nabla\cdot\vec{F}\right). \tag{2}$$

where ε_0 and μ_0 are the permittivity and permeability of free-space. Assuming that the rectangular waveguide operates in the dominant mode (TE$_{10}$) and applying boundary conditions (continual of tangential components of electric and magnetic fields at discontinuities of sample-air interfaces) to field quantities, forward reflection and transmission S-parameters between reference planes P1 and P2 can
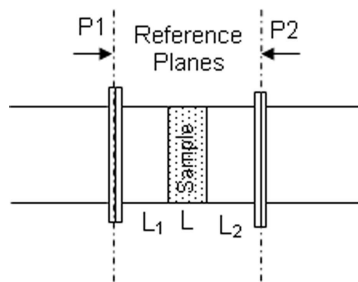

Figure 1. Illustration of the problem: permittivity determination of thin materials located between reference planes in a rectangular waveguide.

be written as [15, 31]

$$S_{11} = |S_{11}| e^{j\theta_{11}} = R_1^2 \Gamma \frac{\left(1 - T^2\right)}{1 - \Gamma^2 T^2}, \tag{3}$$

$$S_{21} = |S_{21}| e^{j\theta_{21}} = S_{12} = R_1 R_2 T \frac{\left(1 - \Gamma^2\right)}{1 - \Gamma^2 T^2}, \tag{4}$$

where $|\cdot|$ denotes the magnitude of expressions; Γ and T are, respectively, the reflection coefficient when the sample is infinite in length and the propagation factor; and R_1 and R_2 are the calibration plane transformation factors. Their corresponding equations are

$$\Gamma = \frac{\gamma_0 - \gamma}{\gamma_0 + \gamma}, \; T = \exp\left(-\gamma L\right), \tag{5}$$

$$R_1 = \exp\left(-\gamma_0 L_1\right), \; R_2 = \exp\left(-\gamma_0 L_2\right), \tag{6}$$

$$\gamma_0 = j 2\pi / \lambda_0 \sqrt{1 - \lambda_0^2 / \lambda_c^2}, \; \gamma = j 2\pi / \lambda_0 \sqrt{\varepsilon - \lambda_0^2 / \lambda_c^2}. \tag{7}$$

Here, L_1 and L_2 are the distances between the calibration plane and the sample end surfaces; $\lambda_0 = c/f$ and $\lambda_c = c/f_c$ correspond to the free-space and cut-off wavelengths; and f, f_c, and c are the operating and cut-off frequencies and the speed of light in vacuum, respectively.

The problem is to measure the ε of thin materials arbitrarily located inside the sample holder in Fig. 1 using reflection-only measurements at one fixed frequency using any one-port VNA.

2.2. Permittivity Determination

For ε determination of thin materials by the proposed method from reflection-only measurements of a one-port VNA, we utilize the measurement configurations in Fig. 2.

While the measurement configuration in Fig. 2(a) corresponds to sample holder (a waveguide section) in which the sample is arbitrarily positioned, that in Fig. 2(b) shows the configuration pertaining to the inversed sample holder. In each configuration in Fig. 2, calibration planes coincide with each other and it is assumed that sample holder is terminated by a matched waveguide load.

Using S_{11} measurements of these two configurations, we obtain

$$S_{11} = R_1^2 \Gamma \frac{\left(1 - T^2\right)}{1 - \Gamma^2 T^2}, \tag{8}$$

$$S_{11}^{in} = R_2^2 \Gamma \frac{\left(1 - T^2\right)}{1 - \Gamma^2 T^2} \tag{9}$$

where the superscript 'in' denotes the measurements when the sample holder is inversed and R_1, R_2, Γ and T are given in (5) and (6).
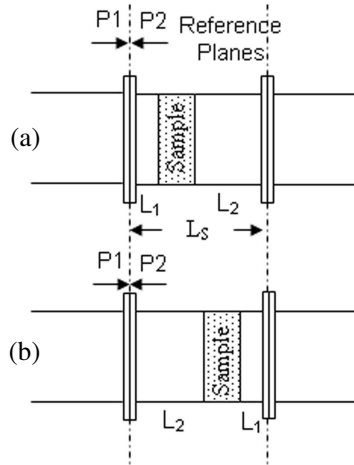
Figure 2. Measurement configurations for permittivity determination of thin materials by the proposed method: (a) sample holder in which the sample is arbitrarily located and (b) inversed sample holder.

Using (8) and (9), we find the difference between L_1 and L_2 as

$$L_1 - L_2 = \frac{1}{2\gamma_0} \left[\ln \left(S_{11}^{in}/S_{11} \right) \mp j2\pi n \right], \qquad (10)$$

where $n = 0, 1, 2, 3, \ldots$.

Assuming the length of the sample holder (L_s) is known, we can write another expression for L_1 and L_2 as

$$L_1 + L_2 = L_s - L. \qquad (11)$$

Combining (10) and (11) and eliminating L_2, we find L_1 as

$$L_1 = \frac{1}{4\gamma_0} \left[\ln \left(S_{11}^{in}/S_{11} \right) \mp j2\pi n \right] + \frac{(L_s - L)}{2}. \qquad (12)$$

After measuring L_1 from (12) and substituting it into (6), we can determine R_1. Then, any suitable method based on reflection S-parameter measurements such as in [16] can be employed. Although explicit expressions for ε_r are available for a known sample length in [16], in this research paper, we employ least-squares-minimization technique [32] as

$$\min \left\| S_{11}^m \left(\Gamma, T \right) - S_{11}^p \left(\Gamma, T \right) \right\|. \qquad (13)$$

Here, the superscript 'm' denotes the measured quantity whereas the superscript 'p' designates the predicted quantity.

It is seen from (12) and (13) that although L_1 and thus ε_r are functions of L, as in the case for ε_r determination of thin materials in [9], the proposed method employs reflection-only measurements for the sample purpose. In this way, the proposed method not only eliminates the measurement uncertainties arising from air gaps present between sample surfaces in contact with the waveguide inner walls and waveguide, but also offers an attractive method for accurate ε_r of thin materials using any one-port VNA.

For accurate L_1 measurements from (12), we assumed that the length of the sample holder is known. Its length can precisely be measured using a micrometer. Another option is to measure it using S_{21} measurements at closely or largely separated frequencies [27]. The main idea behind this simple approach is that, for S_{11} and S_{21} measurements at different frequencies, the length of the sample holder does not change. What changes is its electrical length only.

It is obvious from (12) that, to measure L_1 or L_2, the value of n must be known. To determine which n value corresponds for a measurement configuration, we can employ measurements at different frequencies, as discussed in previous paragraph. However, this time we are not given liberty at selecting measurements at different frequencies. For small frequency shifts, we can determine n [33]. To elaborate on this, for our problem, using (10) for two frequencies, the following criterion should be satisfied

$$|\Delta L| = |L_2 - L_1| < \frac{\lambda_{02}\lambda_0}{2\left|\lambda_0\sqrt{1 - \lambda_{02}^2/\lambda_c^2} - \lambda_{02}\sqrt{1 - \lambda_0^2/\lambda_c^2}\right|}, \qquad (14)$$

where λ_{02} corresponds to the free-space wavelength at another frequency, f_2, and we assume that ε_r does not alter with the frequency shift. It is clear from (14) that, in order to increase the possibility to satisfy the criterion in (14), either the difference length $|\Delta L|$ should be made smaller enough (sample should be located almost at the center of the sample holder) or the difference between wavelengths at two different frequencies should be lesser, or both. This can easily be recognized by letting $\lambda_c \to \infty$ in (14). This circumstance also shows that the criterion in (14) can also be used in coaxial-line or free-space measurements.

3. UNCERTANTY ANALYSIS

Several factors affect the accuracy of ε_r determination by the proposed method as: 1) the uncertainty in measured S-parameters; 2) errors in the sample length and the holder length; 3) the uncertainty in reference plane positions; 4) guide losses and conductors mismatches;

5) air gaps between the external surfaces of the sample (and holder) and inner walls of waveguides; and 6) higher-order modes. All these uncertainties are extensively treaded in the literature [15, 20, 34, 35]. In this research paper, the two pointof- interest uncertainties are the effect of air gaps between external surfaces of the sample and inner walls of waveguides and the effect of air gaps present at the flanges of waveguides at reference planes in Fig. 2. The correction for the former uncertainty can be taken into account by applying the transverse resonance condition [31, 36] to the regions of the material and the air gap inside the waveguide. Since the derived equation from this condition [28] requires the knowledge of the average height of the air gap, its correction mainly depends on the prepared samples [20]. In addition, for small air gaps, employing a conducting paste to the external surfaces of the sample will reduce the errors.

The latter uncertainty factor can be analyzed as follows. Firstly, we represent the air gap between the planes P1 and P2 in Fig. 2 as

$$S_{11}^x = R_x^2 S_{11}, \ R_x = \exp\left(-\gamma_0 L_x\right), \tag{15}$$

where L_x denotes the length of this air region. Then, we apply the differential uncertainty model [37] to (15) and (8) and obtain

$$\frac{\partial \varepsilon_r}{\partial L_x} = \frac{-2\gamma_0 S_{11}}{\left(\frac{\partial S_{11}}{\partial \Gamma} \frac{\partial \Gamma}{\partial \varepsilon_r} + \frac{\partial S_{11}}{\partial T} \frac{\partial T}{\partial \varepsilon_r}\right)}. \tag{16}$$

As a result, the expression in (16) allows us to assess the effect of any air gap between planes P1 and P2. For example, Figs. 3(a) and 3(b), respectively, demonstrate the dependence of $\partial \varepsilon_r / \partial L_x$ over normalized sample length (L/λ_s where λ_s is the wavelength of the sample) for two different values of ε_r as representatives for low-loss materials and $f = 10\,\text{GHz}$ and $f_c = 6.555\,\text{GHz}$.

It is seen from Figs. 3(a) and 3(b) that $\partial \varepsilon_r / \partial L_x$ considerably decreases at multiple half-wavelengths. This is a similar tendency reported in [15, 23, 34]. The main reason of this can be the diminishing value of $|S_{11}|$ in (16) at multiple half-wavelengths. In addition, it is seen that $\partial \varepsilon_r / \partial L_x$ reduces when the sample length increases. The reason for this can be the increasing value of $\partial T / \partial \varepsilon_r$ in the denominator in (16) for an increase in L.

4. MEASUREMENT RESULTS

A general purpose waveguide measurement set–up is used for validation of the proposed method [22, 29, 36]. Because, in our laboratory, we do not have a one-port network analyzer, we could not directly test the proposed method using a one-port network analyzer. Instead, we used
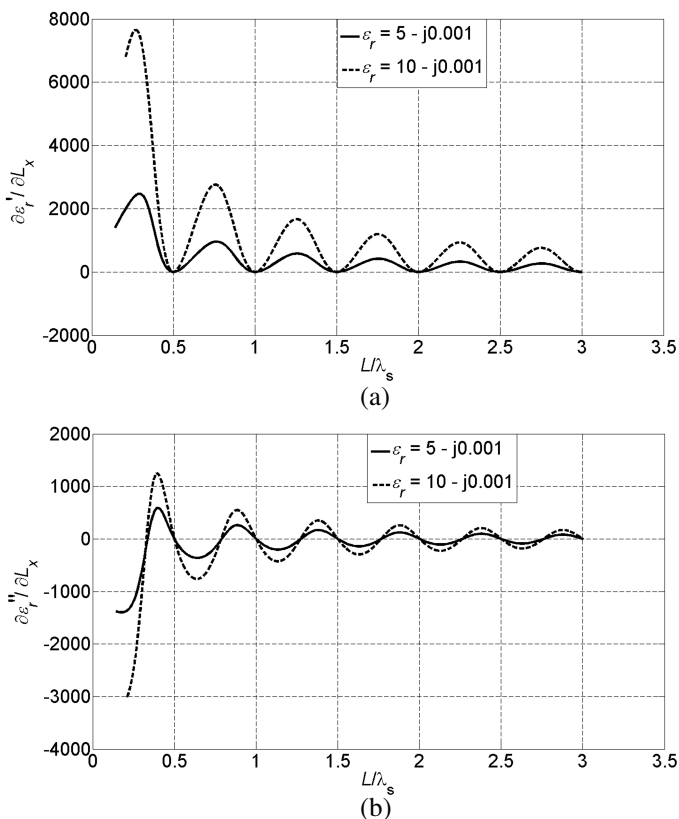
Figure 3. Uncertainty in L_1 versus L_x for different values of L_1.

forward reflection S-parameter measurements for two configurations in Fig. 2 by a two-port network analyzer (HP8720C VNA). It is connected as a source and measurement equipment. It has a 1 Hz frequency resolution (with option 001) and 8 ppm (parts per million) frequency accuracy. The waveguide used in measurements has a width of $22.86 \mp 5\%$ mm ($f_c \cong 6.555$ GHz). We employed two extra waveguide sections with lengths greater than $2\lambda_0$ at X-band between the calibration (reference) plane and coaxial-to-waveguide adapters to filter out any higher order modes [34].

The thru-reflect-line (TRL) calibration technique [38] is utilized before measurements. We used a waveguide short and the shortest waveguide spacer ($44.38 \mp 5\%$ mm) in our lab for reflect and line standards, respectively. The line has a $\pm 70°$ maximum offset from $90°$ between 9.7 GHz and 11.7 GHz. It should be noted that the TRL calibration technique is a 12-error term correction technique

applicable for two-port network analyzers. For one-port network analyzer calibration, the reader can use the calibration techniques proposed in [30]. Since the one-port calibration techniques in [30] and the TRL calibration technique [38] solely use line and reflect standards (open and match standards are very difficult to realize over a broadband), we expect that the accuracy of the one-port calibration techniques should be comparable or almost equal to that of the TRL technique. After calibration of the set–up, we paid special attention to preparing 2 mm long polyvinyl-chloride (PVC) and polytetrafluoro-ethylene (PTFE) samples with no scratches, nicks, or cracks [34]. We machined the samples so that they fit precisely into the line standard to reduce the air gaps between their external surfaces and the holder [34].

We positioned the samples into the line standard and then collected 801 data points evenly spaced between 9.7 GHz and 11.7 GHz. Next, we applied the time-domain gating over the main transmission properties of the samples to decrease post reflections, which may arise after the TRL calibration, and to obtain smoother S-parameter measurements. For example, Figs. 4 and 5 demonstrate the extracted ε_r of the prepared samples.

It is seen from Fig. 4 that there is a good agreement between the measured spectral data of PTFE samples by the proposed method and the measurements in [39] and the published data in the literature [40]. At ordinary room temperature, the ε_r of the PTFE sample given by Von Hippel is $2.08 - j0.00076$ at 10 GHz [40].
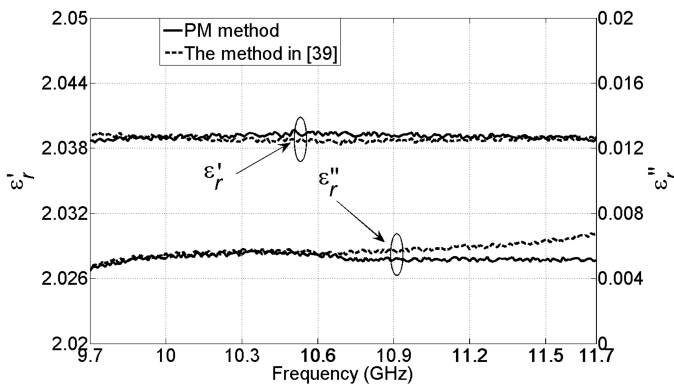

Figure 4. Measured relative complex permittivity of a 2 mm long PTFE sample by the proposed method (solid line) and the method (dashed line) in [39].
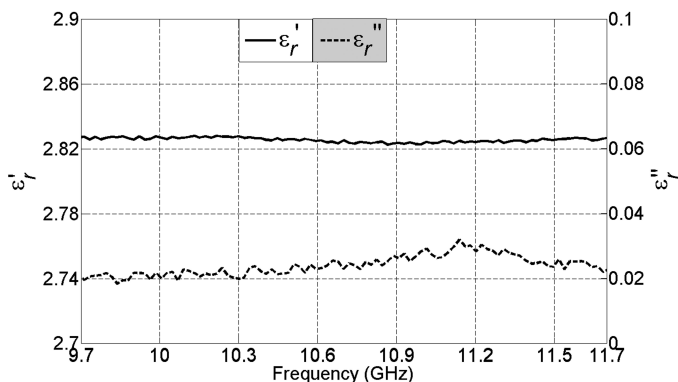
Figure 5. Measured relative complex permittivity of a 2 mm long PVC sample by the proposed method.

To validate and assess the accuracy of the proposed method, we also illustrate the measured ε_r of the PVC sample. At ordinary room temperature, the ε_r of PVC samples given by Von Hippel is $2.84 - j0.00156$ at 3 GHz [40]. It is seen from Fig. 5 that there is a good agreement between the measured ε_r and the data in the literature.

It is crucial to note that, to fully assess the advantages and drawbacks of the proposed method, we should have tested it for different samples with a wide range of permittivity values. Since, in our laboratory, we do not have solid samples with larger dielectric constant and loss tangent values expect that the tested PTFE and PVC samples, we could not perform such an accuracy analysis. However, we can deduce how the accuracy will change with different dielectric constant and loss tangent values. It is well known that the accuracy of phase measurements of reflection S-parameters drastically decrease at frequencies corresponding to minimum amplitudes of reflection S-parameters. An increase in dielectric constant for low-loss samples results in a decrease in the accuracy of permittivity measurements by the proposed method at the aforementioned specific frequencies. On the other hand, an increase in loss tangent for lossy samples can increase the accuracy of permittivity measurements by the proposed method compared to any other method which employs reflection and transmission measurements or only transmission measurements. The reason for this is the intolerable increased uncertainty in transmission S-parameters. It is also instructive to discuss the accuracy level of the proposed method for thicker samples. It is expected that the accuracy of the proposed method will increase with an increase in sample thickness, as shown in Figs. 3(a) and 3(b). Its reason is two-

folds. First, the measurement accuracy of the sample length by a micrometer increases by sample length. Second, the homogeneity upon which the theoretical foundations are constructed in Section 2 increases by sample length. However, we note that at some specific frequencies, we can measure unexpected ripples emerging from the effect of increased uncertainty in phase of reflection S-parameters at frequencies yielding minimum amplitudes of reflection S-parameters.

5. CONCLUSION

A microwave method has been proposed for complex permittivity determination of thin materials from reflection-only measurements. The method eliminates the problems arising from inaccurate knowledge of phase shift of measured reflection scattering parameter by utilizing two measurements of the sample holder at one frequency. The method is attractive in complex permittivity measurements by any one-port network analyzer, which is less expensive than their two-port counterparts. In addition, since the proposed method solely uses reflection measurements for permittivity inversion, it also removes the problems occurring from air gaps present between sample surfaces, which are in contact with the waveguide and waveguide inner walls. Furthermore, the method extracts the permittivity from point-by-point (or frequency-by-frequency) measurements. Therefore, it is applicable to band-limited applications. We have validated the proposed method by measurements of two thin low-loss samples with results obtained from the method in the literature and available data in the literature.

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