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PROCEDURE FOR ACCURATE AND STABLE CONSTITUTIVE PARAMETERS EXTRACTION OF MATERIALS AT MICROWAVE FREQUENCIES

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Abstract—A non-resonant microwave method has been proposed for accurate and stable constitutive parameter measurement of low-loss dispersive and non-dispersive isotropic materials. The method uses transmission-only measurements of two configurations: a) the sample inside a sample holder and b) the sample backed by a reference sample inside the same holder. It is not prone to undesired ripples in the extracted constitutive parameters arising from measured similar reflection properties. In addition, its accuracy is higher since it is not much affected by surface roughness and/or unevenness of the sample or the reference sample. It is based on frequency-by-frequency extraction and thus suitable for dispersive materials. However, it requires the selection of an appropriate reference sample. The method has been validated by measurements at X-band (8.2–12.4 GHz) of a low-loss sample located into a waveguide sample holder.

1. INTRODUCTION

Various microwave techniques have been proposed to characterize the electrical properties of materials with consideration of the frequency range, required measurement accuracy, sample size, state of the material (liquid, solid, powder and so forth), destructiveness and non-destructiveness, contacting and non-contacting, etc. [1–47].

Transmission-reflection non-resonant methods have extensively been employed for relative complex permittivity (ε_r) and/or relative complex permeability (μ_r) measurements of completely-loaded low-, medium-, and high-loss (solid, liquid, or granular) materials [4–47].

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These methods, when compared to resonant methods, are relatively simple to apply, give accurate information of ε_r and/or μ_r over a wide frequency range, require relatively less sample preparation, and allow frequency- and time-domain analyses [1].

Measured reflection and/or transmission scattering (S-) parameters can be utilized for broadband ε_r and/or μ_r extraction. However, measured transmission S-parameter (S_{21}) has several superior advantages over measured reflection S-parameter (S_{11}) as: a) it provides longitudinal averaging of variations in sample properties, which is particularly important for relatively high-loss heterogeneous materials such as moist coal and cement-based materials [20, 21]; b) it undergoes less deterioration from surface roughness at high frequencies [18]; c) it is more sensitive to the dielectric properties of high-loss samples [39]; and d) it offers a wide dynamic range for measurements [39].

In the literature, various methods based on solely S_{21} measurements have been proposed for ε_r and/or μ_r measurement of low-loss materials [35–43]. The method in [35] utilizes magnitude-only measurements at slightly different frequencies for unique ε_r extraction. However, it is not applicable to low-loss materials. For low-loss dielectric material electrical characterization, methods in [36–38] can be employed. While the method in [36] assumes that the sample is low-loss and thin, the method in [37] uses a second-order approximation to derive a one-variable objective function for fast ε_r measurements. We also derived a one-variable objective function for rapid and broadband ε_r extraction of thin or thick low-to-high-loss materials [38]. However, these methods [36–38] require a good initial guess for electrical properties of samples since complex exponential term in the expression of S_{21} yields multiple solutions [33, 38]. To avoid the need for inputting an initial estimate for ε_r, swept-frequency measurements of S_{21} of low-loss samples over a broadband can be directly utilized to obtain unique ε_r [39–42]. Whereas the proposed method in [39] may sometimes require an additional measurement of S_{21} for unique ε_r measurement in addition to two measurements of S_{21} at specific frequencies, the expressions of ε_r in [40] are complex in nature although it only requires two measurements of S_{21} for accurate ε_r determination. The methods in [39, 40] utilizes phase measurements of S_{21}. It is well-known that magnitude-only measurements are advantageous to complex (or phase) measurements in that the systems measuring amplitude-only information are relatively inexpensive, require less microwave components, and thus are desirable for industrial-based applications [18–22]. For industrial-based applications, the methods in [41, 42] can be utilized. The method in [41] exploits the oscillatory behavior of the magnitude of S_{21} measurements over a frequency band

and determines unique ε_r using measurements at frequencies resulting in extreme values of the magnitude of S_{21}. Although this technique is attractive and applicable to low-loss samples, it is not appropriate for thin samples with lower dielectric constants. To eliminate this drawback of this method, the method in [42] can be applied for unique ε_r determination of low-loss dielectric materials from measured S_{21} measurements. However, these methods [35–42] are only applicable for ε_r measurements of dielectric materials. In order to measure general electrical properties (ε_r and μ_r) of materials, the method in [43] can be employed. It resolves the problem of undesired ripples in the extracted constitutive parameters by using S_{21} measurements at slightly separated frequencies. Though, it is not suitable for highly dispersive materials and requires a good initial estimate for ε_r and μ_r for their accurate determination.

For constitutive parameter measurements of dispersive and other types of materials, the methods in [44–47] can be employed. While the method in [44] is suitable for constitutive parameter extraction of dispersive isotropic materials, those in [45, 46] are applicable to bianisotropic metamaterials. Nonetheless, these methods [44–46] may produce undesired ripples if similar reflection properties are measured. Besides, although the method in [47] extracts ε_r and μ_r of isotropic dispersive or non-dispersive materials non-iteratively and without any undesired ripples or inaccuracy peaks by utilizing preferably a medium-loss reference sample, it employs reflection-measurements into constitutive parameter extraction. As mentioned above, transmission measurements are advantageous to reflection measurements for electrical characterization of low-loss materials. In this research paper, we propose another microwave method for stable ε_r and μ_r inversion of low-loss isotropic materials from measured transmission-only measurements.

2. THE METHOD

2.1. Background

The measurement steps (configurations) for accurate and stable ε_r and μ_r determination of a low-loss sample by the proposed method are shown in Fig. 1. In this figure, while Fig. 1(a) corresponds to the measurement configuration of the sample itself inside a waveguide (or coaxial line) holder, Fig. 1(b) corresponds to the measurement configuration of the sample backed by a reference sample. The main reason of using a reference sample (preferably a medium-loss sample with known electrical and physical properties) will be discussed in Subsection 2.3.

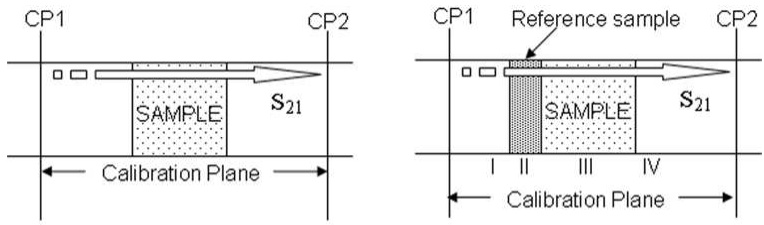

Figure 1. Measurement configurations of complex permittivity and complex permeability measurement of a low-loss sample completely filling a waveguide section between calibration planes (CP1 and CP2).

Between calibration planes, CP1 and CP2 in Fig. 1, S_{21} for two configurations can be expressed as [27, 44]

$$S_{21}^{(a)} = T\left(1-\Gamma^2\right)/\left(1-\Gamma^2T^2\right), \tag{1}$$

$$S_{21}^{(b)} = \frac{\left(1-\Gamma_{12}^2\right)\left(1-\Gamma_{23}^2\right)T_rT}{\left(1+\Gamma_{12}\Gamma_{23}T_r^2\right)\left(1+\Gamma_{12}\Gamma_{23}\right)-\left(\Gamma_{23}+\Gamma_{12}T_r^2\right)\left(\Gamma_{12}+\Gamma_{23}\right)T^2}, \tag{2}$$

where $S_{21}^{(a)}$ and $S_{21}^{(b)}$ are the transmission S-parameters for the configurations in Fig. 1; T and T_r are the propagation factors of the sample and the reference sample; Γ is the reflection coefficient when the sample is semi-infinite in length; Γ_{23} is the reflection coefficient at the interface II-III in Fig. 1(b) when both the sample and reference sample are infinite in length; Γ_{12} is the reflection coefficient at the interface I-II in Fig. 1(b) when both the air region and reference sample are infinite in length. The expressions of T, T_r, Γ, Γ_{23}, and Γ_{12} are given as

$$T = \exp(-\gamma L), \quad T_r = \exp(-\gamma_a L_a), \tag{3}$$

$$\Gamma = \frac{\mu_r\gamma_0-\gamma}{\mu_r\gamma_0+\gamma}, \quad \Gamma_{23} = \frac{\gamma_a\mu_r-\gamma\mu_a}{\gamma_a\mu_r+\gamma\mu_a}, \quad \Gamma_{12} = \frac{\gamma_0\mu_a-\gamma_a}{\gamma_0\mu_a+\gamma_a}, \tag{4}$$

$$\begin{gathered}\gamma = jk_0\sqrt{\varepsilon_r\mu_r-(f_c/f)^2}, \quad \gamma_a = jk_0\sqrt{\varepsilon_a\mu_a-(f_c/f)^2}, \\ \gamma_0 = jk_0\sqrt{1-(f_c/f)^2},\end{gathered} \tag{5}$$

In (3)–(5) γ, γ_a, and γ_0 are, respectively, propagation constants of the sample-, reference sample-, and air-filled sections; L and L_a are the lengths of the sample and the reference sample; k_0 is the free-space wave number (assumed as the wave number of light in vacuum); ε_r, μ_r, ε_a, and μ_a are, respectively, the relative complex permittivity and

relative complex permeability of the sample and the reference sample; and f and f_c are operating and cut-off frequencies.

2.2. Constitutive Parameter Extraction

In the analysis, it is assumed that the length between the calibration planes in Fig. 1 is known (transmission S-parameter measurements are not dependent on the position inside the calibration planes for a uniform and non-dispersive sample holder). It is seen from (1)–(5) that we have two complex S_{21} measurements and two unknown parameters (ε_r and μ_r). Therefore, it is possible to extract or invert ε_r and μ_r from (1)–(5) Inversion of ε_r and μ_r requires the determination of Γ (or Γ_{23}) or T. Because complex T has multiple solutions, it is wise to eliminate it from (1) and (2). Toward this end, we first express Γ_{23} in terms of Γ as

$$\Gamma_{23} = \frac{\gamma_a (1+\Gamma) - \gamma_0 (1-\Gamma)\mu_a}{\gamma_a (1+\Gamma) + \gamma_0 (1-\Gamma)\mu_a} = \frac{z_1 + z_2\Gamma}{z_2 + z_1\Gamma}, \tag{6}$$

where

$$z_1 = \gamma_a - \gamma_0\mu_a, \quad z_2 = \gamma_a + \gamma_0\mu_a. \tag{7}$$

Then, we obtain T from (1) and (2) as

$$T = \frac{S_{21}^{(a)} S_{21}^{(b)} \left[(\Gamma_{23}+\Gamma_{12}T_r^2)(\Gamma_{12}+\Gamma_{23}) - (1+\Gamma_{12}\Gamma_{23}T_r^2)(1+\Gamma_{12}\Gamma_{23})\Gamma^2 \right]}{\left[S_{21}^{(b)}(1-\Gamma^2)(\Gamma_{23}+\Gamma_{12}T_r^2)(\Gamma_{12}+\Gamma_{23}) - S_{21}^{(a)}\left(1-\Gamma_{12}^2\right)\left(1-\Gamma_{23}^2\right)T_r\Gamma^2 \right]}. \tag{8}$$

Here, we note that we could have obtained T directly from either (1) or (2). However, this approach would lead to two valid solutions for T, which intricate obtaining unique solution for T (using the constrain expression $|\Gamma| \leq 1$ can allow one to obtain unique T). Next, we derive a metric function for unique Γ determination. Toward this end, we substitute Γ_{23} in (6) and T in (8) into (1), and obtain the following metric function

$$\Omega_1\Gamma^6 + \Omega_2\Gamma^5 - \Omega_1\Gamma^4 + \Omega_3\Gamma^3 - \Omega_1\Gamma^2 + \Omega_2\Gamma + \Omega_1 = 0, \tag{9}$$

where

$$\Omega_1 = S_{21}^{(b)} z_1 \left(1 - T_r^2\right) \frac{\left(4\gamma_a\gamma_0\mu_a\right)^2}{z_2^3} \left[S_{21}^{(b)} \left(z_2^2 - z_1^2 T_r^2\right) - 4S_{21}^{(a)} (\gamma_a\gamma_0\mu_a) T_r \right], \tag{10}$$

$$\Omega_2 = \frac{\left(4\gamma_a\gamma_0\mu_a\right)^2}{z_2^4} \left[S_{21}^{(b)} \left(z_2^2 - z_1^2 T_r^2\right) - 4S_{21}^{(a)} \left(\gamma_a\gamma_0\mu_a\right) T_r \right]^2 + S_{21}^{(b)2} \left(1 - S_{21}^{(a)2}\right) \left(\frac{4\gamma_a\gamma_0\mu_a}{z_2} \right)^2 z_1^2 \left(1 - T_r^2\right)^2, \tag{11}$$

$$\Omega_3 = -2S_{21}^{(b)2}\left(1-S_{21}^{(a)2}\right)\left(\frac{4\gamma_a\gamma_0\mu_a}{z_2}\right)^2 z_1^2\left(1-T_r^2\right)^2 + 2\frac{\left(4\gamma_a\gamma_0\mu_a\right)^2}{z_2^4}\times \left\{2\left(4\gamma_a\gamma_0\mu_a\right)\left(z_2^2-z_1^2T_r^2\right)S_{21}^{(a)}S_{21}^{(b)}T_r - S_{21}^{(b)2}\left(z_2^2-z_1^2T_r^2\right)^2 - S_{21}^{(a)2}\left(4\gamma_a\gamma_0\mu_a\right)^2T_r^2\right\}. \quad (12)$$

The roots of Γ can be obtained using the "roots" function of MATLAB. After obtaining the roots, they can be checked whether they comply with the constrain $|\Gamma| \leq 1$ (for passive samples and if the sample with/without reference sample is not terminated by a short circuit [48]). We have validated the derived expressions in (9)–(12) by assuming some test values of ε_r, μ_r, ε_a, μ_a, L, L_a, f, and f_c, and observed that one solution for Γ can be assigned if we use the constrain $|\Gamma| \leq 1$.

Finally, we substitute the extracted unique Γ into (8) and determine the constitutive parameters of the sample using the following two steps:

$$\Lambda = -j\left(2\pi L\right)/\ln\left(T\right), \quad \lambda_{0g} = 1/\sqrt{1/\lambda_0^2 - 1/\lambda_c^2}, \quad (13)$$

$$\mu_r = \lambda_{0g}\left(1+\Gamma\right)/[\Lambda\left(1-\Gamma\right)], \quad \varepsilon_r = \lambda_0^2\left[1/\Lambda^2 + 1/\lambda_c^2\right]/\mu_r. \quad (14)$$

It should be noted that the order of extraction of constitutive parameters from (13) and (14) does not matter (first ε_r and then μ_r, or other way). What is important is whether extracted constitutive parameters produce undesired ripples (or inaccuracy peaks) [28]. We will discuss this point in following subsection.

2.3. Stability Analysis of the Extracted Constitutive Parameters

In this research paper, our goal is to measure accurate and stable constitutive parameters of low-loss materials from measured transmission properties. In this regard, we utilize a medium-loss reference sample. The basis why it is employed in the extraction process will become clearer if we let $T_r^2 \to 1$ (an approximation for a low-loss reference sample at some certain frequencies corresponding to $L_a = n(\lambda_a/2)$ where n is an integer number). Incorporating this case into (10)–(12), we find

$$\Omega_1 \to 0, \quad (15)$$

$$\Omega_2 \to 4\frac{\left(4\gamma_a\gamma_0\mu_a\right)^2}{z_2^4}\left(\gamma_a\gamma_0\mu_a\right)\left(S_{21}^{(b)} - S_{21}^{(a)}\right)^2 \to 0, \quad (16)$$

$$\Omega_3 \to -2\frac{(4\gamma_a\gamma_0\mu_a)^4}{z_2^4}\left(S_{21}^{(a)} - S_{21}^{(b)}\right)^2 \to 0. \tag{17}$$

Therefore, a meaningful and accurate ε_r and μ_r determination will not be possible under this case. We note that the second limiting case in (16) and (17) is obtained by assuming that $S_{21}^{(a)}$ will approach $S_{21}^{(b)}$ since when $T_r^2 \to 1$ and the reference sample is a very low-loss sample, we find from (2)

$$S_{21}^{(b)} \xrightarrow[T_r\to 1,\ \Gamma_{12}\to 0]{} \frac{\left(1-\Gamma_{23}^2\right)T}{1-\Gamma_{23}^2T^2} = S_{21}^{(a)}. \tag{18}$$

Using a lossy reference sample assures that T_r does not approach to one even at some specific frequencies resulting in $L_a = n(\lambda_a/2)$ situation. This is because, from (3),

$$T_r^2 = \exp(-2\gamma_a L_a) = \exp(-2\alpha_a L_a - j2\beta_a L_a) \xrightarrow[\beta_a L_a = n\pi]{} \exp(-2\alpha_a L_a) \neq 1, \tag{19}$$

where α_a and β_a are, respectively, the attenuation constant and phase constant (wavenumber or wave constant) inside the reference sample. We note that the above conclusion is completely in agreement with thickness-resonance phenomenon in transmission-only measurements [49]. We circumvent this phenomenon by using a lossy reference sample.

3. MEASUREMENTS

A general purpose X-band waveguide measurement set-up is used for validation of the proposed method ($f_c \cong 6.555$ GHz) [34]. The waveguide has a broader dimension of 22.86 mm and a narrower dimension of 10.16 mm. An HP8720C vector network analyzer is connected as a source and measurement equipment. The thru-reflect-line (TRL) calibration technique [50] is utilized before measurements. We used a waveguide short and the shortest waveguide spacer (44.38 mm) in our lab for reflect and line standards, respectively. The line has a $\pm 70^\circ$ maximum offset from 90° between 9.7 GHz and 11.7 GHz. In order to assess the accuracy of measurements, we measured the magnitude of reflection S-parameter for waveguide through measurements and noted that it ranges from -40 dB to -75 dB.

For verification of the proposed method, we machined a 8.06 mm long polytetrafluoro-ethylene (PTFE) sample and positioned it into a waveguide holder [44, 47]. Then, we measured forward and reverse S-parameters. Next, we positioned the reference sample next to

the PTFE sample (we poured a 5.04 mm long distilled water sample over the PTFE sample) and again measured forward and reverse S-parameters [44, 47]. The electrical parameters of distilled water are obtained by assuming $\mu_a = 1 - j0$ and using the Debye model

$$\varepsilon_a = \varepsilon_{a\infty} + (\varepsilon_{as} - \varepsilon_{a\infty})/(1 + j\omega\tau_a), \tag{20}$$

with the following parameters: $\varepsilon_{a\infty} = 5.2$, $\varepsilon_{as} = 78.5$, and $\tau_a = 8.3$ ps (the relaxation time) at ordinary room temperature. Finally, we applied our retrieval procedure. Figs. 2 and 3 demonstrate the measured constitutive parameters of the PTFE sample by the presented method. In order to assess its accuracy, we also superimpose into Figs. 2 and 3 the extracted ε_r and μ_r by similar methods in the literature [44, 47].

It is seen from Figs. 2 and 3 that the extracted constitutive parameters of the PTFE sample by the presented method and those in [44, 47] are in good agreement with each other and the reference data [51] (At 10 GHz, the ε_r of the PTFE sample given by von Hippel is $2.08 - j0.00076$. The nominal value for μ_r for the PTFE sample is approximately $\mu_r \cong 1 - j0$). It is known that the electrical properties of the PTFE sample do not much change with frequency over X-band. Since the methods in [44, 47] are based upon S_{11} measurements and since their theoretical formulation assumes flat front and end surfaces for the sample and the reference sample, the oscillatory behavior (and extracted values of $\mu_r' < 1$) seen in Figs. 2 and 3 for the extracted ε_r and μ_r by the methods in [44, 47] is attributed to the uneven surface of the reference sample as a consequence of meniscus formation on its top surface [47]. However, the presented method partially eliminates this oscillatory behavior and allows one to extract stable constitutive parameters throughout the frequency band.

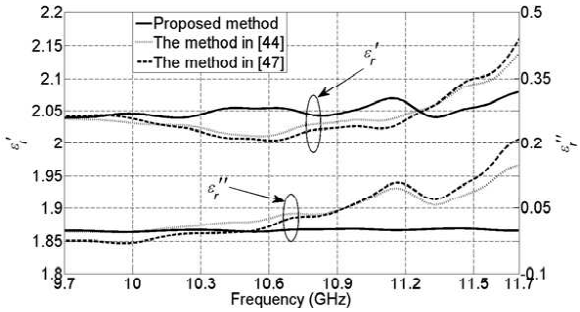

Figure 2. Measured ε_r of a PTFE sample using our method and those in [44, 47].

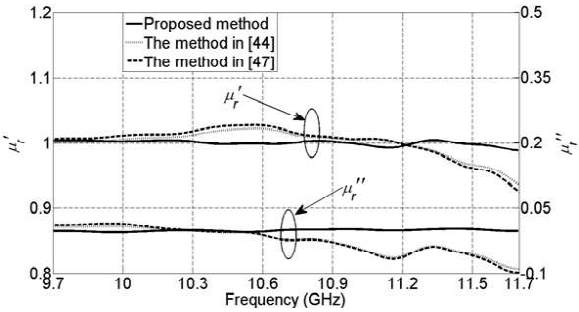

Figure 3. Measured μ_r of a PTFE sample using our method and those in [44, 47].

This is because the theoretical formulation in the presented method is based upon transmission S-parameter measurements (Fig. 1) and because transmission S-parameters are affected by surface roughness (or unevenness) less than reflection S-parameters although surface roughness (or unevenness) still plays an important role.

It is instructive to compare the proposed method with other methods in the literature. First, it necessitates less computation time for extracting the constitutive parameters as compared to that in [43]. Second, it should not produce undesired ripples (or inaccuracy peaks) at Fabry-Pérot frequencies since it utilizes a reference sample (preferably medium-loss) for increasing the reflection properties [24, 25]. Third, since its formulation is not based on asymmetrical reflection properties, it does not suffer from similar reflection S-parameter measurements at some discrete frequencies over the band [44–46]. In addition, this formulation is solely based on transmission properties and thus is suitable for electrical characterization of low-loss materials [47]. Finally, it is based on frequency-by-frequency extraction and thus is fitting for constitutive parameter measurement of dispersive materials [43]. However, the accuracy of the proposed method gets lower for constitutive parameter measurement of very low-loss samples if one uses a very lossy reference sample, since the information of the sample may be overwhelmed by that of the reference sample. Therefore, the reference sample should be selected so that it does not reduce transmission properties considerably. More information on this selection can be found in [47]. On the other hand, the presented formulation can only extract the constitutive parameters of isotropic materials. In the near future, we would like to extend the methodology for constitutive parameter measurement of anisotropic and bianisotropic materials [45, 46].

4. CONCLUSION

A non-resonant microwave method has been proposed for accurate and stable constitutive parameter inversion of low-loss dispersive and non-dispersive isotropic materials. It is based on transmission-only measurements and thus is less prone to surface irregularities (or roughness) and/or surface unevenness than reflection-only measurements. In addition, it does not experience any undesired ripples arising from similar reflection measurements. Finally, it is based on frequency-by-frequency extraction and thus is suitable for dispersive materials characterization.

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