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Non-Resonant Waveguide Technique for Measurement of Microwave Complex Permittivity of Ferroelectrics and Related Materials

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ABSTRACT

A waveguide method is developed to study the materials with relatively large dielectric constants at microwave range. Basically, the method is similar to the previous waveguide methods represented by short-circuit line and transmission/reflection measurement methods. However, the complex permittivity is not determined by the shift in resonance frequencies, but by numerical analysis of measured scattering parameters. In order to enhance microwave penetration into the specimen with relatively large permittivity, a dielectric plate with lower permittivity is employed for impedance matching. The influences of air gap between the specimen and waveguide wall are evaluated, and the corresponding errors are estimated. The propagation of higher order modes is also considered. Experimental results for several reference ceramics are presented.

***Key words :** Microwave complex permittivity, Rectangular waveguide, Non-resonant, Short-circuit line, Transmission/ Reflection*

1. Introduction

Microwave materials are of increasing interest for various fields of microwave engineering. Microwave dielectrics are widely investigated for numerous applications in basic microwave components like dielectric resonators, dielectric filters, phase shifters,[1] and multilayer circuit modules embedded in Low-Temperature Co-fired Ceramics (LTCC) systems.[2,3] Especially, high dielectric constant (ε) materials such as tunable ferroelectrics have been investigated extensively due to their potentials for both substantial miniaturization of microwave devices and integration with microelectronic circuits.[1,4] Ever-increasing microwave and millimeter-wave applications of dielectric materials often require precise determination of their complex permittivity.

Measuring the dielectric properties of ferroelectrics and related materials in microwave ranges is quite unconventional because they have large permittivity, which is often accompanied by large dielectric loss (tan δ). Microwave dielectric properties have been studied predominately by the resonance methods using coaxial transmission lines, which involve dielectric resonator inside a specimen holder.[5,6] However, such methods are quite limited to low-permittivity materials and to small samples that should be accurately machined into symmetrical geometries such as cylinders or disks.[7] High losses of ferroelectrics also limit their applications. Moreover, open microwave systems based on resonators or microstrip line resonance methods suffer from the approximations.[7,8] Additional complications can appear when dielectric properties of ferroelectric crystals are anisotropic and one-directional electric field might be required to determine the anisotropic microwave properties. Such requirements naturally can be satisfied by the rectangular waveguide excited by the TE_{10} mode.

The waveguide methods have been elaborated for over the last 50 years.[9-13] The contemporary waveguide techniques, which employ network analyzers to study frequency dependence of scattering parameters, are well developed especially for the materials with low dielectric constants and low losses.[14,15] The measurements were performed usually at one frequency, and the preference was to use reflections from the specimen whose thickness is a multiple of $\lambda_\varepsilon/4$, where λ_ε is the wavelength in studied dielectrics.[16] A major problem with high-ε dielectrics is a poor interaction of electromagnetic waves with the studied specimen. The waveguide section filled with ferroelectrics has very low impedance and large absorption compared with the section filled with air, which results in large reflection and practically no transmission. Due to the significant difference in the wave impedance, most of electromagnetic energy would reflect from the air-dielectric boundary, and can hardly penetrate the specimen. As such, the short-circuit line method exhibits lack of sensitivity being applied directly for ferroelectrics measurement.

In this work, the waveguide measurement technique is improved by employing a non-resonant broad-band measurement scheme.[17] In order to get scattering parameters of

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rectangular waveguide loaded with studied sample, a vector network analyzer is implemented. The method is applied to study large dielectric constant materials at microwave and millimeter wave range. To overcome the impedance matching problem, a $\lambda/4$-dielectric plate with low-permittivity and low-loss was inserted as an impedance transformer in contact with the studied sample. The influence of air gap between specimen and waveguide inner-walls was also investigated and a method is recommended to overcome the error associated with the potential air gap experimentally.[18,19] Effects of higher order mode propagation are also analyzed to access the uncertainty of the measurement method. The following sections present the original software elaborated for processing the measured scattering parameters by a nonlinear least-squares curve fitting technique.[20] Exactness and validity of experimental methods are analyzed. Measuring system is described in terms of circuit theory utilizing the transmission matrices approach.

2. Method Description

Basic measurement schemes are illustrated in Fig. 1. The frequency-dependent complex reflection coefficients, S_{11}^* of the sample is determined by the shorted waveguide of a 1-port system (Fig. 1(a)). Similarly, in a 2-port system (Fig. 1(b)), both the reflection coefficient, S_{11}^* and the transmission coefficients, S_{21}^* are measured in the whole frequency range of interest. A dielectric transformer might be inserted in the front and at the back of the specimen to enhance impedance matching at the boundaries.

The dielectric constant and loss of the specimen are determined by finding the values numerically fitted to the measured frequency-dependent scattering parameters. Electromagnetic wave in the sections filled with air, a dielectric transformer and the testing sample, respectively, can be represented by the normalized transmission matrix, $\boldsymbol{T}$, which is a function of the structure factors involved with the size of waveguide and inserted materials, and their electromagnetic responses. For the basic TE_{10} mode, the normalized transmission matrix of i-th medium is expressed as in Eq. (1).

$$\tilde{\boldsymbol{T}}_i = \begin{bmatrix} \frac{\gamma_i+\gamma_{i-1}}{2\sqrt{\gamma_{i-1}\gamma_i}}e^{-j\gamma_i d} & \frac{\gamma_i-\gamma_{i-1}}{2\sqrt{\gamma_{i-1}\gamma_i}}e^{-j\gamma_i d} \\ \frac{\gamma_i-\gamma_{i-1}}{2\sqrt{\gamma_{i-1}\gamma_i}}e^{j\gamma_i d} & \frac{\gamma_i+\gamma_{i-1}}{2\sqrt{\gamma_{i-1}\gamma_i}}e^{j\gamma_i d} \end{bmatrix} \quad (1)$$

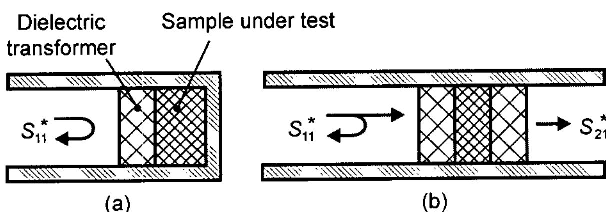


Fig. 1. Measurement schemes of (a) the short-circuit line and (b) the transmission/reflection measurement method.

Here, μ_i is the permeability of i-th medium and γ_i is the propagation constant in i-th medium. d is the length of i-th medium. The transmission matrix of whole network can be obtained by multiplying all the transmission matrices in sequence of the wave propagation from the first section.

$$[\boldsymbol{T}] = \tilde{\boldsymbol{T}}_n\tilde{\boldsymbol{T}}_{n-1}\cdots\tilde{\boldsymbol{T}}_1 \quad (2)$$

Then, the transmission matrix can be converted numerically into the scattering matrix whose parameters are directly measured by network analyzer. In case of non-magnetic medium the scattering parameters can be solved easily at every given frequency only if the sample dimensions and the reference plane positions are known. Nevertheless, this point-by-point technique is strongly affected by the experimental errors and the individual initiations of higher-order modes.[13,20,21] That is why a special data processing procedure has been employed in order to minimize the errors involved in the measurement. As such, a nonlinear least-squares curve fitting technique is used following the procedure represented by,

$$\min_{(\varepsilon',\varepsilon'')}\sum_k \sigma_k(S_k^{meas} - S(f_k,\varepsilon',\varepsilon''))^2 \quad (3)$$

where σ_k is a weight function, S_k^{meas} is the measured scattering parameter at the frequency, f_k while $S(f_k,\varepsilon',\varepsilon'')$ is the calculated value at the same frequency assuming that the test material has the dielectric parameters, ε' and ε''. Real and imaginary parts of scattering parameters are separated numerically and treated independently, which implies that the fitting process is applied to both real and imaginary parts. Weighting of the measured data is optional and is intended to improve the accuracy. However, proper choice of the weight function is important for correct data processing. Among various possible ways, there are weighted derivatives, and the modulus of reflection or transmission coefficients. These are emphasizing the influences of points near the minimum values of the reflection or transmission coefficients, which just have the highest sensitivities to the properties of testing specimen.

The choice between the short-circuit line method and the transmission/reflection method depends on which method has better sensitivity. The approach presented here has several advantages. The lengths of the specimen and the dielectric matching layer need not to be exactly equal to a quarter of wavelengths as did in the early works.[16] In addition, the measurement is carried out over a wide operational frequency range.

3. Uncertainty Analysis

The parameters of interest, namely, the dielectric constant and loss tangent are determined by the indirect measurements, thus it is important to analyze the corresponding uncertainties involved in the specimen preparation, measurement and data analysis schemes. Ideal filling

of measurement cell with the sample is rarely possible. In most cases, a rigid specimen is prepared independently and then, inserted into the metal holder for measurement. Perfect contact of the specimen with the holder is difficult to be achieved. Hence an air gap is unavoidable in the real measurement. The gap affects any measured values even in the case of low-dielectric materials. The previous estimation assumes that the gap of 2.5 – 7 μm is acceptable.[10,12] Several theoretical studies were conducted to analyze the air gap influences for high permittivity dielectrics with some experimental evidences.[18,19] This section presents an original analysis of the air gap effect as a function of broad air gap size and dielectric properties of dielectric materials.

First, the symmetrical gap between the sample and the narrow walls of the waveguide can be considered as shown in Fig. 2(a). Electromagnetic field can be solved by utilizing longitudinal wave representations. Boundary conditions in a form of equality of tangential components of electromagnetic field should be applied at the metal walls and on the air-dielectric boundary. Assuming symmetrical location of the specimen inside of the waveguide, a magnetic wall condition can be applied at the center of the waveguide in order to reduce the number of unknown variables. Finally it yields a complex nonlinear equation with respect to the complex propagation constant as the following equation.[21]

$$\beta_{xa}\cot\left(\beta_{xa}\frac{\Delta}{2}\right)-\beta_{x\varepsilon}\tan\left(\beta_{x\varepsilon}\frac{a-\Delta}{2}\right)=0 \tag{4}$$

Here, Δ is the total gap and a is the width of broad wall of the waveguide. $\beta_{xa}=\sqrt{k^2-\gamma^2}$ represents the transverse wave number in the air filled area and $\beta_{x\varepsilon}=\sqrt{\varepsilon k^2-\gamma^2}$ corresponds to the transverse wave number in the dielectric specimen where γ is the complex propagation constant. Separating real and imaginary parts of Eq. (4) results in two real equations with respect to real and imaginary parts of the propagation constant. They can be solved numerically, and the solutions for the propagation constant are less than those concerned with the totally filled waveguide. It represents so-called the effective dielectric constant, ε_{eff} and the effective loss, tan δ_{eff}. The simulation results of the side gap effect at 10 GHz are shown in Fig. 3. One can notice that the higher the permittivity is, the less the gap effect is. It is obvious that several-tens-of-micrometer air gap that corresponds to common accuracy of sample preparation practically makes no influence on the final result. The same interpretation is possible for the effective dielectric loss.

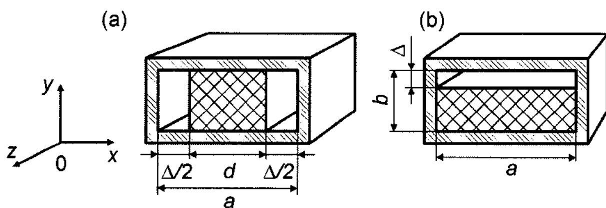


Fig. 2. Partially filled waveguide cross sections with the studied material to create and analyze the effects of air gap.

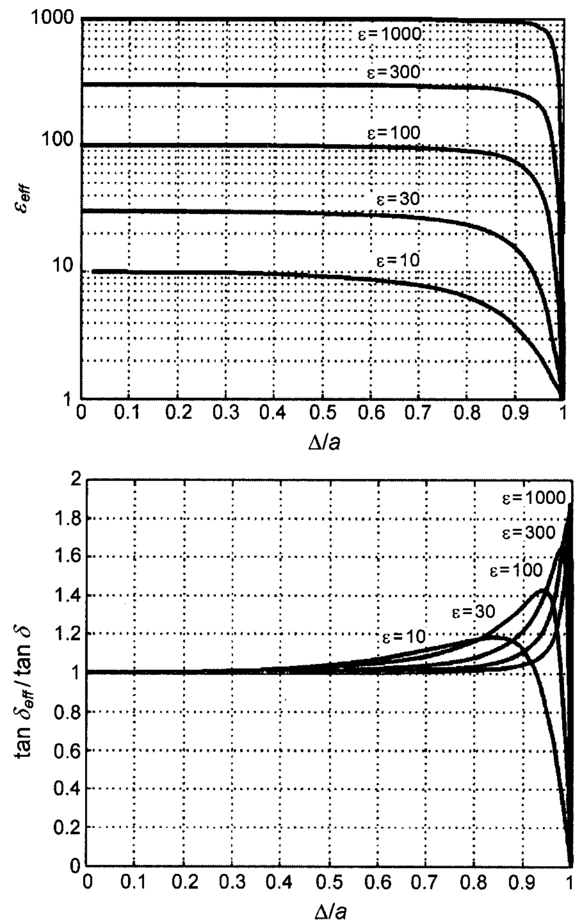


Fig. 3. Effect of the air gap between the specimen and the narrow walls of the waveguide.

That means the air gap created at the side wall of the waveguide is not critical to the accuracy of the test method if the permittivity of the sample is sufficiently large.

The next is the air gap between the sample and the broad wall of the waveguide as shown in Fig. 2(b). In the similar way, the electromagnetic field problem on the air-dielectric boundary can be reduced to the complex nonlinear equation,[21]

$$\beta_{ya}\tan(\beta_{ya}\Delta)+\beta_{y\varepsilon}\tan(\beta_{y\varepsilon}(b-\Delta))=0 \tag{5}$$

Here, Δ is the gap and b is the height of narrow wall of the waveguide. $\beta_{ya}=\sqrt{k^2-\gamma^2-\beta_x^2}$ represents the transverse wave number in the air-filled area while $\beta_{y\varepsilon}=\sqrt{\varepsilon k^2-\gamma^2-\beta_x^2}$ is the transverse wave number in the specimen where γ is the propagation constant, and $\beta_x=\pi/a$ is the transverse wave number for the basic mode. The simulation results at 10 GHz are shown in Fig. 4. Contrary to the previous case, the effect of air gap becomes significant as the permittivity of the sample increases. Furthermore, the effective dielectric properties are influenced noticeably by the air gap. The numerous experiments also indicated that such an air gap causes a significant error in the final result. Such effects of the air gap can be further explained with the analysis of higher order modes propagation conditions.

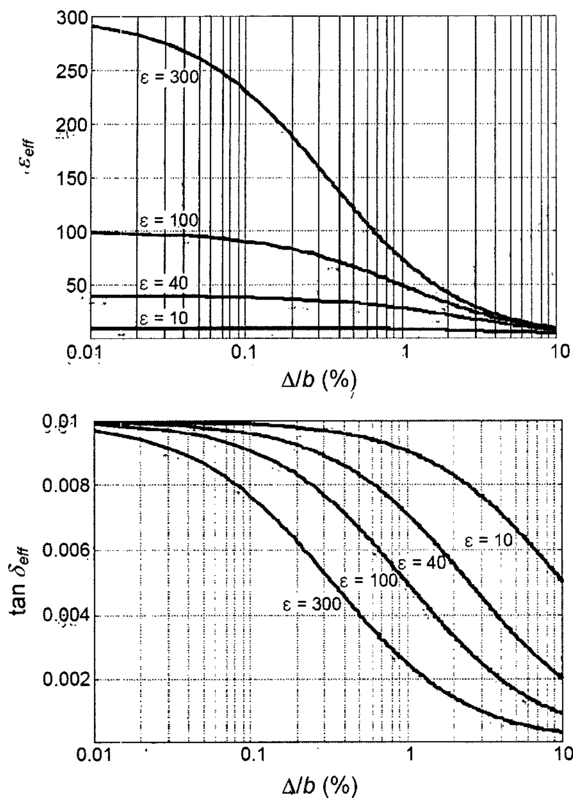


Fig. 4. Effect of the air gap between the specimen and the broad walls of the waveguide.

The discontinuities due to the presence of dielectrics in the waveguide will introduce higher-order modes that cause electromagnetic field reconfiguration. This process consumes some of traveling energy, which can be observed in network analyzer as a sudden dip of $|S_{11}|$ or $|S_{21}|$. If the waveguide system has higher-order modes, the theoretical model introduced here might be broken down. If the phase information is used, change in the propagation characteristics can bear an error in dielectric constant calculation. Particularly, coupling of energy with higher-order modes can be mistakenly assigned to the loss of the sample. In general, as the gap and the wave number increase, the deviation from the basic mode also increases. Consequently, the difference can lead mistakenly to lowering the calculated permittivity. Effective loss has more complicated behavior but generally increases with decreasing air gap.

In summary, the gap between sample and narrow walls of the waveguide is not critical up to a reasonable extent of gap size. On the other hand, introduction of a small air gap with the broad waveguide wall influences significantly on the measured values of dielectric constant and loss. It is thus important to prevent air gap between the sample and broad walls of the wave guide. One way of avoiding air gap is to cover the sides of the specimen contacting the broad waveguide walls with highly conductive metal. As shown in the following section, reliable results could be achieved experimentally as the metal paste was applied on the side walls of sample and touched the waveguide walls intimately.

4. Experimental Procedures and Results

All powders for the bulk samples were prepared and sintered by the general mixed-oxide method based on solid state reactions. Rectangular samples were machined and polished to fit in the X-band waveguide (WR-90 for 8.2 – 12.4 GHz, 22.86 × 10.16 mm). In order to minimize the air gap, the sides facing the waveguide walls were pasted with highly conductive silver (FERRO 3350), which was fired between 410 to 600°C for 10 min. In case of the short-circuit line method, the back side of the sample was also covered with silver paste for better reflection. The specimens for the reflection method look like being embedded in a silver cage, which is equivalent to the situation that the sample has its own waveguide with a short-circuit wall on its back. Whereas, those for the transmission/reflection (transmission) method had silver paste covered only in the side walls. In addition, teflon ($\varepsilon = 2.1$ and tan $\delta = 3.8 \times 10^{-4}$ at 10 GHz) was used as an impedance transformer,[22] which was also machined precisely to achieve a perfect contact to the waveguide wall and the sample. Minute air gaps might be introduced between the transformers and the samples because they were prepared separately, and then inserted into the waveguide. In order to minimize the air gap effects, several samples and transformers were prepared, and scattering parameters were measured repeatedly for each sample several times at the X-band.

Fig. 5 shows the results for rutile (TiO_2) ceramics as an example. Sensitivity of the method is illustrated in the phase characteristics of S_{11}. The optimum value of dielectric constant is $\varepsilon = 96$ following the solid line, and numerical simulations for the cases of $\varepsilon = 94$ and $\varepsilon = 98$ are also shown with the broken lines. It demonstrates that accuracy of the method is with ±2%. The dielectric loss for the rutile ceram-

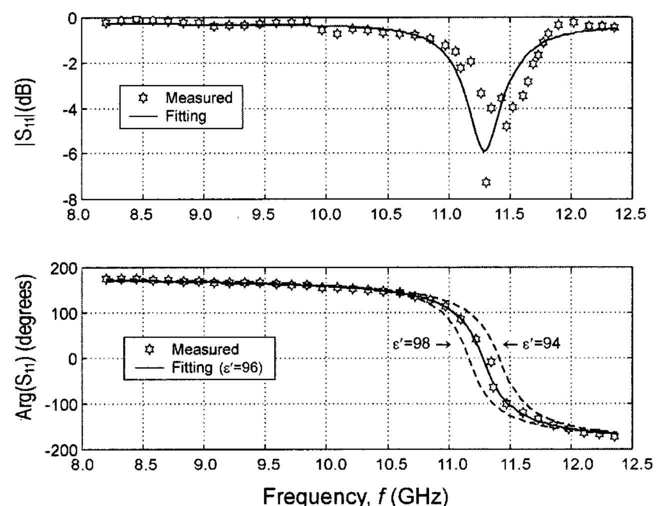


Fig. 5. Measured scattering parameters and fitted results for 2.03 mm-long TiO_2 through the short-circuit line (reflection) method.

Table 1. Measured Dielectric Constant and Loss of $(Mg,Ca)TiO_3$, TiO_2, $SrTiO_3$, and $BaTiO_3$ by the Short-Circuit Line (Reflection) Method and Transmission/Reflection (Transmission) Method, Compared with the Values Reported at around 10 GHz[23,24]

Material	Reflection method		Transmission method		Reference	
	ε	$\tan\delta$	ε	$\tan\delta$	ε	$\tan\delta$
$(Mg,Ca)TiO_3$	20.2	1.5×10^{-3}	-	-	20	2×10^{-4}
TiO_2	96	0.01	95	0.01	100	3×10^{-4}
$SrTiO_3$	290	0.02	270	0.017	320	0.01
$BaTiO_3$	590	0.3	-	-	600	0.22

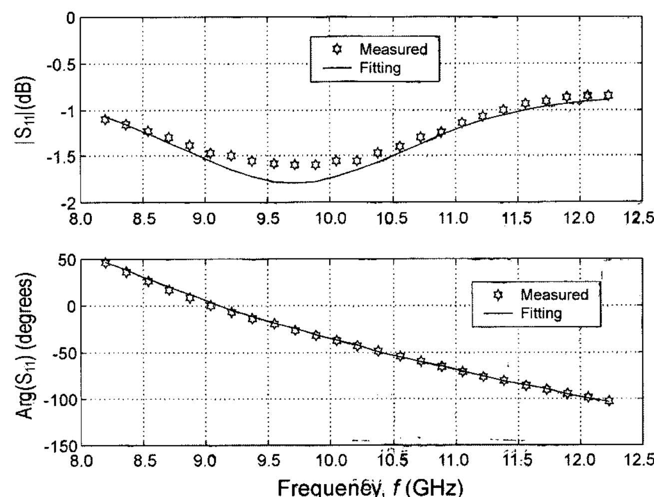
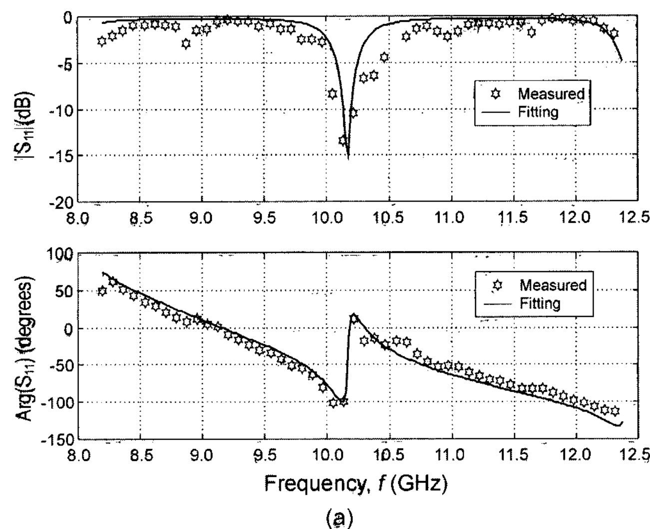


Fig. 6. Measured scattering parameters and fitted results for 1.51 mm thick $BaTiO_3$ with 6.56 mm thick Teflon transformer using the short-circuit line method

ics is found to be about 0.01. Table 1 summarizes the results obtained for four different sample materials with varying dielectric parameters by the short-circuit line method and by the transmission/reflection method, and compared with the values reported at around 10 GHz.[23,24]

As mentioned previously, the waveguide method is not adequate to be applied to very low-loss materials. The reflection investigation is less sensitive due to the influence of short-circuit wall adjacent to the sample. The transmission method might be able to profile the loss factors of 10^{-3} – 10^{-2}. For lower loss materials, the resonant methods are generally more reliable.[5] However, the loss factors of high-permittivity materials usually exceed 10^{-3} at and above the frequency of 10 GHz,[4] which makes the waveguide method quite sufficient for measuring dielectric parameters. Practically there exists no limitation on high-loss material investigation. If the product of the permittivity and loss is larger, only the thinner sample has to be prepared. Fig. 6 shows the results for $BaTiO_3$ ceramics obtained by the reflection method.

Comparison of the reflection and transmission methods is shown for $SrTiO_3$ ceramics in Fig. 7. In both cases the measured data and the fitting results are in a fairly good agreement. There exists little discrepancy between the measured and the fitted values. The measured dielectric constant and loss of $SrTiO_3$ are consistent with known published data.



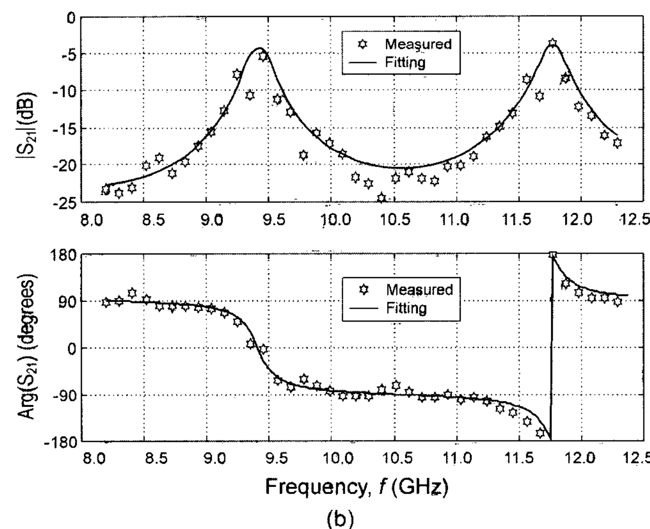


Fig. 7. Measured scattering parameters and fitted results for 3.89 mm thick $SrTiO_3$ with 6.56 mm thick Teflon transformer using (a) the short-circuit line (reflection) method and (b) transmission/reflection (transmission) method.

The difference in the measured values using two different methods is within 10%.

5. Conclusions

A non-resonant waveguide technique is proposed for

measuring microwave complex permittivity. The measured experimental results and numerical analysis show that the proposed method can be utilized safely for characterizing dielectric properties of the materials with relatively high dielectric permittivity and high loss. The proposed method adopts the advantages of the previous waveguide methods. However, the method is not involved with observing well-defined resonance peaks. Instead, the complex permittivity is obtained by a numerical analysis minimizing the total sum of squares of the difference between measured and calculated scattering parameters. Therefore, this method shows relatively high accuracy due to the multi-point measurement schemes. The procedures of sample and fixtures preparation are relatively simple. In order to enhance the reliability of the technique, one might introduce matching dielectric plates in contact with the sample to enhance the penetration of microwave into the specimen.

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