High precision slotted cavity measurement of a novel ceramic state polymer electrolyte

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Abstract. Thin film materials are already used in a variety of microwave and higher frequency applications such as electrically tunable microwave devices, integrated circuits like MMICs, radomes, and radar absorbing coating. The determination of the dielectric properties of these films is thus of significant importance. The measurement of complex dielectric permittivity of thin films is very difficult at microwave, millimeter, and THz frequencies because both the amplitude change and phase shift are not large enough to evaluate the real part of the dielectric permittivity. A specially designed transverse slotted cavity for X-band microwave measurement has been designed and constructed to employ with a vector network analyzer to evaluate the real part of dielectric permittivity of thin films accurately and conveniently. Commercially available polymer thin films are measured to validate the methods.

1. Introduction

Although there are various well documented methods for determining the dielectric properties of low-loss ultra-thin films, finding methods that are applicable and sensitive enough for use on ultra-thin materials is a challenge. Close-ended coaxial probe technique is developed based on measuring the reflection coefficient from a coaxial transmission line. References [1, 2] take advantage of microstrip transmission lines and resonators. Dielectric resonator methods are also implemented on measuring properties of thin films [3-5].

The newly developed slotted cavity technique described in this paper can measure very thin materials around 1 mil (25.4 μm) thickness over microwave frequencies. A slotted cavity measurement scheme is shown here that employs a transverse slot in the narrow wall through which the sample can be inserted and removed. This method is much simpler and the sample occupies the entire cross section of the cavity. The transverse slot is more sensitive than longitudinal slot because the sample is oriented parallel to the wavefront, which can provide a reduced transmission and higher reflection.

The measurement techniques described in this paper on thin films in microwave frequency range are based on waveguide theory. The in-waveguide measurement, cavity measurement, transverse slotted cavity measurement are all employing the rectangular waveguides with Agilent 8510C vector network analyzer (VNA) to record the scattering parameters, frequency shift and quality factor. From the scattering parameters, frequency shift and quality factor, the complex permittivity of the thin films are determined.

2. Measurement technique

Two ports Vector Network Analyzer is used to characterize two port networks. In this study, it was employed to characterize the thin film material under test, which was loaded in waveguides or transverse slotted cavity to form the two-port network. An accurate measurement involves
standardization and calibration of the system at the required frequencies and ambient conditions. Here, measurement was carried out from 2 to 40 GHz. The network analyzer includes a signal source, a receiver and a display unit to process the signals. The block diagram of network analyzer is shown in Figure 1.

![Network Analyzer Block Diagram](image.png)

**Figure 1.** Signal flow inside the vector network analyzer showing how the data is measured and recorded.

The source launches a RF signal at a single frequency towards the sample under test. The test set separates the signal produced by the source into an incident signal, sent to the sample under test, and a reference signal against which the transmitted and reflected signals are later compared. The test set routes the transmitted and reflected signals from the material under test to the receiver. The receiver is tuned to the frequency of the RF signal provided by the source. The source is then stepped to the next frequency and the measurement is repeated to display the reflection and transmission response as a function of frequency. The measured response produces the magnitude and phase for each scattering parameter at that frequency.

The following techniques can be used with the network analyzer for electromagnetic measurements: Free space measurement, Resonant Cavity Measurement, In-Waveguide Measurement, Open-ended waveguide measurement. In this dissertation, in-waveguide measurement method and transverse slotted cavity measurement are shown.

### 2.1. In-waveguide measurement

The Here, a transmission reflection (T/R) based waveguide technique was used to carry out the measurements. The T/R method is a category of non-resonant methods that are widely used for the measurements of electromagnetic properties of materials. In this method, the sample under test is inserted into a segment of transmission line, such as waveguide or coaxial line, which forms the two port network shown in Figure 2. The cables from the network analyzer are connected to across this network. The Vector Network Analyzer records the s-parameter values. Scattering equations are used to analyze the fields at the sample interfaces. These equations relate the s-parameters of the segment of transmission line filled with the sample under study to the permittivity and permeability of that sample. In T/R method, all the four s-parameters can be measured, so we have a record of more data than what we have in reflection measurements. The modified sparameters used in the measurements in this work are given below,

\[
\begin{align*}
\tilde{S}_{11} &= S_{11} e^{j(\alpha l - \beta_0 d)}; \tilde{S}_{21} = S_{21} e^{j((d-j)k_0 d - \beta_0 d)}
\end{align*}
\]

where, \(l\) is the quarter wavelength difference between thru and line (in air), \(d\) is the thickness of the sample inside the waveguide, \(k_0\) is the wavenumber of the sample and \(k_c\) is the cutoff wavenumber. These equations take into account the effect of using samples with thickness \((d)\) values that are smaller than the waveguide shim used in the experimental setup. [6] Return losses of less than -50 dB from the air inside the waveguide are easily achieved using these calibration techniques. This enables us to
neglect any unwanted reflections from the inner walls of the waveguide when analyzing the S-parameters. The reflection and transmission by the scattering parameters inside the waveguide, in which the transmission and reflection resemble the free space formulation, can now be presented as follows:

\[
\Gamma = K \pm \sqrt{K^2 - 1}; K = \frac{\bar{S}_{11} - \bar{S}_{21} + 1}{2\bar{S}_{11}}; T = \frac{\bar{S}_{11} + \bar{S}_{21} - \Gamma}{1 - \{\bar{S}_{11} + \bar{S}_{21}\}}
\] (2)

**Figure 2.** Setup of in-waveguide measurement method.

The transmission coefficient through the material may also be written as \( T = e^{-\mu d} = e^{-(\sigma + j\delta)d} \).

The propagation constant through the material inside the waveguides has been derived to be:

\[
\gamma_{TE_{01}} = \frac{\ln\left(\frac{1}{|T|}\right)}{d} + j\frac{2m - \phi_t}{d}
\]

The complex permeability and permittivity associated with the propagation constant are then:

\[
\mu = \frac{\eta \gamma_{TE_{01}}}{\gamma_{TM_{01}}} = -j\left(1 + \Gamma\right)\left(\frac{1}{2md}\right)\left\{\ln\left(\frac{1}{|T|}\right) + j(2m - \phi_t)\right\}\frac{1}{\lambda_0}\left(\frac{1}{2a}\right)^2
\]

\[
\varepsilon = \frac{\mu}{\eta} = \frac{\varepsilon_\infty}{\eta}\left(\frac{1}{\lambda_0}\right)^2 - \left(\frac{1}{2a}\right)^2
\]

(4)

In our waveguide measurement technique the propagating wave was assumed to be the TE10 mode. Permittivity is then calculated as follows:

\[
\varepsilon = \left(\frac{\varepsilon_\infty}{\eta}\right)\left(\frac{1}{\lambda_0}\right)^2 - \left(\frac{1}{2a}\right)^2
\]

(5)

It was also noticed that the permeability and permittivity of the loaded sample affect the cut-off frequency for the waveguide band. This was accounted for in the calculations by including the cut-off frequency for each band in the derivation of permeability and permittivity from the data for s-parameters. The divergence in data was eliminated by using the electrical delay function of the network analyzer. The experiments carried out in the lab suggest that these modifications are necessary and known materials were measured to confirm the accuracy of the measurement technique. The derived permeability and permittivity data is very reliable and not affected by the scattering voltage ratios of the vector network analyzer.

3. Slotted cavity measurement
The In this section, a special designed transverse slotted cavity is built in X-band. It was applied to determine the complex permittivity of thin film materials by employing perturbation theory. The slotted cavity technique is a combination of in-waveguide method and cavity method. The cavity method employs waveguide with a hole where the cylinder sample can be inserted in the waveguide. Each cavity has resonant frequencies depending on the dimension of the cavity. The inserted sample will slightly change the distribution of electromagnetic fields inside the cavity thus change the resonant frequency and quality factor.

However, a key difference in this transverse slotted cavity method compared to the traditional cavity method is that slots can be made in the waveguide wall transverse to wave propagation direction through which the sample can be fed into the waveguide \[7\]. This allows a very accurate measurement of the difference in scattering parameters with and without the sample in place which, in turn, allows for much greater measurement sensitivity. It is similar to a perturbation technique in that differences in scattering parameters are used in the calculations rather than the scattering parameters themselves.

Figure3 shows the WR-90 slotted X-band waveguide used for the measurement. The waveguide is 1” long with the sample placed at the center. The sample is fed through narrow slots in both side walls (rather than the longitudinal slot[7]) prior to the first measurement. It is held taut by pulling on both ends of the sample. This novel approach enabled the sample to stay in place without foam or Teflon supports. After the first measurement is taken, the sample is removed through one of the slots, and a second measurement is taken of the system without the sample.

The transverse slotted cavity comes from the rectangular waveguide resonator. This resonator is basically a section of rectangular waveguide which is enclosed on both ends by conducting irises to form an enclosed conducting box. We assume the same cross-sectional dimensions as the rectangular waveguide (a, b) and define the longitudinal length of the resonator as c shown in Figure4. Given the conducting walls on the ends of the waveguide, the resonator modes may be described by waveguide modes which are reflected back and forth within the resonator (+z and -z directions) to form standing waves.

Given a resonator made from a conductor such as copper or aluminum, the ohmic losses are very small and the quality factor is large (high Q, small bandwidth). Thus, resonators are used in applications such as oscillators, filters, and tuned amplifiers. Comparing the modes of the rectangular resonator with the propagating modes in the rectangular waveguide, we see that the waveguide modes exist over a wide band (the rectangular waveguide acts like a high-pass filter) while the rectangular resonator modes exist over a very narrow band (the rectangular resonator acts like a band-pass filter).

In the past, many researchers have reported the theoretical [8-9] and experimental [10-12] results of the cavity perturbation techniques. The measurements of permittivity and permeability of the dielectric materials are performed by inserting a small and appropriately shaped sample into a cavity from a...
pinhole or longitudinal slot and determining the properties of the sample from the resultant change in the resonant frequency and loaded quality factor of the cavity. The basic idea of the cavity perturbation is the change in the overall geometric configuration of the electromagnetic fields with the insertion of a small sample must be small. Based on this assumption, a detailed derivation of the perturbation equation for the frequency shift upon the insertion of a sample into a cavity was given by Harrington [13].

In practice, most cavity measurements are also subject to certain pitfalls. The thin material must be self-supporting or supported in the proper place within the cavity. Typically, this is done with some low permittivity material such as foam or Teflon, whose effects must usually be calibrated out. This introduces sources of error to the measurement if the cavity is not assembled in exactly the same way for both the sample-loaded measurement and the unloaded reference measurement (i.e., with and without the thin material of interest). Problems can arise if the sample holder shifts position or a fastening bolt has even slightly different tension on it. Even the process of handling the cavity may cause temperature variations which could affect the geometry enough to be significant to the measurement, which requires measuring frequency shifts well under 1%.

To solve this problem, a novel slotted cavity is employed that allows the sample to be inserted and removed without perturbing the measurement setup. The same waveguide section used in the waveguide method discussed above is used again here with the addition of two coupling irises. Each iris is 0.03" thick and 0.08" radius and serves to couple energy from WR-90 input waveguide into the dominant TE101 mode of the cavity. Two-port measurements were selected over one port because very good signal-to-noise ratio can be obtained in S21 by increasing the input source power. Figure 5 shows the cavity with irises.

![Slotted Cavity with Coupling Irises](image)

**Figure 5.** Slotted Cavity with Coupling Irises. Two irises are added to the slotted X-band waveguide.

From Equation of the cavity resonant frequency, we can observe that the change of dielectric permittivity and magnetic permeability will lead the resonant frequency changing.

$$f_{resonant} = \frac{1}{2\sqrt{\mu \varepsilon}} \left( \frac{m \pi}{a} \right)^2 + \left( \frac{n \pi}{b} \right)^2 + \left( \frac{p \pi}{c} \right)^2$$

(6)

In the unperturbed state, the empty cavity, By some mathematical manipulations, we obtain

$$\varepsilon' = 1 + \frac{f - f_0}{f_0} \varepsilon'' = 1 + \frac{1}{2} \left( \frac{1}{Q} - \frac{1}{Q_0} \right) \varepsilon$$

(7)

where the quality factor (Q) of a waveguide resonator is defined as

$$Q = \frac{f_{resonant}}{\Delta f_{3dB \_ bandwidth}}$$

(8)

The Agilent 8510C vector network analyzer is used to make the slotted cavity measurement. Figure 6 shows the special slotted cavity setup. The thin films can be placed inside the waveguide. TRL calibration was used in order to minimize the systematic errors in the measurement process.
advantage of a transverse slotted cavity for X band connected at the other port to measure the thin films was also utilized here. The transverse slotted waveguide method is a novel technique and a number of known samples such as Teflon, Mylar, and black polyester are measured for validation of this new technique.

The advantage of a transverse slotted cavity for X band connected at the other port to measure the films was also utilized here. The uncertainty in imaginary part was minimized by repeating measurements for more than 10 times. TRL calibration was used in order to minimize the systematic errors in the measurement process. The sample thickness was increased by adding multiple layers. The systematic error was significantly reduced with increasing number of layers. The final uncertainty of measured data is in ±5%.

4. Measurement Results
Real and imaginary permittivity from 8.2 GHz to 40 GHz are shown in Figure 7 and Figure 8.

![Figure 6. Setup for transverse slotted cavity measurement for the measurement at X–band. The cavity has a slot in the transverse direction.](image)

**Figure 6.** Setup for transverse slotted cavity measurement for the measurement at X–band. The cavity has a slot in the transverse direction.

**Figure 7.** Real part of relative permittivity of solid polymer electrolyte from 4 GHz to 40 GHz. The dielectric constant keeps reducing through the frequency range as the ionic relaxation weakens.

**Figure 8.** Imaginary part of relative permittivity of solid polymer electrolyte from 4 GHz to 40 GHz. The field direction changes faster than the ions fully diffuse through the electrolyte. Thus the imaginary part of dielectric permittivity decreases.
Figure 9. Real part of relative permittivity of solid polymer electrolyte from 4 GHz to 40 GHz. The dielectric constant keeps reducing through the frequency range as the ionic relaxation weakens.

Figure 10. Imaginary part of relative permittivity of solid polymer electrolyte from 4 GHz to 40 GHz. The field direction changes faster than the ions fully diffuse through the electrolyte. Thus the imaginary part of dielectric permittivity decreases.

Loss tangent spectra in low frequency from 50 Hz to 20 kHz are shown in Figure 9. Real conductivity spectra measured by in-waveguide method in 4 GHz to 40 GHz are shown in Figure 10. The Cole-Cole plot is shown in Figure 11.

Figure 11. Cole-Cole plot of the electrolyte in microwave frequencies. The x-axis is the real $\varepsilon'$, the y-axis $\varepsilon''$. Essentially it is a representation of the Nyquist plot at microwave frequencies. It is the higher frequency tail of a traditional semicircle Cole-Cole plot.

The comprehensive result is showed in TABLE 1.

**TABLE 1.** Dielectric Measurement Result

| Frequency (GHz) | $\varepsilon''$ | $\varepsilon'$ | Loss Tangent | Conductivity (S/m) |
|-----------------|----------------|---------------|---------------|-------------------|
| 9.019           | 0.55178        | 3.835256      | 0.1438708     | 0.276856998       |
| 10.006          | 0.53999        | 3.811258      | 0.1416830     | 0.300591531       |
| 11.014          | 0.52827        | 3.78703       | 0.1394958     | 0.323694431       |
| 12.001          | 0.51712        | 3.76358       | 0.1374018     | 0.345256059       |
| 13.016          | 0.50598        | 3.739747      | 0.1352989     | 0.3663907         |
| 14.024          | 0.49525        | 3.716363      | 0.1332627     | 0.386392639       |
| 15.004          | 0.48514        | 3.693899      | 0.1313342     | 0.404948834       |
| 16.012          | 0.47505        | 3.671072      | 0.1294047     | 0.42317381        |
| 17.02           | 0.4653         | 3.648528      | 0.1275315     | 0.4405802         |
| 18              | 0.45614        | 3.626881      | 0.1257658     | 0.45677094        |
| No.  | 19.02 | 20.04 | 21.0175 | 22.0375 | 23.015 | 24.035 | 25.0125 | 26.0325 | 27.04 | 28.0525 | 29.065 | 30.01 | 31.0225 | 32.035 | 33.0475 | 34.06 | 35.005 | 36.0175 | 37.03 | 38.0425 | 39.055 | 40       |
|------|-------|-------|----------|----------|--------|--------|----------|----------|-------|----------|--------|------|----------|--------|----------|--------|--------|----------|--------|--------|----------|--------|------|
|      | 0.44693 | 0.43806 | 0.42987  | 0.42166  | 0.41411 | 0.40657 | 0.39965  | 0.39276  | 0.38629 | 0.38012  | 0.37428 | 0.36912 | 0.36393  | 0.35906 | 0.35453  | 0.35033 | 0.34671 | 0.34316  | 0.33993 | 0.33704 | 0.33448  | 0.33239 |
|      | 3.604634 | 3.582676 | 3.561905  | 3.540515  | 3.520287 | 3.499463 | 3.479778  | 3.459521  | 3.439796 | 3.420258 | 3.401005 | 3.383292 | 3.364591  | 3.346174 | 3.328043  | 3.310197 | 3.293797 | 3.276503 | 3.259493 | 3.242768 | 3.226329 | 3.211243 |
|      | 0.1239875 | 0.1222715 | 0.120687  | 0.1190971 | 0.1176365 | 0.1161794 | 0.1148487  | 0.1135302  | 0.1122996 | 0.1111365 | 0.1100485 | 0.1091022 | 0.1081640  | 0.1073056 | 0.1065284  | 0.1058341 | 0.1052622 | 0.1047324 | 0.1042898 | 0.1039360 | 0.1036725 | 0.1035092 | 0.1035092 |
|      | 0.472911275 | 0.48838248 | 0.502635377 | 0.516963689 | 0.530226167 | 0.543631569 | 0.556115153  | 0.568817968  | 0.581096097 | 0.59322192 | 0.605190077 | 0.616267868 | 0.62809038 | 0.639920244  | 0.651813454 | 0.663826259  | 0.675195622 | 0.687598916 | 0.700286606 | 0.713314864 | 0.726739927 | 0.739677551 |

5. Conclusion
Real part and imaginary part of complex permittivity of this thin film samples were accurately determined in frequencies. The novel ceramic state polymer electrolyte is measured and analyzed and exhibits high conductivity as desired. The newly developed slotted cavity method, which allows the sample to be inserted and removed without perturbing the measurement setup, is thus established as useful methods to characterize complex permittivity of thin film materials. The slotted cavity method is very convenient for it is easy to insert and the thin film material does not need any external support. And the measurement precision under shift position in dielectric measurement is improved. The work on thin films composed of known materials validates the ability of precise determination of dielectric permittivity by such technique. In the future, new samples can be studied using these techniques to determine their material properties and hence suitable applications at this frequency range. An extensive repeated measurements and signal averaging will increase the signal to noise ratio and the Q-factor significantly to allow us the determination of the imaginary part of complex dielectric permittivity and the loss tangent of thin film materials at microwave frequencies.

6. Reference
[1] J. Baker-Jarvis, R. G. Geyer, J. H. Grosvenor Jr, M. D. Janezic, C. A. Jones, B. Riddle, C. M. Weil, and J. Krupka, "Dielectric characterization of low-loss materials a comparison of techniques," Dielectrics and Electrical Insulation, IEEE Transactions on, vol. 5, pp. 571-577, 1998.
[2] B. Chung, "A convenient method for complex permittivity measurement of thin materials at microwave frequencies," Journal of Physics D: Applied Physics, vol. 39, p. 1926, 2006.
[3] H. Jin, S. Dong, and D. Wang, "Measurement of dielectric constant of thin film materials at microwave frequencies," Journal of Electromagnetic Waves and Applications, 23, vol. 5, pp. 809-817, 2009.
[4] A. Vyas, V. Rana, D. Gadani, and A. Prajapati, "Cavity perturbation technique for complex permittivity measurement of dielectric materials at X-band microwave frequency," 2008, pp. 836-838.
[5] M. N. Afsar, "Dielectric measurements of millimeter-wave materials," Microwave Theory and Techniques, IEEE Transactions on, vol. 32, pp. 1598-1609, 1984.
A. Sharma, "Design of Broadband Microwave Absorbers for Application in Wideband Antennas," Master of Science, Electrical and Computer Engineering, TUFTS UNIVERSITY, 2011.

B. Chung, "A convenient method for complex permittivity measurement of thin materials at microwave frequencies," Journal of Physics D: Applied Physics, vol. 39, p. 1926, 2006.

R. Coccioli, G. Pelosi, and S. Selleri, "Characterization of dielectric materials with the finite-element method," Microwave Theory and Techniques, IEEE Transactions on, vol. 47, pp. 1106-1112, 1999.

Y. Xu and R. G. Bosisio, "Analysis of different coaxial discontinuities for microwave permittivity measurements," Instrumentation and Measurement, IEEE Transactions on, vol. 42, pp. 538-543, 1993.

B. Viswanathan, R. Raman, N. Raman, and V. Murthy, "Microwave power loss and XPS measurements on high TcNd---Ba---Cu---Oxide superconducting system," Solid state communications, vol. 66, pp. 409-411, 1988.

V. Murthy and R. Raman, "A method for the evaluation of microwave dielectric and magnetic parameters using rectangular cavity perturbation technique," Solid state communications, vol. 70, pp. 847-850, 1989.

B. Meng, J. Booske, and R. Cooper, "Extended Cavity Perturbation Technique to Determine the Complex permittivity of dielectric materials," Microwave Theory and Techniques, IEEE Transactions on, vol. 43, pp. 2633-2636, 1995.

R. F. Harrington, "Time-Harmonic Fields," New York: McGraw-Hili, 1961.