Dielectric Material Measurement of Thin Samples at Millimeter Wavelengths
Kenneth E. Dudeck and Leonard J. Buckley

Abstract—Complex permittivity and permeability measurements at millimeter wave frequencies of thin samples (0.12 to 1.0 mm) were obtained. The methodology and considerations for making automated waveguide material measurements of thin samples in the 26.5 to 40 GHz (R-Band) frequency range are described herein. Measurement results for various dielectric materials are included.

I. INTRODUCTION:
Numerous methods of obtaining the complex permittivity and permeability at microwave frequencies have been compiled since the 1940's [1]. Since then, extensive measured results of many dielectrics have been published [2], [3]. In recent years, automated network analyzers have been successfully utilized to obtain the S-parameters over the microwave bands, and the material properties are then subsequently calculated [4], [5]. Measurement of material properties in the millimeter bands has been accomplished primarily by either free-space methods [6], or dielectric waveguide cavity resonators [7].

Free-space techniques circumvent the problem of precision sample fit to the waveguide or cavity walls and are nondestructive to the sample. A major disadvantage of the free-space method is that it is not an enclosed measurement, and therefore a greater sample area needs to be fabricated to avoid diffraction effects around the sample edges. Some reduction of the required sample size for free-space microwave measurements has been successfully demonstrated by the incorporation of spot-focusing horn lens antennas [8], but is limited in bandwidth due to the focusing nature of the lenses.

The need to extend the microwave waveguide technique to the millimeter region became apparent when attempting to characterize new materials that are expensive and difficult to fabricate. The small waveguide cross-sectional area for R-band, approximately 0.25 cm², required only small portions of sample volume to fill completely the cross section of the waveguide. The well-established waveguide measurement technique was therefore a very effective way to characterize small quantities of materials. The technique used to carry out these measurements and the results of several samples are included in this paper.

II. EXPERIMENTAL
A. Materials
Four thin-film polymers were used in the study. Polymethylmethacrylate (Plexiglas, amorphous polymer), polyimide (Kapton), and polytetrafluoroethylene (Teflon) were used as received in thin-film samples (0.1-0.8 mm). These materials are low-loss DC insulators. The fourth sample was the electrically conductive polymer, polyaniline, that was synthesized by the standard chemical oxidation process as described by McDairmid et al. [9]. Polyaniline was studied in two forms, the insulating base, and the conductive salt that was treated with 0.1 molar (M) tosylic acid.

Fig. 1. Sample holder geometry.

B. Sample Measurement and Calculation of Intrinsic Material Properties
The samples were fitted into a quarter wavelength R-Band waveguide sample holder, and the S-parameters were obtained using a Hewlett-Packard 8510B Network Analyzer with the millimeter wave option, as specified by the manufacturer [10].

The calculation of the relative intrinsic material properties, defined as:

\[ \mu = \mu' - j\mu'' \] (complex permeability)
\[ \varepsilon = \varepsilon' - j\varepsilon'' \] (complex permittivity)

is accomplished by the implementation of an algorithm developed by Nicolson and Ross [4]. The Nicolson-Ross calculation assumes that the sample completely fills the entire sample holder, meaning that the sample thickness, S, equals the sample holder thickness, H, from Fig. 1. In general, H is greater than S, which represents unfilled space. However, if the sample holder is assumed to be lossless, the unfilled regions account for only phase delay, or more negative angles, in the measured S-parameters. The sample offset distance, D, from the reflection port accounts for a round-trip phase delay in S₂₁, while the total unfilled line length, H-S, causes a forward phase delay to the measured S₂₁. The phase delay contributes to a more negative (delayed in time) phase angle than would exist if the sample holder were shortened to the same length as the sample. The effect of the unfilled regions can be mathematically removed by adding (making less negative) the amount of phase delay caused by the unfilled regions to the measured phase. This operation, shown by (1) and (2), causes the S-parameters to be "rotated" counter-clockwise on the complex plane, and thus represents the measured phase of the sample in a holder of equal length:

\[ \arg(S_{11} \text{ rotated}) = \arg(S_{11}) + 720° \frac{D}{\lambda_x} \] (1)
\[ \arg(S_{21} \text{ rotated}) = \arg(S_{21}) + 360° \frac{(H-S)}{\lambda_x} \] (2)

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### Table I

**R-Band Waveguide Measurements of Relative Permeability and Permittivity for Various Materials**

<table>
<thead>
<tr>
<th>Freq. (GHz)</th>
<th>Plexiglas 0.1082 cm</th>
<th>Kapton 0.0132 cm</th>
<th>Teflon 0.0132 cm</th>
<th>Teflon 0.0803 cm</th>
<th>Polyaniline (Emeraldine Base) 0.0272 cm</th>
<th>Polyaniline (Emeraldine Salt) 0.0175 cm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \mu' - j \mu'^* )</td>
<td>( \epsilon' - j \epsilon'^* )</td>
<td>( \mu' - j \mu'^* )</td>
<td>( \epsilon' - j \epsilon'^* )</td>
<td>( \mu' - j \mu'^* )</td>
<td>( \epsilon' - j \epsilon'^* )</td>
</tr>
<tr>
<td>26.5</td>
<td>0.99 - j0.02</td>
<td>2.62 - j0.01</td>
<td>1.05 - j0.04</td>
<td>3.14 - j0.02</td>
<td>0.96 - j0.00</td>
<td>1.98 + j0.00</td>
</tr>
<tr>
<td>28.0</td>
<td>0.99 - j0.01</td>
<td>2.62 - j0.01</td>
<td>1.04 - j0.04</td>
<td>3.13 - j0.02</td>
<td>0.96 - j0.00</td>
<td>1.97 + j0.00</td>
</tr>
<tr>
<td>30.0</td>
<td>0.98 + j0.00</td>
<td>2.64 - j0.01</td>
<td>1.05 + j0.01</td>
<td>3.10 - j0.02</td>
<td>0.97 - j0.00</td>
<td>1.95 + j0.00</td>
</tr>
<tr>
<td>35.0</td>
<td>0.98 + j0.00</td>
<td>2.62 - j0.03</td>
<td>1.02 + j0.01</td>
<td>3.10 - j0.04</td>
<td>0.93 - j0.11</td>
<td>1.92 + j0.01</td>
</tr>
<tr>
<td>40.0</td>
<td>1.00 - j0.01</td>
<td>2.60 - j0.02</td>
<td>0.93 - j0.11</td>
<td>3.12 - j0.01</td>
<td>1.01 - j0.01</td>
<td>2.09 - j0.01</td>
</tr>
</tbody>
</table>

Error: \( \pm 0.04 \pm j0.02 \pm 0.04 \pm j0.03 \pm 0.29 \pm j0.03 \pm 0.22 \pm j0.02 \)

Publ.: \( 0.92 - j0.05 \) \( 2.45 - j0.05 \) \( 1.01 - j0.01 \) \( 2.09 - j0.01 \)

<table>
<thead>
<tr>
<th>Freq. (GHz)</th>
<th>Teflon 0.0803 cm</th>
<th>Polyaniline (Emeraldine Base) 0.0272 cm</th>
<th>Polyaniline (Emeraldine Salt) 0.0175 cm</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>( \mu' - j \mu'^* )</td>
<td>( \epsilon' - j \epsilon'^* )</td>
<td>( \mu' - j \mu'^* )</td>
</tr>
<tr>
<td>26.5</td>
<td>0.94 - j0.05</td>
<td>2.08 + j0.01</td>
<td>1.15 - j0.10</td>
</tr>
<tr>
<td>28.0</td>
<td>0.94 - j0.03</td>
<td>2.06 - j0.01</td>
<td>1.13 - j0.13</td>
</tr>
<tr>
<td>30.0</td>
<td>0.94 - j0.03</td>
<td>2.03 + j0.00</td>
<td>1.11 - j0.07</td>
</tr>
<tr>
<td>35.0</td>
<td>1.00 - j0.08</td>
<td>1.94 + j0.05</td>
<td>1.03 - j0.09</td>
</tr>
<tr>
<td>40.0</td>
<td>1.12 - j0.23</td>
<td>1.90 - j0.01</td>
<td>0.95 - j0.07</td>
</tr>
</tbody>
</table>

Error: \( \pm 0.26 \pm j0.03 \pm 0.14 \pm j0.02 \pm 0.03 \pm j0.01 \pm 0.08 \pm j0.01 \)

Publ.: \( 1.01 - j0.01 \) \( 2.09 - j0.01 \) \( 1.01 - j0.01 \) \( 2.09 - j0.01 \)

\(^1\) Free-space measurement at 56 GHz by Kadaba [6].

\(^2\) Manufacturer's data at 1 kHz [11].

\( \lambda_e \) is the wavelength in the unfilled waveguide and is computed in (3):

\[
\lambda_e = \frac{c}{F \left(1 - \left(F_{ce}/F\right)^2\right)^{1/2}} \quad (3)
\]

where \( F \) is the operating frequency in Hz, \( F_{ce} \) is the TE\(_{10}\) cutoff frequency, and \( c \) is the velocity of light in a vacuum. The remainder of the analysis assumes that all the angles have been correctly "rotated" prior to the implementation of the Nicolson-Ross algorithm.

Unfortunately, there is not a unique solution to the Nicolson-Ross algorithm due to a complex logarithm required in the calculation of \( \mu \) and \( \epsilon \). In general, the logarithm of a complex number \( Z \) is multivalued as shown in (4):

\[
\ln(Z) = \ln|Z| + j(\arg(Z) + 2\pi N) \quad (4)
\]

where \( N = 0, 1, 2, 3, \ldots \)

The correct root, \( N \), corresponds to the integer of the sample's electrical thickness in wavelengths, or \( S/\lambda_e(\mu, \epsilon) \), which is also dependent upon \( \mu \) and \( \epsilon \) of the sample. Generally, thin samples are less than a wavelength making the 0th root correct; however, samples with an electrical phase exceeding 360° require a selection of \( N \) greater than zero. The determination of the correct root, \( N \), can be obtained from comparison of the change in measured wavelength at two adjacent frequencies in the sample, \( \lambda_e(\mu, \epsilon)_m \), which is calculated from the change in \( S_{31} \) phase at the same two frequencies as shown in (5):

\[
\Delta 1/\lambda_e(\mu, \epsilon)_m = -\frac{1}{360\Delta \arg(S_{31})} \quad (5)
\]

The wavelength is obtained from the \( N \)th root of calculated values \( \mu \) and \( \epsilon \), \( \lambda_e(\mu, \epsilon)_N \), where

\[
\lambda_e(\mu, \epsilon)_N = \frac{c}{F \left[\epsilon \mu - (F_{ce}/F)^2\right]^{1/2}}. \quad (6)
\]
In order for (5) to be valid, the discrete frequency steps of the measured data must be small enough such that the change in $\arg (S_{21})$ is less than 360°. The calculation of $\mu$ and $\epsilon$ is correct for the root $N$ that satisfies:

$$\Delta 1/\lambda_2(\mu, \epsilon)_N - \Delta 1/\lambda_2(\mu, \epsilon)_m = 0. \quad (7)$$

### III. RESULTS AND DISCUSSION

#### A. Measurement Errors

The error in the calculated results of $\mu$ and $\epsilon$ is generated from the following sources: the measured mechanical distances (sample thickness, $S$, sample holder length, $H$, and sample position in the holder, $D_i$), the instrument repeatability, the curvature of the sample, and the contact to the waveguide walls. The lengths $H$ and $S$ can easily be measured to within 0.01 mm, while the sample position can be set within 0.1 mm certainty. The sample holder and sample position distances, however, can be more accurately determined electrically.

The sample holder length is obtained by measuring the sample holder empty and calculating the length from the phase of $S_{21}$. The sample position is obtained by comparing the phase of the forward $S_{22}$, reflection coefficients. If the sample is homogeneous and flat, which is verified by comparison of the magnitudes of $S_{11}$ and $S_{22}$, the difference in phase corresponds to the position of the sample inside the holder.

The samples were measured three times to determine the $S$-parameter repeatability, and an error analysis was done to determine the measurement error calculation of $\mu$ and $\epsilon$.

Although the error associated with the sample's imperfect contact to the walls of the waveguide was not addressed, results obtained here compare well with materials previously measured by other methods as mentioned below.

#### B. Materials' Measurements

The measured relative $\mu$ and $\epsilon$ values for various materials are shown in Table I. The samples were carefully cut to size and fitted into the waveguide. The calculation of the material properties was carried out as outlined in the text, including the determination of the sample holder and offset electrical lengths for improved accuracy.

Free-space measurements at 56 GHz of Plexiglas and Teflon [6] and manufacturer's low-frequency data [11] for Kapton are shown for comparison. Teflon was measured at two different sample thicknesses to verify that $\mu$ and $\epsilon$ are independent of thickness; however, the thicker sample values are more accurate because the percent error in thickness measurement is smaller.

Polyaniline was measured in two oxidation states. The emeraldine base form is a DC insulator, and the 0.1 M tosyl acid-treated material (emeraldine salt) is a semiconductor ($\sigma_{\text{em}} = 1 \text{ S/cm}$).

The error values shown in Table I were obtained using error analysis and represent the total uncertainty in $\mu$ and $\epsilon$ due to the sample length uncertainty, ±0.01 mm, plus the uncertainty due $S$-parameter repeatability. The error values in Table I represent the worst-case situation when both errors are maximum and both cause deviations in $\mu$ and $\epsilon$ in the same direction. Table II shows the "total error" that includes the additional error produced if the sample holder length and sample position were not determined electrically. The "corrected error" illustrates the improvement in uncertainty if the electrically determined distances are used, as was the case for the data in Table I.

### IV. CONCLUSION

The complex permittivity and permeability of various thin samples, including an electroactive polymer (polyaniline), have been successfully measured in waveguide, at the millimeter range of 26.5 to 40 GHz. The measurement error is shown to be reduced when the sample position and sample holder mechanical distances are determined electrically. The remaining error can be further reduced by fabricating thicker samples if possible.

The dielectric behavior of polyaniline was measured in two oxidation states and found to change significantly, by approximately a factor of four. A more detailed study on the control of the complex dielectric behavior of polyaniline merits further investigation.

### REFERENCES


