New Method for Determining Permittivity of Thin Polymer Sheets

R.D. Geryak, J.W. Schultz, Z. Borders

Compass Technology Group Alpharetta, GA, USA Ren.geryak@compasstech.com J.G. Maloney Maloney-Solutions Inc Marietta, GA, USA

J.G. Calzada, J.T. Welter Air Force Research Laboratory Wright-Patterson Air Force Base, OH, USA

Abstract— We present a new method for measuring thin, polymer sheets using a slotted rectangular coaxial transmission line (R-Coax). This method allows a sheet of material to be inserted into the R-Coax slot, greatly simplifying the measurement procedure over traditional waveguide methods. The permittivity inversion is performed with the aid of computational simulations of the R-Coax conducted across a range of expected dielectric properties. In particular, the slotted R-Coax device was optimized to enhance signal strength but has no simple analytical solutions for inversion. This new measurement technique is demonstrated on several thicknesses of commercial polyethylene terephthalate (PET) films, with a maximum thickness of 10 mils (0.254 mm). Due to the coaxial geometry, this technique does not have an intrinsic lower frequency cutoff and has an upper frequency cutoff near 3 GHz from over-modeing within the transmission line, though this frequency range could be extended by shrinking the fixture. However, the signal strength and calibration stability limit the useful range of permittivity measurement to 0.5-3 GHz for 10 mil thick specimens (and a range of ~1 GHz-3 GHz for 0.5 mil thick specimens). Repeatability for the real part of the permittivity ranged between 2-5% and loss tangents of ~0.006 were measured. Thus, this paper demonstrates the R-Coax measurement technique as a potential QA tool for microwave frequency electrical properties of thin polymer films.

I. INTRODUCTION

Polymeric and composite materials are often integrated into electronic components as insulating substrates. Such materials allow sensitive electronic parts to be protected from electrostatic discharge. In cases such as resonant circuits, antennas and radomes, the presence of the dielectric material also strongly affects device performance. For example, a resonant circuit will resonate at a shifted frequency depending on the electrical permittivity of the nearby polymer. Thus the dielectric properties of protective materials and substrates must be included in the device design and closely monitored during manufacture for quality assurance purposes.

While there are DC-Low frequency methods to measure the permittivity of polymers, the options for microwave frequency measurements are limited, particularly in the case of thin sheets with low loss. This makes it challenging to determine how a given batch of materials will impact the performance of a device. Of particular interest are thin dielectric materials with relatively low permittivity (i.e. $\varepsilon < 5$) and low loss tangents.

One way to monitor such materials is with free space methods, such as spot probes. However, it is challenging to build systems that operate below a few GHz, since the free-space wavelength becomes impractically large. Waveguide techniques interrogate specimens that are only about a half-wavelength across, however even they struggle with such measurements. Specifically, the thickness of thin sheets is extremely low relative to the wavelength, so the incident signal is barely perturbed by the material. In addition, waveguide measurement techniques are destructive and require significant sample preparation making them impractical to rapidly measure large quantities of material. However rapid monitoring of sheet materials in an in-line process is needed for manufacturing quality assurance.

The crux of this problem is ultimately one of insufficient signal-to-noise ratio due to the small electrical thickness of the materials. Resonant cavity methods [1] are able to overcome this limitation for select frequencies by effectively amplifying the input signal. Though resonant techniques often have strict constraints on specimen geometries and are sensitive to environmental factors that make them unreliable in a manufacturing situation. An alternative that allows for broadband measurements is through the use of a slotted coaxial transmission line. In this geometry, the sheet under test can be positioned such that length of the material is parallel to the transmission path of the device, greatly increasing the effective electrical length of the material interrogated by the technique.

In this paper, we evaluate the effectiveness of a slotted rectangular coaxial transmission line (R-Coax) in measuring the permittivity of thin polymer films. As a part of this process, computational electromagnetic simulations were performed both to enable the inversion process of the method and to evaluate the intrinsic sensitivity limits of the method. Ultimately the method was tested on several commercially obtained material samples and the practical limits of the method were established.

II. EXPERIMENTAL DETAILS

The present work examines the effectiveness of a slotted rectangular coaxial line or "R-Coax" system, custom designed for continuous measurement of sheet materials. The system can be seen in **Error! Reference source not found.**, and contains a thin slot through which the samples could be inserted. The slot goes through both the outer and inner conductors of the transmission line and is oriented so that it is parallel to the electric field orientation. The system shown here has a cross-sectional area of 6.5x12 cm (inside the outer conductor) with a slot length of 34 cm. It includes a calibration kit of an additional line section and a short to perform a thru-reflect-line (TRL) calibration. This calibration was performed prior to all measurements.

The fundamental mode is transverse electric and magnetic field propagation (TEM) with a cutoff frequency at 0 Hz. As with other coaxial systems, the width and height of the of the coaxial line leads to cutoff frequencies for other higher-frequency transmission modes that complicate the fixture response. For the device in **Error! Reference source not found.**, the upper cutoff frequency has been empirically determined to be above 3 GHz, above which the presence of these higher order transmission modes greatly distorts the device signals. Future efforts could increase this cutoff frequency to higher frequencies by shrinking the device as desired.



Figure 1. Photograph of R-Coax system.

The material of interest in this paper is polyethylene terephthalate (PET), a common electronic substrate material. Several thicknesses of sheets were obtained (0.5, 1, 2, and 10 mils) to test the device at multiple different sizes of materials and to determine accuracy limits for the method. The transmission or S21 scattering parameter signal was used for the inversion of permittivity. In addition to the primary TRL calibration, a response calibration of the measured S21 signal was made with the empty transmission line after every specimen measurement. This additional calibration reduced the errors from thermal drift of the cables and fixture. Each sheet was measured 5 times to determine the variance of the method from repeated measurements and to improve the inversion data via averaging. In addition, a moving window average was used to smooth the calibrated S21 data. Measurements were taken on a two-port vector network analyzer (a Cobalt 4220, Copper Mountain Technologies) using a 10 Hz intermediate frequency bandwidth, with average sweep time of ~30 s. All measurements were caried out at room temperature or approximately 20-25 °C.

Focused beam measurements of the same sheets were also taken to compare and validate the R-coax results. These measurements were performed in a similar manner to the R-coax measurements (i.e. ~30 second sweep time, 5 measurements), but the 0.5 mil specimen was excluded due to specimen mounting difficulties. All data was calibrated to air (clearsite), time gated to remove multipath signals, and inverted with an iterative solver using standard free-space measurement methods [2].

III. COMPUTATIONAL SIMULATION RESULTS

One major disadvantage on the slotted R-coax measurement method is that no simple analytical expression exists to describe the theoretical change in signal with different materials. While it would be possible to create a calibration series on materials to allow for an inversion, this would require an unreasonable number of samples to have high accuracy in the results. Instead, a database inversion method [3] can be used, in which the fixture is simulated in a computational electrodynamics code for a number of different cases, and the measured signal is compared to an interpolated lookup table at each frequency.

Simulations of the R-coax fixture were performed in a proprietary finite difference time domain (FDTD) solver (OpenTDA, Maloney-Solutions). All simulations were performed with 0.5 mm cubic Yee cells and a simulated TRL calibration to match the measured data. An electric-field cross-section cut from the simulation can be seen in **Error! Reference source not found.** The black areas in this image show conductive regions and the colors show the strength of the electric field, with blue being a lower intensity, green a medium intensity and red a higher intensity. The material is thus inserted through the slot with is oriented vertically in this image.

As this image shows, the electric field within the transmission line is concentrated between the inner and outer conductors, with very little of the field leaking through the slotted region of the outer conductor. This field profile leads to high sensitivity to any material placed in the slot and high isolation of the signal within the R-coax.



Figure 2. Simulated electric field plot of a cross-section of the r-coax slot located in the center of the device .

For the FDTD calculations, material specimens in the slot were simulated with a simple dielectric model of a single number for the real part (real part of ε , ε_r) and a single number for the microwave conductivity (σ), leading to a frequency dependent range of complex permittivities shown in equation 1.

$$\varepsilon = \varepsilon_r - j \, \frac{\sigma}{e_0 \, \omega} \tag{1}$$

where j is the imaginary unit, e_0 is the permittivity of free space, ω is the angular frequency, and other parameters defined as above. Computational electromagnetic simulations were run for a wide range of different permittivity and conductivity combinations to span the expected range of sheet properties.



Figure 3. Calculated S21 amplitude (top) and phase (bottom) for different values of real permittivity and conductivity.

In particular, the R-coax geometry was simulated for each combination of permittivity and conductivity, and the simulated geometry include the slotted region of the fixture as well as the transitions on either side. The transmitted complex signal (S21) signals were calibrated using the same TRL method of the experimental measurements. The resulting data were recorded in a lookup table for the database inversion. The simulated S21 data were smoothed with a moving average method to reduce the effects of ringing due to finite simulation times.

Figure 3. shows the calculated amplitude and phase of the S21 signal from these simulations for a range of real permittivities ('eps') and conductivities ('sigma'). Each pixel on these images corresponds to an individual simulation. while the images show the amplitude and phase at 1 GHz, the simulations also contain wide band data from 100 MHz to 3 GHz. One interesting feature (shown in **Error! Reference source not found.**) of the simulated r-coax data is that the amplitude of the S21 signal depends primarily on the simulated σ and the phase of the signal depends on a combination of the frequency and real(ε). The inversion of the materials properties from measured data uses interpolation of the complex S21 and ε , but this feature allows for easy visualization of the data in 2-dimensional plots.

Because of the highly orthogonal relationship between the transmission signal and the inverted permittivity, it is possible to gain an understanding of the sensitivity of the technique by looking at the simulated data over a narrow range of properties. As shown in **Error! Reference source not found.**, the effect of the real permittivity on the phase has a strong frequency dependent effect. The top plot shows phase versus frequency for a 0.5 mil thick film, while the middle plot shows the phase versus frequency for a much thicker 10-mil film. The bottom plot shows a phase sensitivity, computed as the slope of phase vs ε_r over the indicated range of ε_r values. As these data show, the sensitivity of the technique also increases substantially with increasing thickness with a roughly linear dependance on both frequency and thickness.

Similarly, the effect of the microwave conductivity on the signal amplitude is shown in Error! Reference source not found.. The top plot is for a 0.5-mil thick film, while the middle plot is the amplitude dependence for a 10-mil thick film. The bottom plot is the slope of amplitude (in dB) versus conductivity and an indication of the amplitude sensitivity of the method. While the conductivity of the material does not have a frequency dependent effect on the S21 amplitude, the conductivity and imaginary part of ε (ε_i) are intrinsically related by the frequency (see equation 1). For materials with low dispersion and minimal loss tangent, this implies that ε_i will have better sensitivity at higher frequencies as well. From Error! Reference source not found., it can be seen that the amplitude changes in S21 also increase roughly linearly with thickness. Taken together, these results imply that for thin, low permittivity materials, the overall sensitivity should increase nearly linearly with increasing frequency, thickness, and permittivity.



 $\begin{array}{ll} \mbox{Figure 4.} & \mbox{Calculated S21 phase at different values of ϵ_r and thicknesses (top/middle). Computed sensitivity (slope) of phase vs ϵ_r for different thicknesses \\ \end{array}$

Figure 5. Simulated S21 amplitude at different values of σ and thicknesses (top/middle). Computed sensitivity of phase vs σ for different thicknesses

IV. MEASUREMENT RESULTS

As described in section II, four different thicknesses of PET sheets were tested in the R-coax fixture. Multiple measurements were taken so that an averaged result could be obtained and an estimate of variance could also be determined. The average permittivity result for five different measurements of each specimen can be seen in **Error! Reference source not found.** The ε_r of all specimens was found to be between 3.0 and 3.4, which agrees with other literature reported values for PET.

One obvious outlier is the 1 mil PET film, which has a lower ε_r close to 3.1, whereas the other three thicknesses have values closer to 3.3 – a difference of approximately 6%. One potential explanation for this discrepancy is that the manufacturer's tolerances on the PET thickness is only +/- 10%, which would easily fit the observed difference. In contrast, the ε_i of the specimens shows much less consistency at the 0.5 and 1 mil thicknesses. Meanwhile, the 2 and 10 mil thickness materials converge onto an average ε_i of ~0.025, leading to a dissipation factor (loss tangent) of approximately 0.005.



Figure 6. Average (n=5) results for permittivity inversions of PET films at 0.5, 1, 2, and 10 mils.

As shown in Error! Reference source not found., the standard deviation of the inverted permittivity is inversely proportional to the thickness of the PET film. This is in accord with the sensitivity study in section III and likely follows from the increased sample interaction volume. The inversion of the ε_r was found to be very reliable, with a standard deviation of less than 10% for all specimens throughout most of the band. Conversely, the standard deviation for the ε_i is much larger and the 0.5 and 1 mil specimens have deviations above 100% of the mean. The 2-mil specimen has a deviation below 60% for large parts of the band, whereas the 10 mil specimen is close to 10-20% for most of the band. Based on these results, the r-coax inversion method can reliably invert ε_r for PET films down to 0.5 mil thickness. For ε_i measurements, the technique is reliable down to 10 mils for single measurements, and down to 2 mils if multiple (at least 3) measurements are averaged together.



Figure 7. Standard deviation (n=5) results for permittivity inversions of PET films at 0.5, 1, 2, and 10 mils..

As a point of comparison, the permittivity data obtained from the R-coax results was compared to data obtained from free-space focused beam measurements. While the focused beam data was collected in a higher frequency band (5-20 GHz), the low losses in the PET films implies a relatively nondispersive ε_r and the data should line up across the measured frequencies As shown in **Error! Reference source not found.**, the permittivity values obtained for both the real and imaginary components align well with the focused beam data. This result serves as an important validation, since most errors (excluding thickness measurement error) are likely to be uncorrelated between the two methods. Of note, the gap in ε_r between the 1 mil film and others is confirmed by focused beam measurements.



Figure 8. Average (n=5) results for permittivity inversions of PET films at 1, 2, and 10 mils with focused beam data added (data from 5-20 GHz).

One crucial factor in obtaining accurate data was performing a response calibration measurement shortly after measuring a specimen (within about 1 minute) due to drift in the data. This drift is likely due to thermal changes in the coaxial cables connecting the r-coax device to the VNA and could be mitigated by improving the insulation on the cables. Another source of variance was the loading of the samples, which due to their small thicknesses and low stiffness, were not positioned perfectly in the center of the slot. This issue likely exaggerates the poor performance of the thinner specimens, as they were more likely to bend and deform. This problem could be mitigated in future studies by maintaining constant tension on the films through a roller system, such as might be used in a manufacturing environment.

V. CONCLUSION

This paper presented a new method for obtaining low frequency permittivity measurements from thin dielectric materials. The technique relies on the use of a slotted rectangular coaxial transmission line and is a rapid, broadband, and nondestructive method. Based on simulation results, the sensitivity of the method (signal vs permittivity) should scale with both material thickness and frequency. The measurement results demonstrate that the device can obtain permittivity for PET films down to 0.5 mils (for real part only) or 2 mils (for both real and imaginary parts).

One of the key advantages to this method is the ease of use, since full sized sheets can be inserted into the slot with minimal modification. Coupled with the high reliability of the method over a broad range of frequencies, the method could be used for large-scale quality assurance of thin dielectric materials. Future development of the technique will focus on methods to reduce variance in the measurements (e.g. insulation and sample placement). In addition, changes to the device geometries (e.g. shrinking) could allow for broader frequency ranges and higher sensitivity for the device.

REFERENCES

- Q.R. Marksteiner et al, "Cavity resonator for dielectric measurements of high-ɛ, low loss materials, demonstrated with barium strontium zirconium titanate ceramics", Review of Scientific Instruments, 2017.
- [2] J.W. Schultz, Focused Beam Methods, Measuring Microwave Materials in Free Space, Create-Space Publishing, 2012
- [3] J.W. Schultz, "A New Dielectric Analyzer for Rapid Measurement of Microwave Substrates up to 6 GHz", AMTA Proceedings, 2018