Extended frequency range dielectric measurements of thin films

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A method is described to measure the dielectric constant and loss of thin films. It employs a sample holder based on precision 50 Ω air line and can be used from audio frequencies to 1 GHz with a single sample. It is shown to maximize the precision available from commercial instrumentation with minimal data correction. This is confirmed by regions in which the different instruments overlap their frequency coverage. [S0034-6748(00)05106-6]

I. INTRODUCTION

The dielectric properties of materials used in commercial electronics packaging are becoming increasingly important as considerations for their use. As device capabilities increase, it is also becoming more important to measure these properties over an increasingly broadband of frequencies. In addition, many of the newer materials are available only as films and are formed in place.

Older methods, such as American Society for the Testing of Materials D150, were designed with thicker samples in mind and are difficult to implement on thinner samples. They can require considerable sample handling, as in movable electrode schemes and become incompatible with automation.[1] Also, they may not be adaptable to materials that are thin layers on a substrate. Any given scheme may have a restricted range of frequencies so that many configurations may be needed to cover an extended frequency range.

Since these materials must be formed in place, the usual desirability for using preferably as few samples as possible becomes even greater than for bulk materials, as processing variations can add to the sample-to-sample variations normally encountered in many materials such as polymeric ones. If different samples are used to map out the loss over a range of frequencies, the interpretation can be made much more difficult than if sample variations are not present. These variations can be accentuated by different stages of aging, varying sample thickness, chemical composition, and other factors that may be actually more difficult to control than in actual package fabrication. In fact, the sample-to-sample variations can be larger than the dispersive changes in the dielectric constant due to loss.

The desirability for a single sample can easily conflict with the requirement for a broad range of frequencies. Since the dielectric constant for a low-loss sample does not change in frequency very strongly, neither does the sample's equivalent capacitance. As a result, the sample's equivalent parallel admittance becomes proportional to frequency and at low frequencies can become comparable to a residual parallel admittance, while at high frequencies it can become comparable to an equivalent residual series impedance. In addition, at high frequencies, the sample can be a significant fraction of the wavelength and the concept of a capacitor loses its meaning. In what follows, we will demonstrate a method that covers the range from 1 kHz to 1 GHz with improved accuracy for both dielectric constant and loss over simpler methods.

II. APPROACH

As long as a sample between two electrodes is thin compared to the propagation wavelength inside the sample, the sample can still be regarded as a capacitance between the two conductors.[2] Since the materials used in electronics packaging applications are usually available in thin-film form, the thin-film restriction is not much of a physical limitation, especially if the dielectric constant is not too high. Furthermore, as long as a carefully metallized sample is used, air-gap errors can be eliminated, so that extreme demands of flatness for purposes of contact are removed. This is an increasingly important consideration for samples in which the sample surface roughness becomes comparable to the total thickness.

Even if the sample capacitance and loss can be meaningfully defined, their values, usually in terms of an admittance, must be measurable. At low frequencies, the equivalent parallel admittance of a dielectric sample is such that a three-terminal configuration can be used and the contributions from the connecting leads eliminated. This configuration depends on three factors. One is that the series impedance of the leads can be neglected. Another is that, at the measuring terminals, all ground currents are not included in the measurement. The last is that the effective admittance at the end of the leads is the same as at the measuring terminals; that is, there are no measurable propagation effects.

None of these factors can be realized at high frequencies where the length can become long relative to the wavelength along the connecting leads. There will be some frequency above which propagation along a transmission line will become the best way to analyze the data and determine the properties of the material. If the thin sample is placed at the end of a precision, rigid coaxial line terminated with a short, then, within the limits of available instrumentation, the sample can become measurable over a wide-frequency range.

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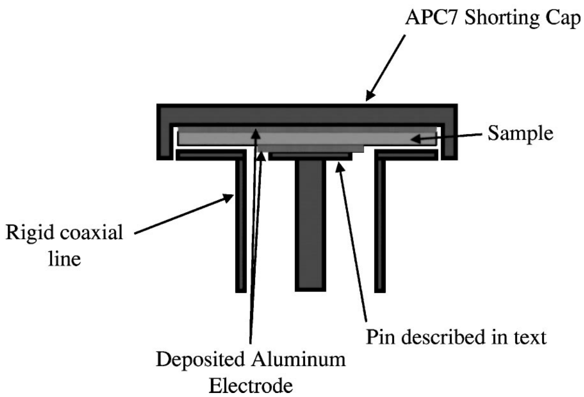

FIG. 1. Coaxial sample holder cross section.

At low frequencies, the sample is completely shielded from the outside. The capacitance between the inner conductor and outer conductor up to the sample is included in the measurement, but it is well defined and can be estimated well enough to be subtracted from the measurement without significantly degrading the accuracy available with automated bridges. The same is true for the equivalent series impedance.

As the frequency becomes high enough, these procedures become inadmissible, but the same capacitance can be viewed as an admittance at the end of a known transmission line.[3] Then, the measured admittance can be translated to the position of the sample by using a transmission line equation and a known propagation constant.

The true advantage of a rigid coaxial system is that there is sufficient overlap between low- and high-frequency methods for comparison. Also, the system is well enough defined so that the contribution of the coaxial line can be easily computed from a few constants at any frequency. This allows the sample measurement to be defined in terms of a quantity directly at the instrument terminals where the accuracy is the highest and corrected back to the actual sample by a computer program that does not rely on the usual limitations of the measuring instrumentation.

III. SAMPLE HOLDER

A sample holder, shown in Fig. 1, was fabricated from a short length of APC7 coaxial line that had a center conductor rigidly fixed to the outer conductor (HP 16091A coupling adapter).[4] The inner conductor was fitted with a small, flat-headed pin to act as a contact for the sample shown as the hatching in Fig. 1. For contact stability it was hard gold plated. The radius of the pin head is chosen to be at least as large as the inner conductor, but smaller than that of the outer conductor. It is also as thin as possible, 0.2 mm, consistent with mechanical rigidity. The top cap is a standard APC7 shorting cap.

The sample was metallized, shown in Fig. 1 as a lattice-like fill. The top surface was metallized over an area much larger than the inner diameter of the outer conductor and contacted the top shorting termination, which was also gold plated. The sample was punched out to just fit inside the terminating screw thread so that it can be accurately centered about the inner conductor. The inner surface was then metallized in a centering mask and jig so that an electrode of well-defined diameter that was at least as large as the pin was deposited. For the thin sample used, the effective area of the capacitor then becomes this electrode area. The sample could then be inserted in the holder so that no torque was applied to the sample and the pressure no more than necessary to contact the metallized electrodes. In our work, we used evaporated aluminum about 100 nm thick. Repeated closure showed that the measured parameters were repeatable to measurement resolution with multiple insertions of the samples.

The actual diameter of the inner electrode was limited by the maximum inner diameter of the outer conductor, the outer diameter of the inner conductor, and the desire to keep the admittance at the highest frequencies within the range set by the impedance meter. Also, for the highest frequencies used, where transmission-line corrections are used, the sample admittance should not be too far (less than a factor of 100, if possible) from the characteristic admittance of the coaxial line, typically, $0.02\,\Omega^{-1}$. This last requirement comes from the desire to minimize the propagation of errors associated with the uncertainty of the definition of the line length.

If the sample consists of an adherent film on a metal substrate, then the substrate replaces the top metallization. In either case, metallization or substrate, the major contact problem is between the reference short and the cap that holds it in place but that is easily verified by inspection.

IV. IMPEDANCE MEASUREMENTS

The impedance measurements were made with three impedance meters, a Hewlett Packard 4284A up to 1 MHz, a Hewlett Packard 4285A from 100 kHz to 30 MHz, and a Hewlett Packard 4191A from 20 MHz to 1 GHz. The manufacturer claims that the first two instruments are about 0.1% accurate in both capacitance and loss over most of the measurement range in frequency and with admittances within 10^{-4} to $10^{4}\,\Omega^{-1}$. The 4191A is claimed to be within 1% for frequencies above 10 MHz and for admittances centered about $0.02\,\Omega^{-1}$.

A. Four-terminal measurements to 30 MHz

The two instruments that cover the range to 30 MHz are presented by the manufacturer as four-terminal devices that allow full compensation of leads of at least 2 m in length. They were both calibrated against standard four-terminal resistors and capacitors, and did meet specifications at their terminals except for a slight increase in uncertainty near 30 MHz, apparently from internal residuals that could not be easily eliminated. This uncertainty, however, could be determined from the standard 100 Ω resistor and 10, 100, and 1000 pF capacitors.

To check their use as four-terminal devices at the end of the leads, as they were intended, a residual-free guarded sample holder was modified so that the 1 m high and low coaxial lead pairs were joined very close to the sample and the instruments calibrated at the sample position. A polyim-

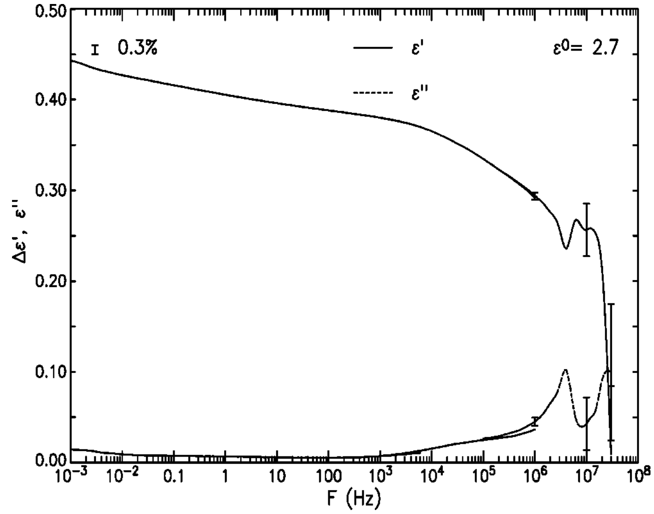

FIG. 2. The real and imaginary component of the dielectric constant of a polyimide. The upper curve is the real component and the lower the loss. The real component is offset by $\varepsilon^0 = 2.7$.

ide sample with 2-cm-diam electrodes was then measured up to 30 MHz, as shown in Fig. 2.

The lowest-frequency data were measured in a three-terminal mode by time-domain methods[5] and have an uncertainty of less than 0.1% in value with a loss resolution in tan δ of 1×10^{-6}. The data cover the range from 10^{-3} to 10^4 Hz. The Hewlett Packard 4284A overlaps the time-domain data within expected combined uncertainty at 10 kHz but there are clearly large errors for both bridges, greater than 0.3%, as the frequencies rise above 1 MHz. This uncertainty seems to be well beyond the apparent claimed accuracy of the bridges and is not too useful for most applications.

This apparent discrepancy actually is not the case. The error bars shown in Fig. 2 have been computed from the 4285A manual for the conditions of use, including the uncertainties from the lengths of cable. Within these larger limits, it can be seen that the data are quite reasonable, especially when one realizes that the wavelength in a cable at 30 MHz is about 7 m, any propagation uncertainties will strongly affect the measurement at the bridge terminals.

B. Coaxial measurements to 30 MHz

Both lower-frequency instruments can be fitted with an adaptor to the APC7 precision coaxial line. When the impedance meters were directly calibrated at their terminals, the adaptor appeared as a 1.7 pF capacitor that was of low loss. There was no obvious frequency dependence up to 30 MHz. The sample holder was then attached to the adapter and measured using a short and open termination without any sample present. These measurements allowed the sample holder plus the adaptor to be represented as the equivalent circuit shown in Fig. 3.

By direct measurement, using the impedance meters and the short and open terminations, the equivalent parallel capacitance C_p was found to be 3 pF while the equivalent series inductance L_s and resistance R_s were 0.3 nH and 0.01 Ω, respectively. The adequacy of the model was shown by the constancy of these values over the entire frequency range.

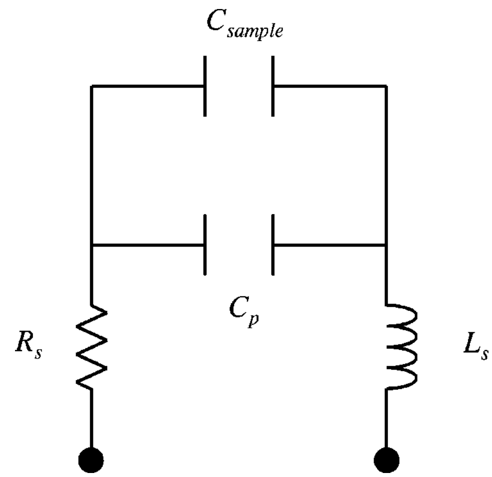

FIG. 3. Equivalent low-frequency circuit of the coaxial holder. C_p is the residual parallel capacitance between the inner and outer conductors, while R_s and L_s are the series resistance and inductance.

C. Coaxial measurements to 1 GHz

As the measurements are extended to 1 GHz using the 4191A impedance analyzer, the use of simple equivalent circuits fails. However, this instrument actually uses a transmission-line measurement that can be referenced to an arbitrary location using a short circuit for definition. Therefore, the holder can be represented by a capacitance at the end of a coaxial line, as shown in Fig. 4.

By setting the sample distance to zero, the capacitance of the sample C_{sample} is determined directly. This can be accomplished by several procedures. If the line length is short, a reference short can be used to define the origin to the plane at which the sample is inserted. If a longer line is used, then the reference could be set at the analyzer terminal and the measurement translated back to this reference plane using

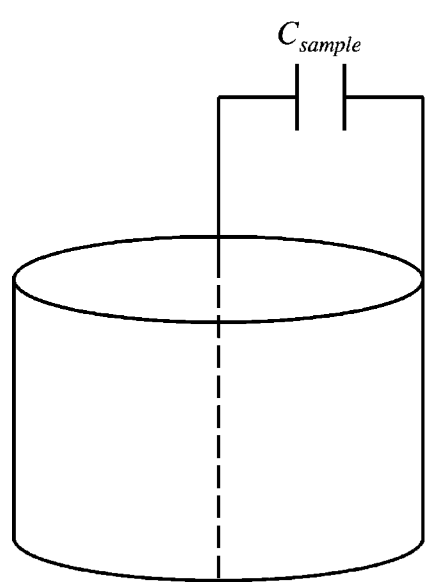

FIG. 4. Transmission line model of the holder. The sample is C_{sample} at the end of the line.

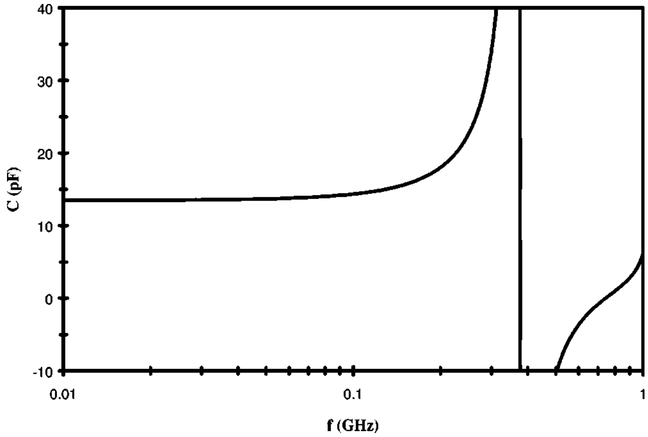

FIG. 5. Measured capacitance for an open circuit 20 cm from the measuring point. The vertical line is the point at which an open transforms into an equivalent short and about which there is a change in sign.

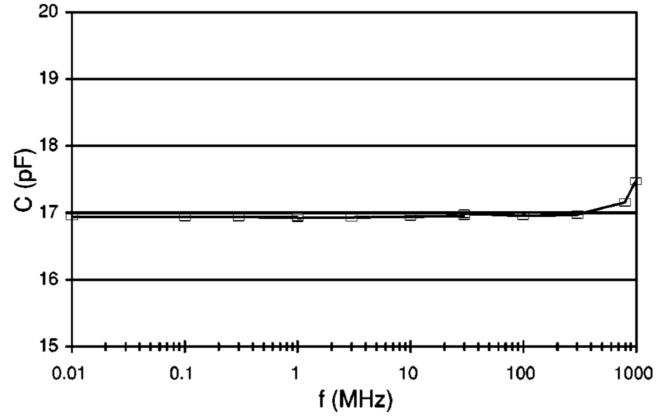

FIG. 6. Capacitance as a function of frequency for a 25-μm-thick polypropylene film in the coaxial holder.

transmission-line equations and an experimentally determined propagation constant and length. If this is done on a computer, then these constants could be stored with different values for different experimental conditions.

This translation from η, which is the admittance of the sample reduced by the characteristic admittance of the coaxial line, at $x=0$ to the value η_r at a location x, along a line with a propagation constant γ, is easily carried out using the transmission-line equation

$$\eta=\frac{1+\rho\ \exp(-\gamma x)}{1-\rho\ \exp(-\gamma x)}\tag{1}$$

of the coaxial line, where ρ is given by

$$\rho=\frac{\eta_r-1}{\eta_r+1}.\tag{2}$$

When this is done, one must be aware that there are lengths of line, especially near odd quarter wavelengths, where the transformation will have numeric difficulties.

This is illustrated in Fig. 5 where a capacitance is computed at the end of an open-circuited 20 cm air line for frequencies of 10 MHz to 1 GHz. The equivalent capacitance at 20 cm from its location goes through a singularity near 400 MHz. If data are measured near this frequency for a sample at the end of this line, it can be shown that there will be a large loss of precision. However, since this region is quite small on a logarithmic scale, it can be easily avoided and data in this narrow window interpolated from measurements on either side.

V. RESULTS AND DISCUSSION

To demonstrate the attainable accuracy of the method, a nominal 25 μm film of capacitor grade polypropylene was chosen. The film measured at 25 °C showed a tan δ of less than 50×10^{-6} measured at 1 kHz with the time-domain spectrometer in a good three-terminal holder. From the known behavior of polypropylene, the film should have zero-measurable loss and constant capacitance to the accuracy of the high-frequency instrumentation. The sample was metallized on one side with aluminum, punched into 9 disks 1.45 cm in diameter, and had a centered spot 0.50 cm in diameter deposited on the opposite side. Each piece was individually measured for thickness using a linear voltage differential transformer and precision micrometer head certified to 0.1 μm. Each sample was measured at frequencies from 1 kHz to 1 GHz in a 1, 1.5, 2, 3, 4.5, 6, 8, 10 sequence for each decade in frequency. The results are shown in Fig. 6.

The data shown in Fig. 6 are for one specific sample using the three bridges that covered the range from 1 kHz to 1 MHz, 100 kHz to 30 MHz, and 20 MHz to 1 GHz. The data in the regions of instrumental overlap were within the manufacturers's claim of accuracy, less than 0.1% for the first bridge, 0.2% to 5 MHz rising to 0.3% at 30 MHz for the second, and 2% for the third. The measured sample-to-sample relative uncertainty for the nine samples was less than 1%. No calibrations were applied to the data except for subtraction for the known, constant residuals measured without the samples for the first two bridges.

A comparison with Fig. 2, which is drawn to a comparable vertical scale, will immediately show the great improvement in precision up to 30 MHz, the limit of the earlier data. Except for the data above 500 MHz, the data can be regarded as essentially constant to measurement accuracy, which is expected for these samples as a result of their very low loss and the very small associated dispersion. There is a small rise in the capacitance starting above 500 MHz and rising to 5% at 1 GHz that is outside the limits of the bridge error and is probably due to the presence of higher-order modes becoming excited, possibly in a radial direction. This excitation marks the limit of accuracy for any lumped circuit approximation and the limit of this method. This phenomenon remains to be confirmed using smaller diameter inner electrodes where this effect should be minimized.

For the measured loss, tan δ was within zero to the measurement uncertainties for all frequencies. The data are not plotted as they would lie along the frequency axis with a scatter comparable to that in Fig. 6. These results for the loss measurements are expected, as mentioned previously, and demonstrate that the limit of loss resolution is being set by the measuring instrumentation rather than the sample holder. This result was verified using low-frequency measurements that had much higher loss resolution. Therefore, any measurable loss will be subject to the specified uncertainties at the bridge terminal, just as for the capacitance.

The use of a precision coaxial air line and connector has allowed a sample to be measured as a direct capacitance and an associated tan δ. For most free-standing films, it is possible to define an inner electrode area that does not compromise the overall bridge resolution over the entire frequency range. In addition, the sample holder does achieve the goal of covering a very broad range of frequencies on a single sample and within the best possible published instrumental uncertainty.

[1] M. G. Broadhurst and A. J. Bur, J. Res. Natl. Bur. Stand., Sect. C **69**, 165 (1965).

[2] K. Baba, T. Yamakami, and T. Fujimura, Jpn. J. Appl. Phys., Part 1 **24**, 1532 (1985).

[3] C.G. Montgomery, in *Principles of Microwave Circuits*,, edited by R. H. Dicke and E. M. Purcell (McGraw-Hill, New York, 1948).

[4] Specific instrumentation is mentioned in order to properly characterize the procedure. This does not imply any endorsement by the National Institute of Standards and Technology for suitability.

[5] F. I. Mopsik, Rev. Sci. Instrum. **55**, 79 (1984).