Broadband Permittivity Measurements of High Dielectric Constant Films

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Abstract* - Our investigation concerns measuring broadband dielectric permittivity and loss tangent of thin film high dielectric constant dielectric materials at microwave frequencies. The measurements are made in an APC-7 coaxial configuration where the test specimen represents a load terminating an air-filled coaxial transmission line. In contrast to conventional lumped capacitance approximations, the parallel plate capacitor filled with a dielectric film is treated as a distributed component consisting of a depressive, transmission line with a capacitance. The model expression for input impedance takes into consideration the wave propagation within the dielectric specimen section and correlates the network parameters with the relative complex permittivity of the specimen. The method is suitable for testing high-k polymer-composite materials having nominal thickness of 1 μm to 300 μm at frequencies of 100 MHz to 12 GHz. With proper calibration and computation the frequency range can be extended to 18 GHz .

***Index Terms* - dielectric materials, high frequency measurements, coaxial discontinuity**

I. INTRODUCTION

Broadband dielectric spectroscopy allows direct insight into molecular dynamics of materials. It covers a broad frequency range and therefore can be employed to study fast and slow molecular motions [1]. Broadband permittivity measurements of film dielectrics are also important in practical applications because the wide frequency range information is essential for many different industrial requirements. Among them, high permittivity films (high-k) are being developed for electronic applications operating at microwave frequencies [2] at which the existing broadband testing procedures frequently fail to produce meaningful results.

A parallel plate capacitor terminating a coaxial waveguide (transmission line) has been widely used in the broadband complex permittivity measurements. In this configuration, often referred to as a lumped capacitance method, a dielectric disk or rod sample of the diameter of the center conductor is placed at the end of coaxial waveguide as a capacitive termination [3].

Iskander and Stuchly proposed a highly refined technique for measuring the broadband dielectric properties of such materials utilizing the reflection coefficient. As a sample holder, they used a small-gap shunt capacitor terminating a coaxial line [4]. This technique is accurate at frequencies where the sample holder can be treated as a lumped capacitance. They empirically determined that this technique is accurate up to a frequency at which input impedance of the specimen decreases to one tenth (0.1) of the characteristic impedance of the coaxial line. This frequency limit is lower for thinner specimens that have a higher dielectric constant. For a 100 μm thick specimen with a dielectric constant of about 60, measured in the APC-7 configuration, the upper frequency limit falls to within 85 MHz. This is well below the desirable frequency range of about 10 GHz to 20 GHz. Marcuvitz analyzed an equivalent circuit of a coaxial line, terminated by a small gap capacitance, treating it as a quasi-electrostatic problem. His analysis assumes a principal propagating mode in the coaxial line and no propagation in the gap [5].

A more fundamental analysis of TEM wave scattering in a coaxial line terminated by a gap was performed by Eom et al, using a Fourier transform and mode matching technique [6]. The results agreed with the Marcuvitz model up to frequencies of 12 GHz, but no satisfactory physical solution was obtained for gaps thinner than 100 μm and a dielectric constant larger than 10.

In our earlier work we analyzed in detail the wave propagation in the film specimen terminating a coaxial line. We considered it as a distributed component having complex capacitance with residual inductance, for which we formulated an expression for the input impedance [7, 8]. In this paper, we present an application of this expression to measure complex permittivity of high-k film materials.

II. ANALYSIS

At frequencies where the specimen may be treated as a lumped capacitance, the input impedance, $\mathbf{Z}_{in}$, is given by expression (1a) and the real (ε') and imaginary (ε") component of the dielectric permittivity can be obtained from equations (1b) and (1c) respectively [3, 4]:

$$\mathbf{Z}_{in} = \frac{1}{j\omega C_p \varepsilon_r^*} \tag{1a}$$

$$\varepsilon' = \frac{-2|\mathbf{S}_{11}|\sin\phi}{\omega Z_0 C_p \left(1 + 2|\mathbf{S}_{11}|\cos\phi + |\mathbf{S}_{11}|^2\right)} \tag{1b}$$

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} = \frac{1-|\mathbf{S}_{11}|^2}{-2|\mathbf{S}_{11}|\sin\phi} \tag{1c}$$

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$|\mathbf{S}_{11}|$ is the magnitude and ϕ is the phase of the measured scattering coefficient, $\omega = 2\pi f$ is the angular frequency, and C_p is the specimen geometrical (air filled) capacitance.

$$C_p = \varepsilon_0\,(\pi\, a^2/4d) \qquad (2)$$

a is the specimen diameter, and d is the dielectric thickness of the specimen. Permittvities ε_0 and $\varepsilon_r^* = \varepsilon' - j\varepsilon''$ are that of air and the specimen material respectively.

In practice, the conventional formulas (1a - c) are accurate up to a frequency at which the input impedance of the specimen decreases to about one tenth (0.1) of the characteristic impedance of the coaxial line, i.e., about 5 Ω. Depending on the dielectric permittivity and thickness this upper frequency limit is typically below 1GHz.

At higher microwave frequencies, the specimen section filled with a high-k material represents a network of a transmission line with capacitance $C_p\varepsilon_r^*$. The input impedance, $\mathbf{Z}_{in}$, of such network is given by equation (3).

$$\mathbf{Z}_{in} = \frac{x\cot(x)}{j\omega C_p \varepsilon_r^*} + j\omega L_s \qquad (3)$$

L_s is the specimen residual inductance that we determined numerically using a High Frequency Structure Simulation software:

$$L_s = 1.27\ 10^{-7}\,[\mathrm{H\,/\,m}] * d\,[\mathrm{m}] \qquad (4)$$

The propagation term x is given by (5):

$$x = \omega l \sqrt{\varepsilon_r^*}\big/2c \qquad (5)$$

where, $l = 2.47 \times 10^{-3}$ m (2.47 mm) represents the propagation length in the specimen section and c is speed of light ($c = 2.99792\ 10^{8}$ m/s) [8].

At low frequencies, below series resonance frequency, f_{LC}, the propagation term $x\ cot\ (x)$ approaches 1, L_s can be neglected and equation (1) simplifies to well known formula (1a) for a shunt capacitance, $C_p\varepsilon_r^*$, terminating a transmission line. The relationship between the measured scattering coefficient $\mathbf{S}_{11}$ and the input impedance of a the specimen is given by (6):

$$\mathbf{Z}_{in} = Z_0\,(1+\mathbf{S}_{11})/(1-\mathbf{S}_{11}) \qquad (6)$$

where Z_0 is characteristic impedance of the APC-7 air-filled coaxial line, $Z_0 = 50$ Ω.

Combining equations (3) and (6) one can obtain equation (7) that relates the complex dielectric permittivity, ε_r^*, of the test specimen with the measurable scattering parameter $\mathbf{S}_{11}$.

$$\varepsilon_r^* = \frac{x\cot(x)}{j\omega C_p\left(Z_0\,(1+\mathbf{S}_{11})/(1-\mathbf{S}_{11}) - j\omega L_s\right)} \qquad (7)$$

Because the propagation term x depends on permittivity, equation (7) does not have direct analytical solution and needs to be solved iteratively. Description of a suitable procedure can be found in the reference [9]. The right-hand-side of (7) can be labeled as φ and re-arranged into a compact form (7a), which is more convenient in describing the iterative procedure shown below.

$$\varepsilon_r^* = \varphi(\varepsilon_r^*) \qquad (7a)$$

The iterative algorithm involves the following steps:

1. Computation of the complex permittivity by using equations (1b) and (1c). This is an initial trial solution of the iterative process for i=0, where i is the iterative step:

$$\varepsilon_r^*[i=0] = \varepsilon' - j\varepsilon'' \qquad (7b)$$

2. Computation of successive approximations for subsequent iterative steps i.

$$\varepsilon_r^*[i+1] = \varphi(\varepsilon_r^*[i]) \qquad (7c)$$

(i = 0, 1, 2, 3…)

3. The iteration procedure is terminated when the absolute value, Δ, of equation (7d) is sufficiently small, for example Δ smaller than 10^{-5}.

$$|\,\varepsilon_r^*[i] - \varepsilon_r^*[i-1]\,|\,/\,|\,\varepsilon_r^*[i]\,| < \Delta \qquad (7d)$$

Typically, it may require five to about twenty iterations to reach the terminating criterion. Commercially available software can be used to program and automate the computational steps 1 through 3 and solve (7) numerically for ε_r^* and the corresponding uncertainty values. The software should be capable of handling simultaneously both real and imaginary parts of complex $\mathbf{S}_{11}$, $x\,cot\,(x)$ and ε_r^*, (for example Agilent VEE or National Instruments LabView programming platforms can be employed).

III. EXPERIMENT

3.1 Test Fixture

The test fixture consists of two sections *A* and *B*, where the specimen is placed in between, as shown in Fig. 1. Section *A* is an APC-7 to an APC-3.5 microwave adapter with characteristic impedance of 50 Ω (Agilent 1250-1746). Section B is an altered APC-7 short termination (Agilent 04191-85300 or equivalent may be used), with a custom-machined gap to accommodate a specimen of particular thickness. When sections *A* and *B* are assembled, the depth, *d*, of the gap is equals the specimen thickness. Specimens with different thickness will require separate sections *B*. In the case of a specimen thinner than 10 μm, the center conductor of the APC-7 section *A* may be replaced with a fixed

3.05 mm diameter pin, machined precisely to achieve a flat and parallel contact between the film specimen and the

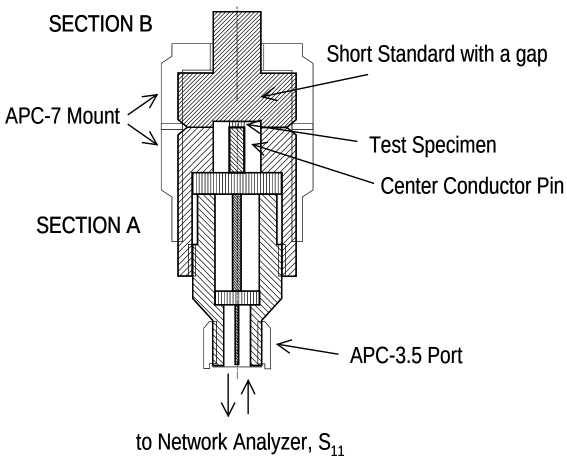

Fig. 1. Test fixture with a test specimen between sections A and B.

terminating section *B*. The diameter of the outer conductor, *b*, of section *A* is made 7.0 mm.

3.2 Test specimen

The test specimen consists of a circular disk capacitor having the nominal diameter, *a*, of 3.0 mm with metal electrodes on both sides. The dielectric thickness, *d*, may be in the range of 5 μm to 300 μm. Conducting metal electrodes, thickness of 0.1 μm to 0.5 μm, need to be coated on both sides of the dielectric. Sputtered copper or gold is recommended as a conducting material. To avoid electrical shorting, the diameter of the top electrode, which faces the section B of the test fixture (Fig.1), can be made 2.85 mm to 3.0 mm using a shadow mask. The diameter of the bottom electrode that faces the section A (Fig. 1), is within 3.0 mm to 3.05 mm, matching the diameter of the center conductor pin (Fig. 1). Diameter *a* along with the specimen dielectric thickness, *d*, determines the specimen geometrical capacitance, C_p (see equation (2) in section II). The diameter of the dielectric should be equal to the diameter of the bottom electrode.

In the case of thin dielectric films that are not freestanding and are on a supporting conductor such as copper foil, the supporting conductor can be used as the bottom electrode. A change in position of the calibration plane due to thickness of the bottom conductor can be compensated during measurements by adding an equivalent electrical delay However, the topside conductor should be removed and then the top surface of the dielectric should be re-coated with a 0.1 μm to 0.5 μm thick metal electrode.

3.3 Measurement

Measurements were performed by using an automatic vector network analyzer, Agilent 8720D operating in the frequency range of 100 MHz to 18 GHz. The instrument was equipped with a IEEE 488.2 I/O interface (GPIB) for transferring data between the network analyzer and a computing unit (a PC with a GPIB board). Connection between the test fixture (APC 3.5 adapter of section A) and the network analyzer was made using a phase preserving coaxial cable, Agilent 85131-60013. Open, Load, Short calibration was performed using Agilent 85050B 7 mm

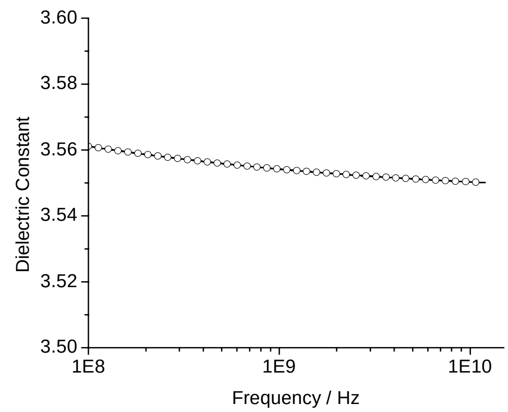

Fig. 2. Dielectric constant measured for a 25 μm thick dielectric with a nominal dielectric constant value of 3.5.

calibration standard. Figures 2 and 3 illustrate the dielectric constant measurements from 100 MHz to 12 GHz performed according to the above test method for typical low and high k materials. The results were obtained for a 25 μm thick dielectric with nominal value of the dielectric constant 3.5 (Fig. 2) and 11 (Fig. 3)

The uncertainty increases with increasing frequency. The maximum relative uncertainty in the dielectric constant is about 5%. The standard deviation in the dielectric loss tangent is about10^{-3}.

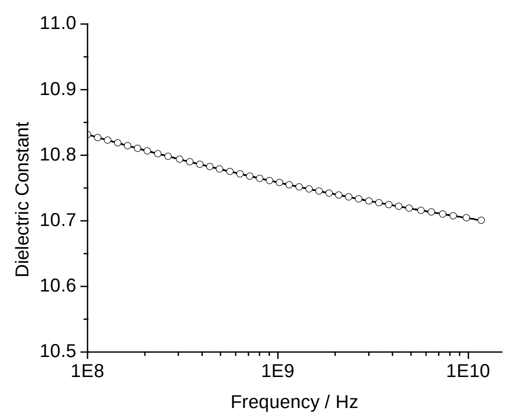

Fig. 3. Dielectric constant measured for a 15 μm thick dielectric with a nominal dielectric constant value of 11.

The presented APC-7 test fixture design may be utilized in the frequency range of 100 kHz to 18 GHz. The computational algorithm and in particular equations (4) and (5) have been validated up to the first cavity resonance frequency, f_{cav}, which is determined by the propagation length l, and the dielectric constant of the specimen:

$$f_{cav} = \frac{c}{l\,\mathrm{Re}(\sqrt{\varepsilon_r^*})} \approx 121/(\sqrt{\varepsilon_r}\ [GHz] \tag{8}$$

where Re indicates the real part of complex square root of permittivity and l =2.47 mm, which is the propagation length for the test fixture presented in Fig.1, [5]. For example, in the case of a specimen having the dielectric constant of 100 f_{cav} is about 12 GHz.

3.4 Accuracy considerations

Several uncertainty factors such as instrumentation, dimensional uncertainty of the test specimen geometry, roughness and conductivity of the conduction surfaces contribute to the combined uncertainty of the measurements. The complexity of modeling these factors is considerably higher within the frequency range of the LC resonance. Adequate analysis can be performed, however, by using the partial derivative technique [3] for equations (1b) and (1c) and considering the instrumentation and the dimensional errors. The standard uncertainty of $\mathbf{S}_{11}$ can be assumed to be within the manufacturer's specification for the network analyzer, about ± 0.005 dB for the magnitude and ± 0.5 ° for the phase. The combined relative standard uncertainty in geometrical capacitance measurements is typically smaller than 5 %, where the largest contributing factor is the uncertainty in the film thickness measurements.

Equation (4) for the residual inductance has been validated for specimens 8 μm to 300 μm thick.

Measurements in the frequency range of 100 MHz to 12 GHz are reproducible with relative combined uncertainty in ε'and ε" of better than 8 % for specimens having $\varepsilon' < 80$ and thickness $d < 300$ μm. The resolution in the dielectric loss tangent measurements is < 0.005. Additional limitations may arise from the systematic uncertainty of the particular instrumentation, calibration standards and the dimensional imperfections of the implemented test fixture. Furthermore, results may be not reliable at frequencies where $|\mathbf{Z}|$ decreases below 0.05 Ω.

IV. SUMMARY

We developed an expression for the input impedance of a coaxial line terminated with a complex capacitance specimen. The expression correlates the scattering parameters of the network with the complex permittivity of the specimen. Our approach allows overcoming the limits of the lumped capacitance methods and enables accurate complex permittivity measurements of film samples at microwave frequencies. The experimental permittivity results obtained in the broad frequency range correlated closely with published data. Especially encouraging are the accurate permittivity measurements obtained for high dielectric constant films. These materials are currently being developed for electronic applications operating at microwave frequencies at which the existing lumped element approximations for thin dielectric films fail to produce meaningful results. Results of our analysis also enable broadband dielectric measurements of thin films at higher, microwave frequencies that are of interest to bio-and nano-technology.

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