



IPC-TM-650 TEST METHODS MANUAL

1 Scope This test method describes procedure for measuring dielectric permittivity and loss tangent of embedded passive materials. The measurements are made in an APC-7 coaxial configuration where the test specimen represents a load terminating an air-filled coaxial transmission line. The method is suitable for testing high dielectric constant (high-k) polymer-composite materials having nominal thickness of 1 μm to 300 μm at frequencies of 100 MHz to 12 GHz. With proper use and computation the frequency range can be extended to 18 GHz. Existing measurement methods (see Section 2) assume quasi-static conditions in the thin film specimen, whereas this test method produces meaningful results at frequencies greater than hundreds of megahertz and where high-dielectric constant, thin film materials are to be measured.

This test method is for qualification of filled and unfilled, distributed capacitance, thin film materials where the permittivity in the frequency range of 100 MHz to 12 GHz is a critical functional parameter. The method is also applicable to polymer resist materials for embedded passive devices. This method fills a test method gap within the IPC-TM-650 Test Methods Manual for thin film, high-k dielectrics.

2 Applicable Documents

IPC-TM-650 Test Method Manual

- 2.5.5.1 Permittivity (Dielectric Constant) and Loss Tangent (Dissipation Factor) of Insulating Material at 1 MHz (Contacting Electrode Systems)
- 2.5.5.4 Dielectric Constant and Dissipation Factor of Printed Wiring Board Material-Micrometer Method
- 2.5.5.9 Permittivity and Loss Tangent, Parallel Plate, 1 MHz to 1.5 GHz

ASTM D 150 Standard Test Methods for AC Loss Characteristics & Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials

3 Terminology

3.1 Complex Permittivity, ϵ^* , $\epsilon^* = \epsilon_0 (\epsilon' - j\epsilon'')$ where $\epsilon_0 = 8.85419 \cdot 10^{-12}$ F/m is the dielectric permittivity of air [1], ϵ' is the relative dielectric constant and ϵ'' is the relative imaginary dielectric constant (the dielectric loss).

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3.2 Relative Permittivity, ϵ_r^* , is a dimensionless ratio of complex permittivity to the permittivity of air, $\epsilon_r^* = \epsilon^*/\epsilon_0 = \epsilon' - j\epsilon''$.

3.3 Dielectric Constant is the real part of the relative permittivity. The symbol used in this document is ϵ' . Other symbols such as K, k, K', k', ϵ_r and ϵ_r' are exchangeable symbols used in the technical literature.

3.4 Dielectric Loss Tangent, $\tan(\delta)$, is a dimensionless ratio of the dielectric loss to the dielectric constant, $\tan(\delta) = \epsilon''/\epsilon'$.

3.5 APC-7, Amphenol 7 mm 50 Ω Coaxial Connector; APC-3.5 Amphenol 3.5 mm Precision 50 Ω Coaxial Connector.

3.6 Scattering Coefficient, \mathbf{S}_{11} , is a ratio of incoming and outgoing power waves measured by a network analyzer through Port 1. \mathbf{S}_{11} is complex entity consisting of magnitude, $|\mathbf{S}_{11}|$, and phase, ϕ . In this document the circuit parameters that are complex numbers are in bold font.

3.7 Input Impedance, \mathbf{Z}_{in} , a complex entity consisting of magnitude and phase.

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

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

4 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 1 μm to 300 μm (1 $\mu\text{m} = 1$ micrometer).

4.1 Preparation Conducting metal electrodes, thickness of 0.1 μm to 0.5 μm , shall be coated on both sides of the dielectric. Sputtered copper or gold is recommended. To avoid electrical shorting, the diameter of the top electrode, which faces the Section B of the test fixture (Figure 1), may be 2.85 mm to 3.0 mm. The diameter of the bottom electrode that faces the Section A (Figure 1), shall be within 3.0 mm to 3.05 mm, matching the diameter of the center conductor pin (Figure 1). This is the diameter a of the specimen that along with the specimen dielectric thickness, d , determines the specimen geometrical capacitance, C_p (see Equation (3) in

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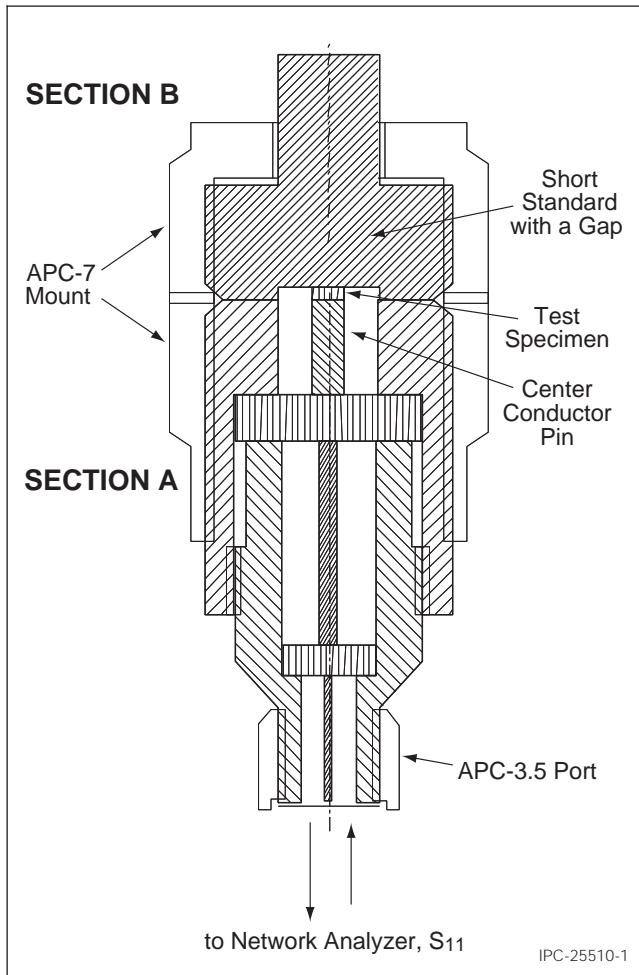


Figure 1 Test fixture with a test specimen between Sections A and B

7.2). The diameter of the dielectric should be equal to the diameter of the bottom electrode.

4.1.1 Preparation of the Test Specimen from Metal Clad Laminates The metal cladding should be removed from the dielectric, unless the thickness of the conductor is already within the recommended range of $0.1\ \mu\text{m}$ to $0.5\ \mu\text{m}$. The surfaces of the bare dielectric should be cleaned from conducting contaminants such as traces of ions to avoid possible corrosion of sputtered thin film metals, by rinsing in deionized water, drying, and then remetalizing by sputtering with copper or gold (see 4.1).

4.1.2 Thin Dielectric Films that are Not Free-Standing and Require Support The supporting conductor can be

used as the bottom electrode of the specimen. The topside conductor should be removed and then the top surface of the dielectric should be recoated to make the top electrode (see 4.1). The thickness of the bottom conductor can be compensated during measurements by adding an equivalent electrical delay (see 6.3.1).

5 Test Fixture The test fixture consists of two Sections A and B, where the specimen is placed in between, as shown in Figure 1. The detailed drawings are given in Section 11. Section A is an APC-7 to an APC-3.5 microwave adapter with characteristic impedance of $50\ \Omega$ (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 equal to the specimen thickness. Specimens with different thickness will require separate Sections B. In the case of a specimen thinner than $10\ \mu\text{m}$, the center conductor of the APC-7 Section A may be replaced with a fixed $3.05\ \text{mm}$ diameter pin, machined precisely to achieve a flat and parallel contact between the film specimen and the terminating Section B. The diameter of the outer conductor, b , of Section A is $7.0\ \text{mm}$ (see drawing in Section 11).

6 Measurement Procedure

6.1 Apparatus The measurement requires an automatic vector network analyzer operating in the frequency range of $100\ \text{MHz}$ to $18\ \text{GHz}$, for example an Agilent 8720D or equivalent. The instrument should be equipped with a IEEE 488.2 I/O interface for transferring data between the network analyzer and a computing unit, e.g., a personal computer (PC) with a General Purpose Input/Output Board (GPIB).

Connection between the test fixture (APC 3.5 adapter of Section A) and the network analyzer shall be made using a phase preserving coaxial cable, for example an Agilent 85131-60013 or equivalent.

6.2 Calibration Procedure Set the measurements range to be between $100\ \text{MHz}$ and $12\ \text{GHz}$. The number of data points should be in the range of 800. The power level should be set to $0\ \text{dBm}$ with a dynamic range of at least $-40\ \text{dBm}$ (desirably to $-60\ \text{dBm}$). Select the one Port S_{11} measuring mode and Smith-Chart format. Connect the phase preserving cable to the Port-1 of the network analyzer and to Section A of the test fixture. Attach a calibration standard to Section A of the test fixture. Perform an APC-7 Open, Load, Short calibration using suitable calibration standards (Agilent 85050B

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7 mm calibration kit or equivalent) in accordance with the manufacturer specification for the network analyzer. After calibration verify the following:

- The Open Standard produces an 'open trace' on the Smith Chart.
- The Broad Band 50 Ω Standard Load produces a dot trace located in the middle of the Smith Chart at 50 Ω , with phase angle equal to zero degree.
- The Short Standard produces a dot trace at 0 Ω , with a phase angle of 180°.

6.3 Measurements Determine the specimen dielectric thickness, d . The thickness of the sputtered conductor may be neglected. However, if the specimen was made on a conducting support (see 4.1.2) thicker than 0.5 μm , the thickness of the bottom conductor should be compensated by adding an equivalent electrical delay (see 6.3.1.). Verify that the diameter of both electrodes satisfies the required values (see 4.1). Ensure that the diameter of the bottom electrode facing the center conductor of Section A is in the range of 3.0 mm to 3.05 mm. Place the test specimen at the center conductor of Section A. Attach Section B of the test fixture.

Measure the complex scattering coefficient, \mathbf{S}_{11} . For a capacitive load (a dielectric specimen), the trace should represent a semicircle on the lower half portion of the Smith Chart (Figure 2), going from a high impedance region at lowest frequencies towards a low impedance region as the frequency increases. The radius of the semicircle represents the reflection coefficient, which for a loss-less dielectric approaches the value of one. In the case of an inductive specimen, the trace should represent a semicircle on the higher half portion of the Smith Chart, going from a low impedance region, $Z \approx 0$ at lowest frequencies, towards a high impedance region as the frequency increases.

Example measurements obtained for a specimen having the dielectric thickness of 80 μm , dielectric constant of 69 and the dielectric loss tangent of 0.0023 are shown in Figure 2. The trace crosses the zero impedance point at the series resonance frequency, f_{LC} , of 5.1 GHz, beyond which the load character changes from capacitive to inductive. A local loop on the chart indicates the first cavity resonance at f_{cav} of 14.65 GHz.

After the frequency scan is completed, transfer the entire digitized trace spectrum containing the \mathbf{S}_{11} amplitude and \mathbf{S}_{11} phase at each measured frequency to a PC via a GPIB link.

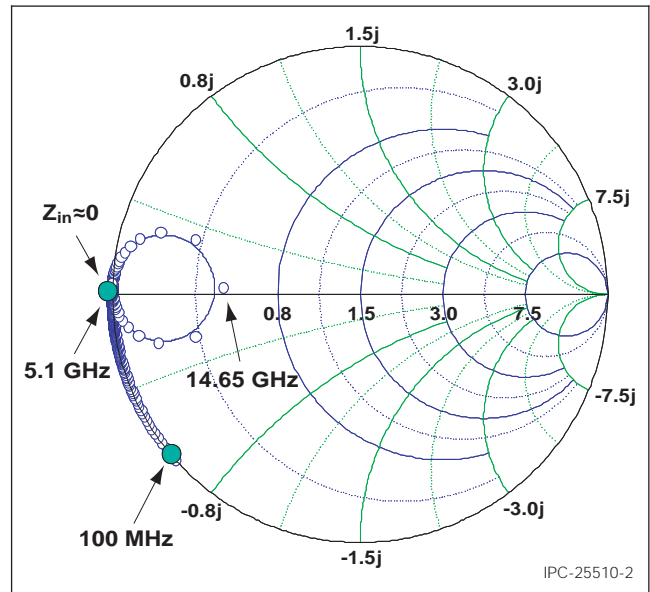


Figure 2 Example measurements plotted in a Smith chart Format for an 80 μm thick specimen with permittivity of 69 - $j0.16$.

6.3.1 Compensation for a Finite Thickness of the Specimen Bottom Conductor

Adding an electrical delay to the test structure can compensate thickness of the bottom electrode conductor. This procedure moves the reference plane established during calibration (see drawings of the test fixture in Section 11), to a new position located at the interface between the bottom conductor and the dielectric. The plane should be moved away from the generator a distance equal to the actual thickness of the bottom conductor. The electrical delay procedure should be conducted in accordance to the operating manual for the network analyzer before transferring the data to a PC.

7 Calculations

7.1 Impedance Determine the experimental complex impedance, Z_{in} , of the specimen at each frequency point, f , according to Equation (1) presented in 3.7. Example results obtained for a 25 μm thick dielectric with ($\epsilon' = 10$ and $\tan(\delta)$ of 0.01 are shown in Figure 3.

7.2 Specimen Permittivity At frequencies where the specimen may be treated as a lumped capacitance, where $|Z|$ is larger than 5 Ω (see Figure 3, References [2,3]), the input impedance is given by Equation (2a) and the real (ϵ') and imaginary (ϵ'') component of the dielectric permittivity can be

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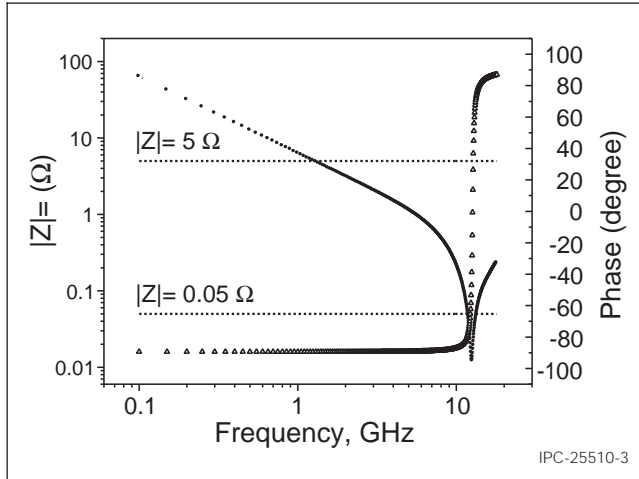


Figure 3 Impedance magnitude (circles) and phase (triangles) for a 25 μm thick dielectric film with ϵ' of 10 and $\tan(\delta)$ of 0.01.

obtained directly from Equations (2b) and (2c) respectively Reference [2]:

$$\mathbf{Z}_{in}^s = \frac{1}{j\omega C_p \epsilon_r^*} \quad (2a)$$

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

$$\tan \delta' = \frac{\epsilon''}{\epsilon'} = \frac{1 - |\mathbf{S}_{11}|^2}{-2|\mathbf{S}_{11}| \sin \phi} \quad (2c)$$

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

$$C_p = \epsilon_0 (\pi a^2 / 4d) [F] \quad (3)$$

a is the specimen diameter, and d is the dielectric thickness of the specimen (in units of meters). Permittivities ϵ_0 and ϵ_r^* are defined in 3.1 and 3.2. In Equation (3), the specimen diameter $a = 3.0 \times 10^{-3}$ m (3.0 mm), should match the diameter of the central conductor pin (see 4.1, Figure 1). Note that the actual diameter of the top electrode may be between 2.85×10^{-3} m to 3.0×10^{-3} m (2.85 mm to 3.0 mm in 4.1).

In practice, the conventional formulas (2a - 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. In the example given in Figure 3, this upper frequency limit is about 1.5 GHz. Some practical considerations regarding this limitation are discussed in References [4 and 5].

At higher microwave frequencies, the specimen section filled with a high-k material represents a network of a transmission line with capacitance $C_p \epsilon_r^*$. The input impedance, \mathbf{Z}_{in}^s , of such network is given by Equation (4) (see Reference [6]).

$$\mathbf{Z}_{in}^s = \frac{x \cot(x)}{j\omega C_p \epsilon_r^*} + j\omega L_s [\Omega] \quad (4)$$

L_s is the specimen residual inductance,

$$L_s = 1.27 \cdot 10^{-7} [H/m] * d [m] \quad (5)$$

and the propagation term x is given by (6):

$$x = \omega \sqrt{\epsilon_r^*} / 2c \quad (6)$$

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 ($c = 2.99792 \cdot 10^8$ m/s). At low frequencies, below series resonance frequency, f_{LC} , the propagation term $x \cot(x)$ approaches 1, L_s can be neglected and Equation (4) simplifies to well known formula (2a) for a shunt capacitance, $C_p \epsilon_r^*$, terminating a transmission line.

7.3 Computational Algorithm for Permittivity Combining Equations (1) and (4) leads to Equation (7) that relates the dielectric permittivity, ϵ_r^* , of the test specimen with the measurable scattering parameter \mathbf{S}_{11} .

$$\epsilon_r^* = \frac{x \cot(x)}{j\omega C_p (Z_0 (1 + \mathbf{S}_{11}) / (1 - \mathbf{S}_{11}) - j\omega L_s)} \quad (7)$$

Because the propagation term x depends on permittivity (Equation (6)), Equation (7) needs to be solved iteratively. Description of a suitable procedure can be found in the Reference [7].

According to the Reference [7], the right-hand-side of (7) can be labeled as ϕ and rearranged into a compact form (7a), which is more convenient in describing the iterative procedure shown below.

$$\epsilon_r^* = \phi(\epsilon_r^*) \quad (7a)$$

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For each frequency repeat the following procedure:

1. Compute the complex permittivity using Equations (2b) and (2c). This is an initial trial solution of the iterative process for $k=0$, where k is the iterative step:

$$\epsilon_r^* [k = 0] = \epsilon' - j\epsilon'' \quad (7b)$$

2. Compute successive approximations for subsequent iterative steps k .

$$\epsilon_r^* [k + 1] = \Phi (\epsilon_r^* [k]) \quad (7c)$$

$(k = 0, 1, 2, 3, \dots)$

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

$$|\epsilon_r^* [k] - \epsilon_r^* [k - 1]| / |\epsilon_r^* [k]| < 10^{-5} \quad (7d)$$

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 Equation (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 Visual Basic, C or Agilent VEE and National Instruments LabView programming platforms can be employed).

8 Report The report shall include:

- Dimensions of the specimen.
- Plot of magnitude and phase of the measured impedance as a function of frequency, (similar to Figure 3) or Smith Chart.
- Plot of ϵ' and ϵ'' or ϵ' and $\tan \delta$ as a function of frequency.

9 Notes

9.1 Measurements at Frequency Range Above 12 GHz

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:

$$f_{cav} = \frac{c}{l \operatorname{Re}(\sqrt{\epsilon_r^*})} \approx 121 / (\sqrt{\epsilon_r} [\text{GHz}]) \quad (8)$$

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 Figure 1, [5]. For example, in the case of a specimen having the dielectric constant of 100 f_{cav} is about 12 GHz.

9.2 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 [1] for Equations (2b) and (2c) 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 $\pm 0.5^\circ$ for the phase. The combined relative standard uncertainty in geometrical capacitance measurements is typically better than 5%, where the largest contributing factor is the uncertainty in the film thickness measurements.

Equation (5) for the residual inductance has been validated for specimens 8 μm to 300 μm thick. However, since residual inductance becomes smaller with thinner dielectrics, measurements can be accurately made for sample thicknesses down to 1 μm .

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 $\epsilon' < 80$ 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 actually implemented test fixture. Results may be not reliable at frequencies where $|\mathbf{Z}|$ decreases below 0.05 Ω , which in Figure 3 is shown as a frequency range of 11.9 GHz to 13.5 GHz.

9.3 Test Software Test software enabling this technique to be performed is available in the Agilent VEE platform. Please contact Dr. Jan Obrzut at NIST-Gaithersburg, MD (jan.obrzut@nist.gov) to obtain such.

9.4 Metric Units of Measure This test method uses only metric units of measure, as is the case with nearly all such high frequency test methods. Conversion to English/Imperial units has not been done in this document, as any conversions

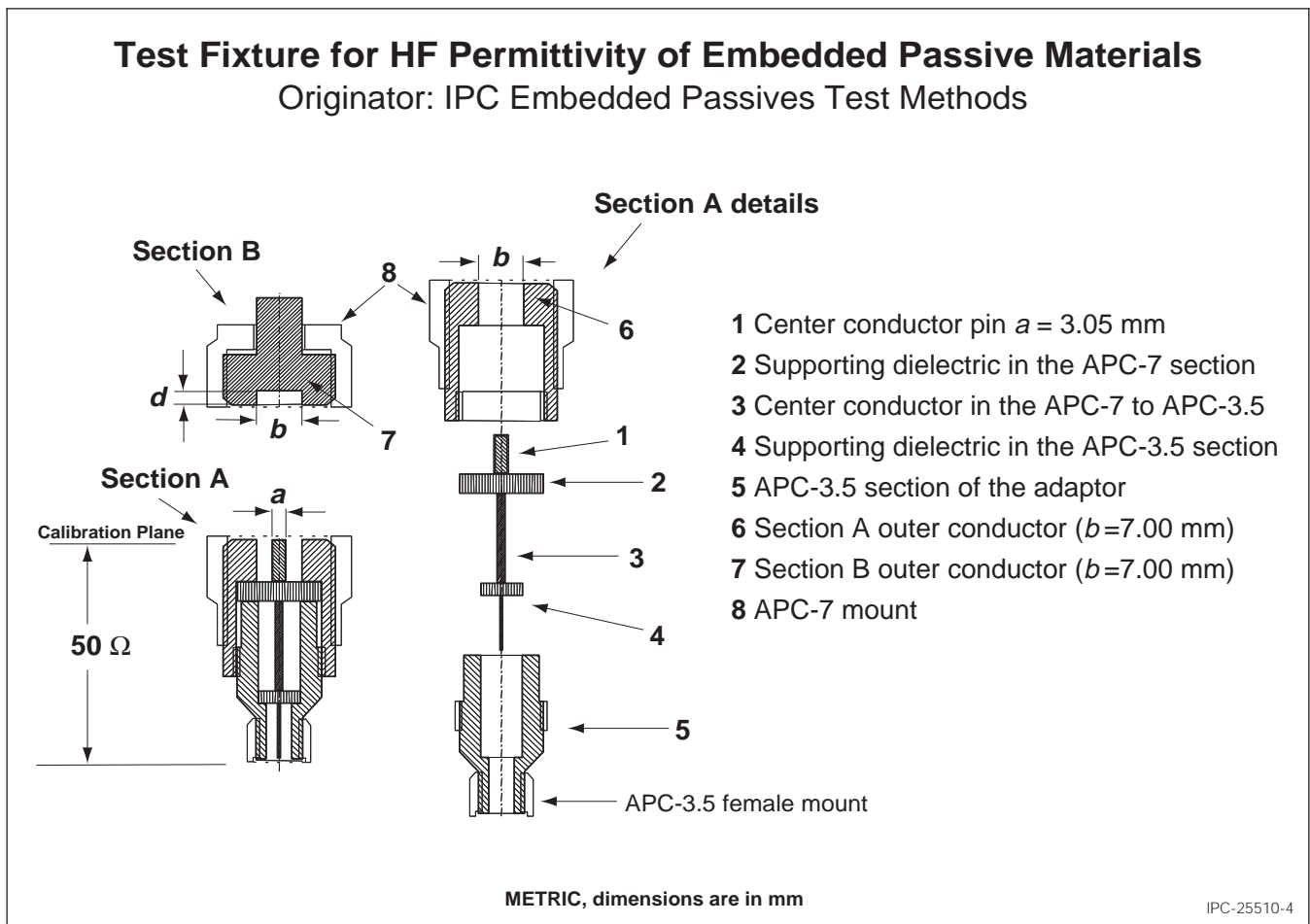
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from metric units will lead to inherent accuracy and/or precision errors.

10 References

- [1] Fundamental Physical Constant, Permittivity, <http://physics.nist.gov/cgi-bin/cuu/Value?ep0>|search_for=permittivity
- [2] M. A. Stuchly, S. S. Stuchly, "Coaxial line reflection methods for measuring dielectric properties of biological substances at radio and microwave frequencies: A review," IEEE Trans. Instrum. Meas., vol. 29, pp. 176-183, 1980.
- [3] N. Marcuvitz, Waveguide Handbook. McGraw-Hill, New York: 1951.
- [4] H. J. Eom, Y.C. Noh, J.K. Park, "Scattering analysis of a coaxial line terminated by a gap," IEEE Microwave Guided Wave Lett., vol. 8, pp. 218-219, 1998.
- [5] N.-E. Belhadj-Tahar, O. Dubrunfaut, A. Fourier-Lamer, "Equivalent circuit for coaxial discontinuities filled with dielectric materials - frequency extension of the Marcuvitz's circuit" J. Electromagnet. Wave, vol. 15, pp. 727-743, 2001.
- [6] J. Obrzut, A. Anopchenko, "Input Impedance of a Coaxial Line Terminated with a Complex Gap Capacitance - Numerical and Experimental Analysis" IEEE Trans. Instrum. Meas., vol. 53(4), Aug. (2004).
- [7] "Mathematical Handbook for Scientists and Engineers," G. A. Korn and T. M. Korn, McGraw-Hill, 2nd edition (1968), page 719.

11 Test Fixture Drawings



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APPENDIX

Example of the specimen preparation

Figure A1 shows an example of three test specimens prepared from a free standing dielectric film. The dielectric was 25 μm thick. About 242 nm thick layer of gold was sputtered on both sides. A shadow mask with a pattern of circles having each diameter of 2.9 mm was used to pattern the top surface. The bottom surface was sputtered without masking. The specimens were extracted from the film using a 3.0 mm puncher.

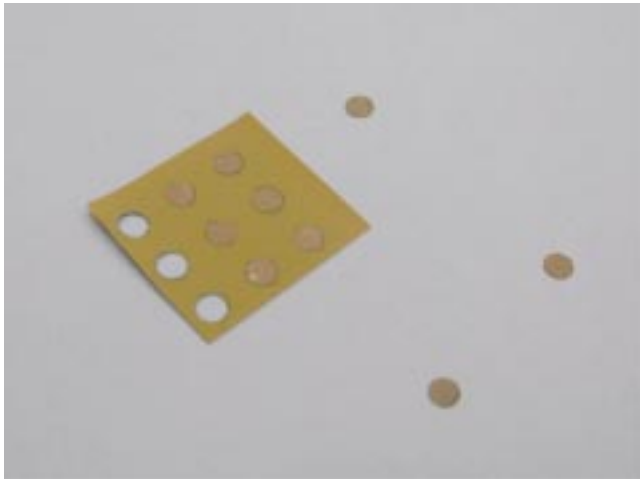


Figure A1 Test specimens

Instrumentation Example

Figure A2 shows a photo of the assembled test fixture during measurements. An APC-7 to APC-3.5 microwave adapter (Agilent 1250-1746) is connected via a phase preserving coaxial cable (Agilent 85131-60013) to a network analyzer. An APC-7 short termination (Agilent 04191-85300), is attached on top of the APC-7 to APC-3.5 adapter. The termination has a custom machined gap to accommodate a specimen of a particular thickness.

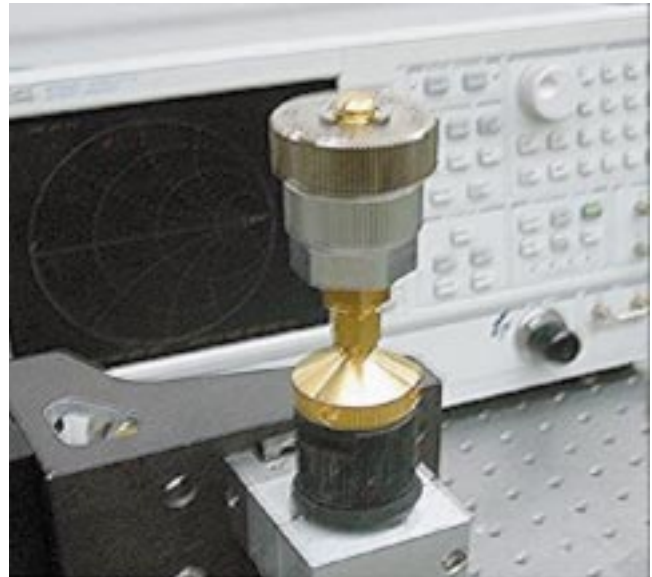


Figure A2 Test instrumentation

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Example measurement results

Figures A3 and A4 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. In the examples, 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 about 0.001.

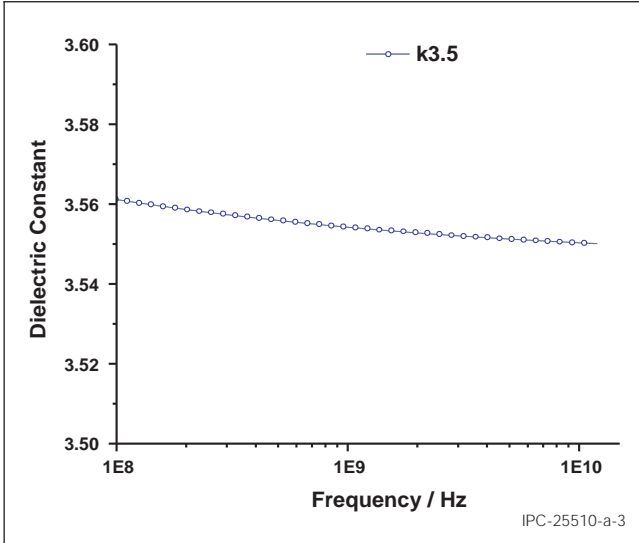


Figure A3 Dielectric constant measured for a 25 µm thick dielectric with a nominal dielectric constant value of 3.5.

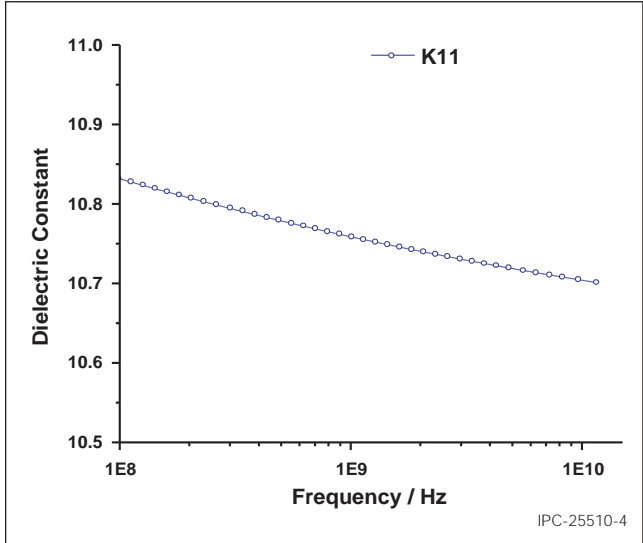


Figure A4 Dielectric constant measured for a 15 µm thick dielectric with a nominal dielectric constant value of 11.

Certain equipment and instrumentation is identified in this document in order to adequately specify the experimental procedure. This does not imply any recommendations that these are the most suitable for that purpose.