



IPC-TM-650 TEST METHODS MANUAL

1.0 Scope

1.1 This test method establishes a procedure for determining the low frequency out of plane dielectric constant and loss tangent of organic free films using a two-fluid method. (Low frequency is defined as less than or equal to 1 MHz in this method.)

1.2 The two-fluid method uses a three-terminal guarded electrode cell and allows for capacitance measurements which are independent of sample dimensions, electrode size, and electrode spacing. The two fluids to be used in this procedure are air and 1.0 centistoke silicone fluid.

1.3 The change in dielectric constant is the key indicator of moisture uptake and can be used to estimate the mass uptake of water by an organic film. For this reason, the dielectric constant is to be measured on films conditioned at two relative humidity levels and the difference used to estimate a value defined as "electrical water" for the sample.

2.0 Applicable Documents

ASTM D 150 Standard Test Methods for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials

ASTM D 1531 Standard Test Method for Relative Permittivity (Dielectric Constant) and Dissipation Factor by Fluid Displacement Procedure

ASTM E 104 Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions

3.0 Test Specimen

3.1 Free films should have a minimum thickness of approximately 25 µm for ease of handling. Films may be stacked to achieve a total test specimen thickness of approximately 50 µm (minimum) or greater for the test.

3.2 Note that sample handling becomes more difficult as the number of film layers used in a test specimen increases, especially when trying to keep air bubbles from between the layers

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when immersed in the silicone fluid. However, greater accuracy is achieved with thicker test specimens.

3.3 Process the organic coating according to the manufacturer's recommended procedures. Specify the film preparation method, including coating method, substrate type, and release method as follows.

3.3.1 Particulate Control Many test methods for determining the physical properties of polymers are sensitive to defects or particulates in the test specimens (e.g., measurement of tensile properties). The preparation of organic coatings should be conducted in a particulate free environment (e.g., laminar flow hood or clean room) to minimize particulate defects in the films.

3.3.2 Preparation of Uniform Coatings

3.3.2.1 Apply the test material using one of the coating methods listed in Column A of Table I to one of the substrates listed in Column B of Table I following the manufacturer's recommended procedure.

3.3.2.2 Process the coating according to the manufacturer's recommended procedures.

Table I

A: Coating Methods:	B: Substrates
Spin Coater	125 mm Polished Silicon Wafer
Automatic Extrusion Coater	100 x 100 mm Ceramic Substrate
Automatic Spray Coater	Polished Aluminum Disk
Motor Driven Blade Applicator	Glass or Quartz Disk
Hand Held Spray Gun	Tin Coated Disk
Hand Held Blade Applicator	Aluminum (or other metal) Panel
Vacuum Deposition	Glass Plate
Sputter	PTFE Panel
Evaporation	PTFE Panel

3.3.3 Patterning Techniques

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3.3.3.1 The organic or metal coating can be patterned using one of the following techniques.

- Wet Etch
- Plasma Etch
- RIE Etch
- Laser Ablation
- Photosensitive Material

3.3.3.2 Patterning may involve the use of organic photoresists or metal hard masks. Descriptions of how these materials were processed must be provided.

3.3.4 Release Methods

3.3.4.1 Buffered HF Solution

3.3.4.1.1 This release method can be used to remove films that have been coated on silicon wafers containing an oxide layer.

3.3.4.1.2 Score completely around the outside of the coated substrates approximately 3 mm (1/8 inch) in from the edge. A template (e.g., inverted funnel) can be used as a cutting aid.

3.3.4.1.3 Place several rubber bands around the coated substrate to prevent the film from floating away.

3.3.4.1.4 Place the coated substrates in a *Teflon® boat, leaving a space between each coated substrate. Be sure to write down the order of the coated substrates since the HF will remove any ink labeling on the backs of the coated substrates.

3.3.4.1.5 Place the boat of coated substrates into a plastic container filled with a buffered (6:1) HF solution.

3.3.4.1.6 Remove the boat of coated substrates from the bath once the films have lifted from the coated substrates (15 min to 2 hrs.). Place the boat in a clean empty container and rinse with water for 1 hour or until all traces of HF have been removed (the water will turn from cloudy to clear).

3.3.4.1.7 Carefully remove the films from the substrates while still wet and place between two absorbent sheets to dry. Label the absorbent sheets, put in a safe place, and allow the films to dry overnight.

3.3.4.2 Buffered HCL Solution

3.3.4.2.1 This release method can be used to remove films coated on aluminum disks or panels.

3.3.4.2.2 Secure the test specimen to a glass plate with several rubber bands to prevent the film from floating away.

3.3.4.2.3 Place the coated substrate in a bath of buffered HCl solution. Stir the bath to keep a fresh supply of HCl flowing past the substrate and to remove any oxide that has formed.

3.3.4.2.4 After the substrate has been completely removed, carefully pick up an end of the free film with tweezers, and place the film in a clean empty container and rinse with water for 1 hour or until all traces of HCl have been removed.

3.3.4.2.5 Remove the films while still wet and place between two absorbent sheets to dry. Label the absorbent sheets, put in a safe place, and allow the films to dry overnight.

3.3.4.3 Mercury Amalgam

3.3.4.3.1 This release method can be used to remove films that are coated onto substrates coated with a layer of tin.

3.3.4.3.2 Place the tin coated substrate film side up in a pool of mercury to remove the tin by amalgamation.

3.3.4.3.3 After amalgamation is completed, carefully pick up an end of the free film with tweezers, and carefully brush the film free of mercury and amalgam with a soft camel hair brush.

3.3.4.4 Boiling Water

3.3.4.4.1 This release method can be used to remove films coated on any of the recommended substrates. This release method generally can not be used if the material contains an adhesion promoter (coupling agent) or if the manufacturer recommends that the material be tested along with the use of an adhesion promoter.

3.3.4.4.2 Place the coated substrate in a bath of boiling water.

3.3.4.4.3 Once the film has lifted from the substrate (or can be easily peeled from the substrate without tearing) carefully remove the films while still wet and place between two absorbent sheets to dry. Label the absorbent sheets, put in a safe place, and allow the films to dry overnight.

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3.4 Reporting of Specimen Processing History

3.4.1 Specify the thermal processing history of the film as follows.

3.4.2 The properties of polymers are highly dependent on the thermal history of the polymer. When performing cross laboratory comparisons of properties, identical processing conditions must be used. The test results must list in specific detail how the polymer was processed. The description should include the following information:

- Coating method and conditions
- Substrate
- Processing conditions (time, temperature, etc.)
- Patterning technique and conditions
- Release method

For example:

The solution was deposited onto 125 mm silicon wafers containing 1500 Å of thermally grown oxide by spin coating at 3000 RPM for 30 sec. The coatings were processed as follows:

Dry: 135°C for 30 min. in air in an oven

Cure: Ramp from ambient to 200°C at 2°C/min in nitrogen

Hold at 200°C for 30 min

Ramp from 200°C to 350°C at 2°C/min

Hold at 350°C for 60 min

Ramp from 350°C to ambient at 5°C/min

The cured coatings were patterned using an aluminum mask and oxygen plasma. The aluminum mask was deposited by sputtering and wet etched. The cured films were released from the silicon wafers by dissolving the oxide in a buffered HF bath.

3.4.3 The physical and electrical properties of polymers are affected by temperature and relative humidity in a manner that may significantly affect test results. In order to make quantitative comparisons of different materials and between different laboratories, the temperature and relative humidity conditions that the test specimen are exposed to both prior to and during testing must be standardized.

3.5 Test samples should be cut into square or rectangular shapes at least 2 cm greater than the electrode diameter.

3.6 Clean the samples with a suitable solvent (e.g., acetone) or other means and allow to dry. Wipe the samples free of

dust and lint particles with Masterwipe cleaning wand and follow with static removal using the Zerostat device prior to relative humidity conditioning.

4.0 Apparatus or Material

- HP 4275 LCR meter or equivalent
- Two-fluid cell reference ASTM D 1531, Figure 1 and ASTM D 150
- High quality coaxial test leads (kept as short as possible)
- 1.0 centistoke silicone fluid, Dow Corning No. 200, or equivalent
- 3M Masterwipe cleaning wand, or equivalent
- Zerostat antistatic instrument, or equivalent
- Nitrogen purged dry box
- Quality paper filter, Whatman No. 1 Qualitative, or equivalent
- Humidity chamber prepared in accordance with ASTM E 104 for conditioning samples at an elevated humidity level

5.0 Procedure

5.1 Sample Preparation For Low Humidity Test (films conditioned at less than 5% R.H.)

5.1.1 Dry the clean prepared films in a nitrogen purged forced air oven overnight at 120°C.

5.1.2 Place the dried films into a nitrogen purged dry box @ 22°C for storage until tested.

5.2 Sample Preparation For Elevated Humidity Test (films conditioned at approximately 53% R.H.)

5.2.1 Place the dry films that were used in the low humidity test into a 53% R.H. chamber for conditioning prior to testing. (Use a chamber prepared with magnesium nitrate ($Mg(NO_3)_2 \cdot 6H_2O$) to achieve 53% R.H. at 22°C.)

5.2.2 Films should be conditioned for 3 to 7 days prior to testing, or until a quasi-steady state weight gain has been achieved. Tests have shown that most of the water uptake occurs very rapidly (within 24 hours) in thin organic films. (See 6.1.1)

5.3 Test Cell Preparation

5.3.1 Connect the high potential leads from the LCR meter to the movable electrode. Connect the low potential leads from LCR meter to the stationary electrode. Connect the guard ring to ground.

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5.3.2 Zero the LCR meter according to manufacturer's instructions.

5.3.3 Clean the inside of the cell with filtered air to remove lint and dust particles.

5.3.4 Set the cell electrode spacing so that the sample occupies at least 80% of the gap. Once set, it is very important that the gap is not changed between any of the measurements.

5.4 Measurement

5.4.1 All measurements should be performed at $22 \pm 1^\circ\text{C}$.

5.4.2 Measure capacitance (C_1) of the cell in air at 10 KHz and 1 MHz.

5.4.3 Transfer test specimen from conditioning environment to test cell and measure capacitance (C_2) of the cell plus sample in air at 10 KHz and 1 MHz.

5.4.4 Remove sample from test cell and return to conditioning environment.

5.4.5 Carefully pour the silicone fluid into the cell to avoid formation of air bubbles.

5.4.6 Measure capacitance (C_3) and loss factor (D_3) of the cell in silicone fluid at 10 KHz and 1 MHz.

5.4.7 Transfer test specimen from conditioning environment to test cell and measure capacitance (C_4) and loss factor (D_4) of the cell plus sample in silicone fluid at 10 KHz and 1 MHz. There must not be any air bubbles trapped between multiple film layers when the sample is immersed in the silicone fluid.

5.4.8 Measurement Considerations

5.4.8.1 Keep test specimens in the conditioning environment as much as possible between measurements.

5.4.8.2 Avoid over-handling of films to prevent contamination with lint or other particles.

5.4.8.3 Thin films tend to pick up static charge during the process of sliding into and out of the electrode gap, therefore, it is necessary to perform static removal periodically during the

test procedure to keep the sample moving freely in and out of the gap.

5.4.8.4 It is important that both measurements are made on the same area of the specimen because the thickness may not be the same at all points on the films. To ensure that the same area is measured each time, a specimen holder may be used. Specimen holders also facilitate handling thin samples that have little stiffness and are difficult to slide down between the electrodes. The specimen holder can be a U-shaped piece of thin plastic sheeting which secures 3 edges of the test specimen.

5.5 Post-Measurement Clean-up

5.5.1 Remove the silicone fluid from the test specimens by wiping with an appropriate solvent (e.g., acetone). Use low linting wipes to minimize particle accumulation on the films.

5.5.2 Wipe the samples free of dust and lint particles with Masterwipe cleaning wand and follow with static removal using the Zerostat device.

5.5.3 Return test specimens to conditioning environment.

5.5.4 Remove the silicone fluid from the test cell by wiping with an appropriate solvent (e.g., acetone). Use low linting wipes to minimize particle accumulation on the interior surface of the cell.

5.5.5 Clean the inside of the cell with filtered air to remove lint and dust particles.

5.5.6 Filter the silicone fluid through a quality filter paper after each use before it is returned to its storage container.

5.6 Duplicate Measurement

5.6.1 Allow the test specimen to remain in the conditioning environment for 3 to 7 days after the initial test.

5.6.2 Repeat steps 5.3 through 5.5.

5.6.3 Perform duplicate measurements for samples conditioned at both the low and elevated humidity levels.

5.7 Calculations – (See 6.1.2)

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5.7.1 Definition of Symbols:

C_1, C_2, C_3, C_4 = capacitance measurements as specified in 5.4.2, 5.4.3, 5.4.6, 5.4.7

D_3, D_4 = loss tangent measurements as specified in 5.4.6, 5.4.7

k' = dielectric constant of sample

C_o = cell vacuum capacitance = C_1/k_a

k_a = dielectric constant of air = 1.00059

D = loss tangent of sample

EW = "electrical water"

5.7.2 Formula for dielectric constant of sample:

$$k' = \frac{1}{C_o} \left[C_1 + \frac{C_4(C_2 - C_1)(C_3 - C_1)}{C_4(C_2 - C_1) C_2(C_4 - C_3)} \right]$$

Report values at 10 KHz and 1 MHz.

5.7.3 Formula for loss tangent of sample:

$$D = D_4 + \frac{(k' C_o - C_4)(D_4 - D_3)}{(C_4 - C_3)}$$

Report values at 10 KHz and 1 MHz.

5.7.4 The formula used to estimate the moisture uptake of the organic film is shown below. This calculated value for moisture uptake defined as "electrical water" is based on the difference between the dielectric constants measured dry and at an elevated humidity level and normalized to 100% R.H.

$$EW = \frac{2[k'(@ 53\% RH, 1 MHz) - k'(@ dry, 1 MHz)]}{53} \times 100\%$$

6.0 Notes

6.1 Additional Reference

6.1.1 Beuhler, A. J., Nowicki, N. R., and Gaudette, J. M., "Dielectric Characterization of Water in Polyimide and Poly(amide-imide) Thin Films," ACS Symposium Series 407, 1989, pp. 67-76.

6.1.2 Johnson, L. G., and Chomicz, J., "New Technique to Measure the Dielectric Properties of Thin Insulations," Insulation, September 1968, pp 61-67.