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IPC-TM-650
TEST METHODS MANUAL

Number	
2.4.24.4	
Subject	
Glass Transition and Modulus of Materials Used in High Density Interconnection (HDI) and Microvias - DMA Method	
Date	Revision
11/98	
Originating Task Group	
HDI Test Methods Task Group (D-42a)	

1 Scope This test is designed to determine the glass transition temperature (T_g) and room temperature storage modulus (E') of dielectric materials used in High Density Interconnect (HDI) and Microvias by the use of dynamic mechanical analysis (DMA).

When testing a stand alone HDI dielectric layer, DMA will provide modulus as a function of temperature and glass transition for this layer. When DMA is used on built-up constructions, the data will be a complex curve representing the composite moduli and glass transitions.

Two methods are presented:

- Method A for thick specimens
- Method B for thin specimens (recommended for HDIS and Microvia dielectric layers).

For anisotropic materials (reinforced dielectrics), the x and y directions will have different modulus vs. temperature behavior. Anisotropic materials shall be tested in both the x and y directions.

2 Applicable Documents

2.1 ASTM Documents

E 1640 Test Method for Assignment of the Glass Transition Temperature by Dynamic Mechanical Analysis

D 4065 Standard Practice for Determining and Reporting Dynamic Mechanical Properties for Plastics

D 4092 Standard Terminology Relating to Dynamic Mechanical Measurements on Plastics

3 Test Specimen

3.1 Size

Method A Flexural bending geometry – thick specimens (>0.5 mm): Specimens shall be approximately 8 mm to 12 mm wide, 20 mm to 40 mm long, and 1 mm to 2 mm thick. The thickness shall be a minimum of 0.5 mm; for thicknesses <0.50 mm, use Method B. An aspect ratio of length/thickness = 10/1 or greater should be maintained. Exact specimen dimensions should be determined by the apparatus used.

Method B Thin film tension geometry – thin specimens (<0.50 mm): Specimens shall be approximately 15 mm to 20 mm long and 2 mm wide. The minimum thickness is determined by the strength of the material; it should not break during testing. Exact specimen dimensions may be determined by the apparatus used.

3.2 All specimens should be fully cured according to manufacturer's recommendations. Thick specimens may be made by use of multiple lamination/cure cycles if required.

3.3 Unless otherwise specified, one specimen shall be tested, to be taken from a random location in the material in question.

4 Apparatus or Material

4.1 A DMA capable of determination of modulus to +1% precision and tan δ resolution of 0.01 over the specified temperature range. The DMA will preferably have computer data acquisition and analysis. The DMA must have an environmental chamber capable of having inert flush gas and capable of heating the specimen to at least 310°C.

4.2 Diamond blade or saw, sanding equipment, or equivalent to provide specimens of the size and edge quality required for Method A

4.3 Scissors, razor blades, or equivalent to provide specimens of size and edge quality for Method B

4.4 Air circulating oven capable of maintaining 105°C ± 2°C

4.5 Dessicator capable of an atmosphere <30% RH at 23°C

4.6 Etching system capable of complete removal of metallic cladding

5 Procedure

5.1.1 Metallic clad specimens shall be tested without the cladding. Etch and dry using appropriate procedures and equipment.

5.1.2 Specimens shall be cut to the specified size using appropriate procedures and equipment to minimize thermal shock and mechanical stress. Method A specimens shall have their edges smooth and burr-free by means of sanding or equivalent (to allow the specimen to rest flat on the mounting stage). Method B specimens shall be rectangular, with their long edges parallel (to ensure good mounting in the film fixture). Method B specimens shall have smooth edges without nicks or tears.

5.1.3 Specimens shall be preconditioned by baking for one hour ± 15 minutes at 105°C, then cooled to room temperature in a dessicator.

5.2 Measurement

5.2.1 Apparatus Set-up

5.2.1.1 Install the Required DMA Clamp

Method A Install and calibrate the DMA with a bending geometry fixture/clamp.

Method B Install and calibrate the DMA with a thin film fixture/clamp.

5.2.1.2 Start the Experiment

Method A Measure the length, width, and thickness of the specimen to within at least +0.01 mm or preferably +0.005 mm. Clamp the specimen in the DMA fixture. Set the sample strain amplitude to operate within the linear viscoelastic range of the material. Strains <1% are recommended and are typically 0.1%. Program the sample temperature range. Enclose the specimen and fixture in the environmental chamber (furnace).

Method B Measure the length, width, and thickness of the specimen to within at least +0.01 mm or preferably +0.005 mm. Sample lengths of 10 mm to 20 mm are typical. Mount the specimen in the clamps of the film fixture according to the manufacturer's instructions. Apply tension force between 10 g and 50 g. A typical base force would be 20 g (see 6.5 for an explanation of the load criteria). Enclose the specimen and probe in the environmental chamber.

5.2.1.3 Provide an inert gas purge (helium or nitrogen) to the environmental chamber. Temperature calibration of the DMA must be performed under the same gas conditions.

5.2.2 Running the DMA Temperature Scan

5.2.2.1 Initial Temperature ($T_{initial}$)

a. For specimens with T_g below or near room temperature, start the scan at least 20°C below the anticipated transition. This may require a DMA with subambitent cooling control of the environmental chamber.

b. For specimens with T_g greater than room temperature, start the scan at 30°C.

5.2.2.2 Sample Heating and Deformation Rate The specimen shall be run at 2°C/min and an oscillation frequency of 1 Hz.

5.2.2.3 Temperature Excursion Heat the specimen to at least 20°C greater than the T_g. This test is general in nature and data may be taken above T_g if required. There is no required upper temperature.

5.3 Evaluation

5.3.1 The DMA storage modulus should resemble the plot shown in Figure 1.

5.3.2 An idealized DMA curve has a linear section below the transition (glassy region below the temperature of T_g) and a stepwise drop through the glass transition region. These linear sections are used in calculating T_g by onset of the modulus drop (see Figure 1).

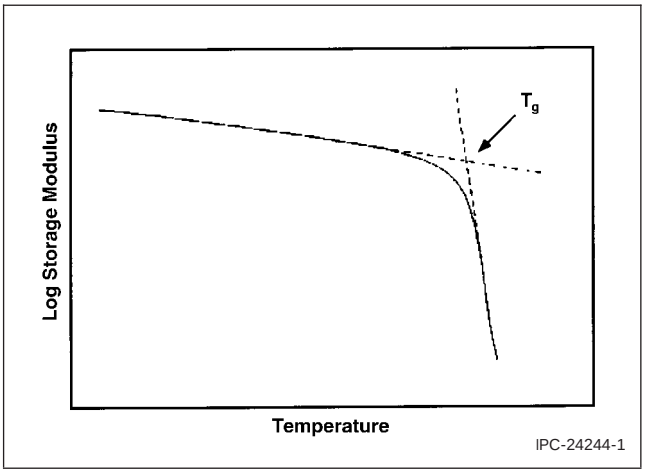

Figure 1 DMA Modulus Plot

5.3.3 Examine all specimens after the test to look for signs of excessive loads, distortions, tears, and other defects. If any defects or sample irregularities are found, discard the sample and the data, rerun another specimen, or pick a different method for determining T_g and storage modulus.

5.4 Calculations

5.4.1 Glass Transition Temperature (T_g) Construct a tangent line to the curve below the transition temperature in the modulus curve. Construct a tangent to the storage modulus curve at or near the inflection point approximately midway through the step change in the transition. The temperature where these tangents intersect is the reported T_g for the material. For consistency it is recommended that the DMA computer analysis software be used for this calculation. See Figure 1 for an example of this tangent intersection method.

5.4.2 Storage Modulus (E') The sample storage modulus (E') shall be calculated at room temperature (22°C) and reported in units of Pa (N/m^2). For consistency it is recommended that the DMA computer analysis software be used for this geometry specific calculation.

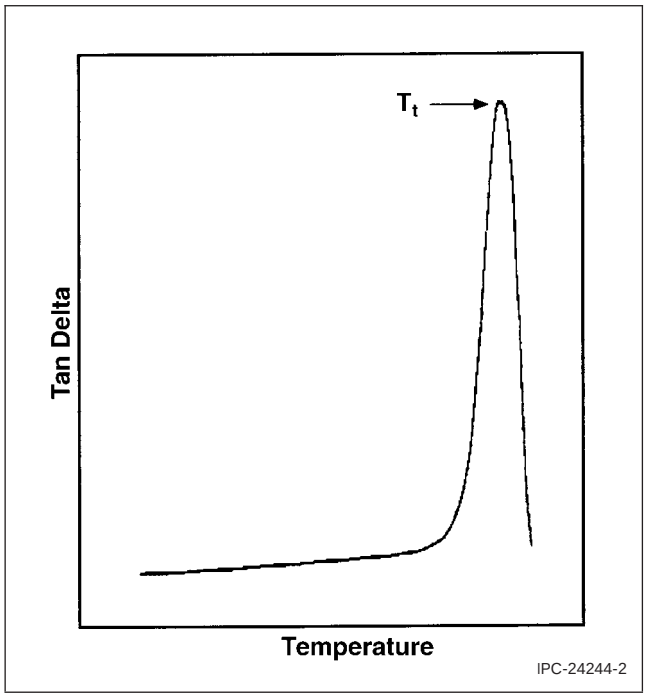

Figure 2 DMA Tan Delta Plot

Note: T_t is the transition peak temperature.

5.4.3 Alternative thermal transitions may be reported as the transition peak temperature in the sample loss modulus (T_l) or tan δ plots (T_t) (see Figure 2 and Figure 3).

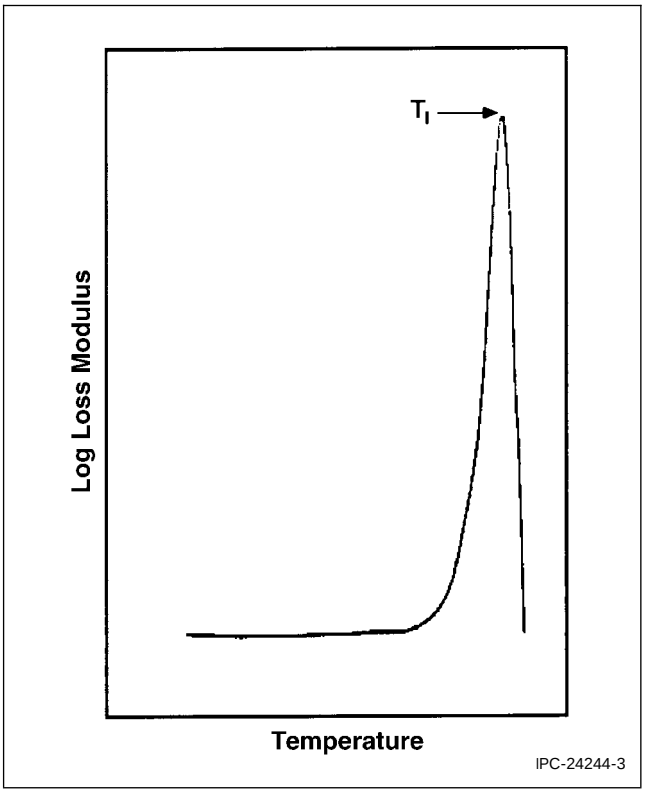

Figure 3 DMA Loss Modulus Plot

Note: T_l is the transition peak temperature.

5.5 Report

5.5.1 Report the glass transition temperature 22°C (room temperature) for each specimen, rounding to the nearest whole number.

5.5.2 Report the modulus in units of Pa (N/m^2) at 22°C.

5.5.3 For anisotropic (reinforced) samples report the both the x and y direction modulus.

5.6 Plot

5.6.1 Plot the storage modulus vs. temperature (°C) for the specimen. If using computer-based analysis, include the T_g

and measurement start and end points and computer generated lines (see Figure 1).

5.6.2 Optionally plot the storage modulus, loss modulus, and tan δ vs. temperature (°C) for the specimen (see Figure 4).

6 ***Notes***

6.1 Calibration of the DMA must be carried out according to the manufacturer's instructions for the relevant sample geometry and thermocouple temperature.

6.2 There are several methods for determining the T_g of organic materials:

- Differential scanning calorimetry (DSC)
- TMA
- DMA

T_g in organic materials is a broad transition, which arises when molecular mobility greatly increases in the specimen as a result of heating. No one method is superior to another; they each measure different physical changes that occur in a specimen near and around T_g.

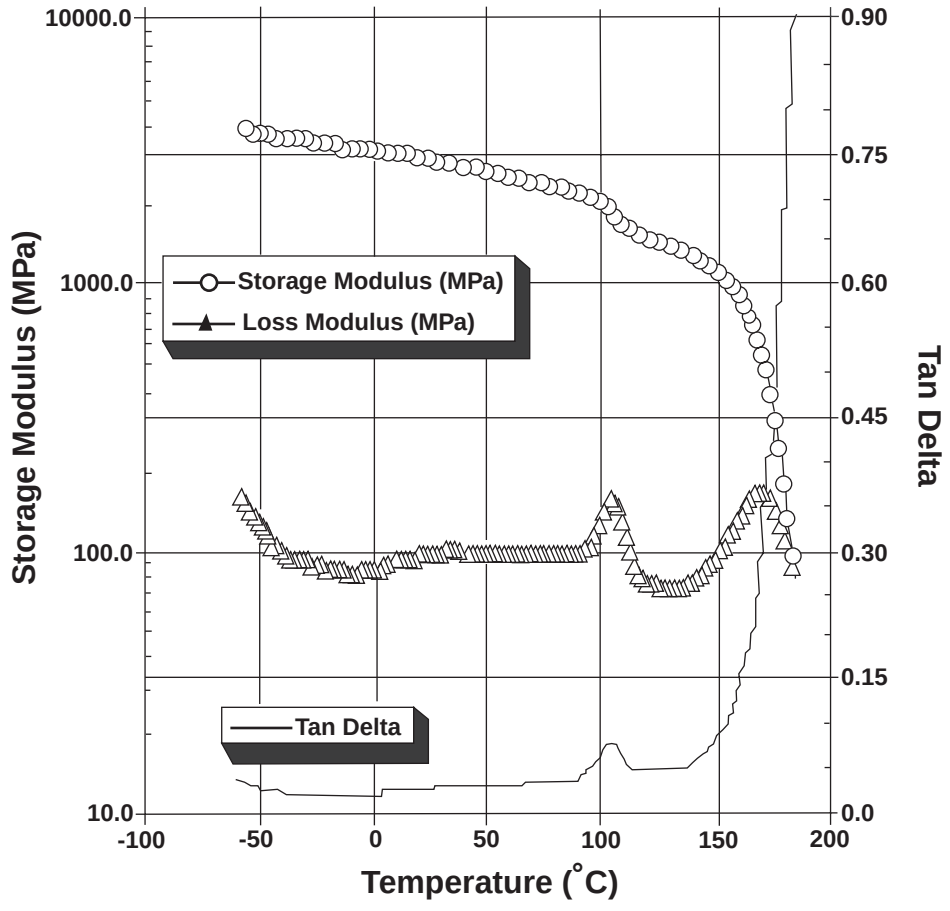

Figure 4 DMA Plot for Storage Modulus, Loss Modulus, and Tan Delta on One Plot

DSC measures the heat capacity of a specimen, TMA measures the expansion of a specimen, and DMA measures the stiffness of the specimen. The T_g determined from TMA, DSC, and DMA may vary significantly (up to 20°C) because they are measuring different physical properties, which change differently as the specimen goes through T_g. As a result, the test equipment used should be noted after the reported T_g value (i.e., 136°C; DSC, TMA, or DMA).

6.3 Most thermal analysis equipment have the software capability to determine sample T_g and modulus values; it is recommended that this approach be used for consistency.

6.4 Load Selection Criteria The initial load should be 5 g of tension (approximately 50 mN). The load (or force) may be adjusted for differences in material types or specimen configuration in order to assure the specimen is being held without slack. Avoid an excessive load (or force), which may result in elongation of the specimen due to the applied tension. Specimens above T_g may become so soft as to be stretched.

Examine all specimens after the test to look for signs of excessive loads, distortions, tears, and other defects.

6.5 Thermal Stresses and Other Anomalies DMA results may be affected by any stresses that might have been frozen into the sample during processing. Samples showing anomalous behavior should be run a second time or preconditioned to remove such stresses. Holding the sample temperature at 20°C above the glass transition and holding for five minutes, followed by slow cooling, will normally remove the stresses in the sample.

6.6 Understanding DMA Refer to ASTM D-4092 for a better understanding of concepts and definitions of terms for dynamic mechanical measurements.

6.7 Instrument Suppliers DMA instruments capable of meeting the requirements of this test method are known to be available from:

TA Instruments
Perkin Elmer Corp.
Seiko Instruments, Inc.
Rheometrics Scientific
Netzsch Instruments, Inc.