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IPC-TM-650
TEST METHODS MANUAL

Number	
2.4.24	
Subject	
Glass Transition Temperature and Z-Axis Thermal Expansion by TMA	
Date	Revision
12/94	**C**
Originating Task Group	
MIL-P-13949 Test Methods Task Group (7-11b)	

1.0 Scope This test is designed to determine the Glass Transition Temperature (T_g) and the Thermal Expansion in the Z-Axis of dielectric materials used in printed boards by the use of thermal mechanical analysis (TMA).

Thermal Expansion (TE) is expressed in Coefficient of Thermal Expansion (CTE) or Percent of Thermal Expansion (PTE).

2.0 Applicable Documents None

3.0 Test Specimens

3.1 Size Specimens shall be approximately 6.35 mm x 6.35 mm [0.25 in x 0.25 in]. The thickness shall be a minimum of 0.51 mm [0.020 in]; for thicknesses less than 0.51 mm [0.020 in], or to increase the accuracy of the test, see 6.4.

3.2 Quantity and Sampling Unless otherwise specified, two specimens shall be tested, to be taken from random locations of the material in question.

4.0 Apparatus or Material

4.1 Thermomechanical analyzer (TMA) capable of determination of dimensional change to within 0.0025 mm [0.0001 in] over the specified temperature range.

4.2 Diamond blade or wheel, sanding equipment, or equivalent, to provide a specimen of the size and edge quality specified.

4.3 Desiccator capable of an atmosphere less than 30% R.H. at 23°C [73.4°F].

4.4 Etching system capable of complete removal of metallic cladding.

4.5 Air circulating oven capable of maintaining 105 ± 2°C [221 ± 3.6°F].

4.6 Micrometer capable of thickness measurements to within 0.00025 mm [0.0001 in].

5.0 Procedure

5.1 Specimen Preparation

5.1.1 Metallic clad laminate shall be tested without the cladding. Specimens taken from multilayer boards shall have no internal metal layers, if possible. Exterior metallic cladding shall be removed by etching using standard industry practices.

5.1.2 Specimens shall be cut to the specified size using appropriate procedures and equipment to minimize mechanical stress or thermal shock.

5.1.3 The edges shall be smooth and burr-free by means of sanding or equivalent (to allow the specimen to rest completely flat on the mounting stage). Use care to minimize stress or heat on the specimen.

5.1.4 Specimens shall be preconditioned by baking for 2 ± 0.25 hours, at 105 ± 2°C [221 ± 3.6°F], then cooled to room temperature in a desiccator.

5.1.5 If applicable, determine the thickness of the specimen (for determination of Percent of Thermal Expansion) and record as T_o.

5.2 Measurement

5.2.1 Mount the specimen on the stage of the TMA and apply a load between 0.1 g and 10.0 g (see note 6.5 for explanation of the load selection criteria).

5.2.2 Initial Temperature for Startup

a. For T_g determination, start the scan at a temperature no higher than 35°C [95°F]. An initial temperature of 23°C [73°F] is recommended.

b. For TE determination start the scan at a temperature sufficiently lower than the specified temperature range such that the specified heat rate is stabilized (see 6.6).

5.2.3 Unless otherwise specified, maintain the scan rate at 10°C [18°F] per minute.

5.2.4 Temperature Excursion

a. For Tg determination, continue the temperature ramp to at least 30°C [54°F] above the anticipated transition region.

b. For TE determination, continue the temperature ramp to

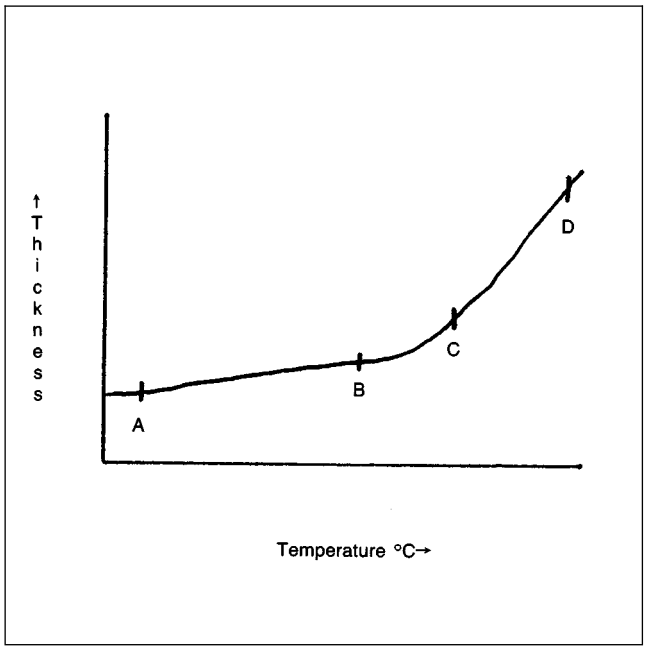

Figure 1

250°C [482°F] or other temperature as specified (such as, representative of a soldering operation).

For example, determination of T_g on a material with an anticipated T_g of 270°C [518°F] would require that the test temperature should reach in excess of 300°C [572°F]. TE measurements should be determined only from that part of the plot reaching 250°C [482°F] (or other temperature of interest).

5.2.5 If residual stresses cause a sudden irreversible deflection at the glass transition, a second scan shall be run, either on the same specimen or if desired, a new specimen.

5.3 Evaluation

5.3.1 The data for the scan should resemble the plot as shown in Figure 1.

5.3.2 From the TMA plot, record the thickness of the specimen as four points: Temperature ''A'' shall be chosen just above room temperature, e.g., 25°C [77°F]. Temperatures ''B'' and ''C'' shall be chosen such that they are on the linear portion of the graph, but just below and above the transition region, respectively. Temperature ''D'' shall be selected to represent a temperature of interest, such as a soldering operation. Unless otherwise specified, Temperature ''D'' shall be 250°C [482°F].

5.4 Calculations

5.4.1 Glass Transition Temperature Determine the point at which lines drawn through points A and B and points C and D will intersect. The temperature at which the tangent lines intersect is the T_g.

5.4.2 Coefficient of Thermal Expansion in the Z-Axis The CTE shall be calculated over the specified regions and recorded in units of ppm/°C.

a. CTE Below the Glass Transition

$$\propto(A - B) = \frac{(t_B - t_A)10^6}{t_A(T_B - T_A)}$$

b. CTE Above the Glass Transition.

$$\propto(C - D) = \frac{(t_D - t_C)10^6}{t_C(T_D - T_C)}$$

c. CTE from Near Room Temperature to 250°C. (Or Other Temperature of Interest)

$$\propto(A - D) = \frac{(t_D - t_A)10^6}{t_A(T_D - T_A)}$$

Where:

T_A = Temperature at point A on plot

T_B = Temperature at point B on plot

T_C = Temperature at point C on plot

T_D = Temperature at point D on plot

t_A = Thickness at T_A

t_B = Thickness at T_B

t_C = Thickness at T_C

t_D = Thickness at T_D

5.4.3 Percent of Thermal Expansion in the z-axis.

5.4.3.1 Select the temperature range over which the expansion in percentage shall be determined. The temperature range from point A to point D is considered most meaningful.

5.4.3.2 The PTE is calculated as follows:

$$\text{Percent TE} = \frac{t_D - t_A}{T_O} \times 100$$

Where

t_o = Initial thickness (see 5.1.5)

t_D = Thickness at Temperature D

t_A = Thickness at Temperature A

5.5 Report

5.5.1 Report the glass transition temperature of each specimen.

5.5.2 Report the TE as CTE in ppm/°C or as PTE in percentage, and the temperature ranges over which the TE has been determined. If specified, report the CTE over the temperature ranges above and below the T_g (A-B and C-D).

5.5.3 Report the scan rate and final TE temperature if other than that specified.

6.0 *Notes*

6.1 Calibration of the TMA must be carried out according to the manufacturer's instructions.

6.2 The Tg for a given material may be significantly different when measured by DSC versus TMA. The test equipment used should be noted after the reported glass transition value, i.e., 136.4° (DSC) or 132.6° (TMA).

6.3 Most thermal analysis equipment have the software capability to determine T_g and CTE values; it is recommended that this approach be used for consistency, provided test parameters (e.g., temperatures, edge smoothing factors, etc.) do not conflict with the procedures specified.

6.4 To improve the accuracy of the test, the thickness should be at least 0.76 mm [0.030 in] and preferably 1.6 mm [0.062 in]. If the material thickness to be measured is less than 0.020 inch, a specimen stack-up to at least 0.51 mm [0.020 in] may be used although the test error probability is greatly increased. A sample of suitable thickness may be prepared from the prepreg used in the manufacture of the base material by laminating and curing as recommended by the supplier. Specimen thickness should not exceed 2.36 mm [0.093 in] to avoid variability from thermal gradients occurring within the specimen.

6.5 Load selection criteria. Initial load is recommended to be 5g. The load should be adjusted for differences in material types or specimen configuration in order to assure intimate contact between the probe, specimen, and stage. Avoid excess load which may result in penetration or distortion of the specimen.

6.6 Initial temperature for starting the scan is determined by an evaluation of the derivative of the time/temperature curve for the equipment. Test data is not valid until the time/temperature curve is stabilized. Refer to operating instruction of the equipment for additional information.

6.7 Desiccator Conditions The Test Methods Task Group determined that a great majority of test laboratories are unable to consistently hold the Relative Humidity in a desiccator to less than 20%. Based on data from participating company lab management, the lowest practically feasible RH for use with the affected IPC Test Methods is 30% maximum.