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IPC-TM-650
TEST METHODS MANUAL

Number	
2.4.24.5	
Subject	
Glass Transition Temperature and Thermal Expansion of Materials Used in High Density Interconnection (HDI) and Microvias - TMA 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 coefficient of thermal expansion (CTE) of dielectric materials used in high density interconnect (HDI) and microvias by the use of thermal mechanical analysis (TMA). For isotropic (unreinforced) materials, either method (A = thick specimen; B = thin specimen) may be used. For anisotropic materials (reinforced), both methods shall be used, since the z axis expansion (Method A) is not the same as the x-y axis expansion (Method B).

2 Applicable Documents None

3 Test Specimens

3.1 Size

Method A Volumetric or Z-axis expansion – thick specimens (>0.50 mm): Specimens shall be approximately 6.5 mm x 6.5 mm. The thickness shall be a minimum of 0.5 mm; for thicknesses <0.5 mm, use Method B. Exact specimen dimensions may be determined by the apparatus used.

Method B In-plane (x-y) expansion – thin specimens (<0.5 mm): Specimens shall be approximately 15 mm to 20 mm long and 2 mm wide, with a minimum thickness of 10 µm and a maximum thickness of 0.75 mm. 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 For Method B, two samples are to be measured, taken at 90° to each other and labeled in the x and y directions. Isotropic materials are anticipated to have the same CTE for x and y, and reinforced materials are likely to have differing x and y CTE.

4 Equipment/Apparatus

4.1 A TMA capable of determination of dimensional change to within 0.0025 mm over the specified temperature range. Preferably the TMA will have computer data acquisition and analysis. The TMA must have an environmental chamber capable of having nitrogen flush gas and heating of the specimen to 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 or 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 less than 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 Correct TMA Probe

Method A Set up the TMA with a non-penetrating quartz expansion probe.

Method B Set up the TMA with a thin film fixture/clamp.

5.2.1.2 Apply the Load

Method A Mount the specimen on the stage of the TMA and apply load at 5 g (see 6.5 for an explanation of load criteria). Enclose the specimen and probe in the environmental chamber.

Method B Mount the specimen in the clamps of the film fixture according to the manufacturer's instructions and apply 2 g tension force (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) at a rate of 30 ml/min to 150 ml/min to the environmental chamber. Temperature calibration of the TMA must be performed under the same gas conditions.

5.2.1.4 Measure the inital specimen thickness (Method A) or length (Method B) prior to each heat cycle (L_O).

5.2.2 Many specimens have built in thermal stresses from the curing step, which relaxes during the specimen heating during a TMA test. This relaxation results in TMA scans, which make determination of T_g and CTE impossible (see Figure 1). Two heat cycles are required to obtain valid T_g and CTE values.

5.2.3 Running the TMA Temperature Scan

5.2.3.1 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 TMA with refrigeration control of the environmental chamber.

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

5.2.3.2 Temperature Rate Depending on sample preparation, two heating cycles may be required to obtain accurate T_g and CTE above T_g. If the sample shows unexpected shrinkage above T_g (see Figure 1), the two heat test method is required. If the sample does not show anomalous behavior, only one heat cycle (the second heat cycle at 5°C/min) is required.

a. First heat: The first heat cycle of the specimen shall be run at 10°C/min.

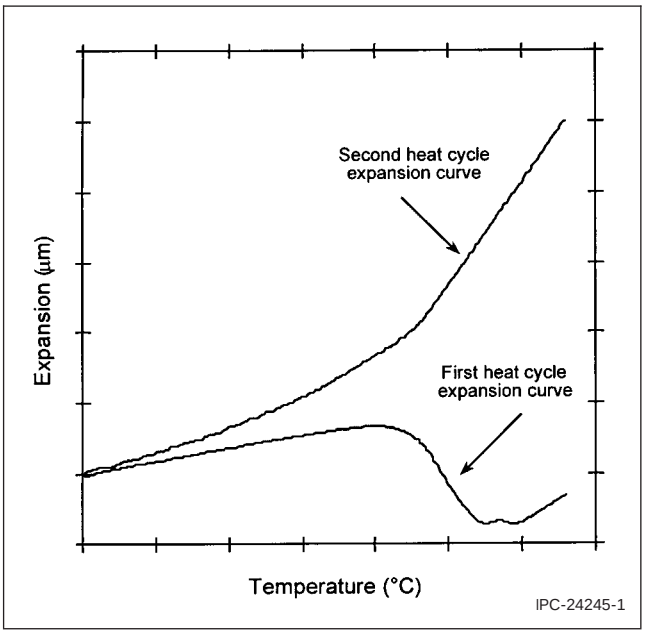

Figure 1 TMA Expansion Curves: First Heat Cycle and Second Heat Cycle

b. Second heat (reportable data heat cycle): The second heat cycle of the specimen shall be run at 5°C/min.

5.2.3.3 Temperature Excursion

a. First heat: Continue heating the specimen to a temperature 20°C greater than the anticipated T_g or until the anomalous thermal relaxation has stopped. See Figure 1 for an example of anomalous first heat behavior. Hold the specimen at this temperature for a minimum of five minutes. Avoid holding the sample at this temperature for too long; sample degradation might occur. Cool the specimen to the initial temperature under temperature control at 5°C/min to 10°C/min. This should prevent reestablishment of thermal stresses.

b. Second heat (reportable data heat cycle): The second heat cycle of the specimen shall continue to 310°C (to ensure good data at 300°C).

5.3 Evaluation

5.3.1 The TMA expansion curve should resemble the plot shown in Figure 2.

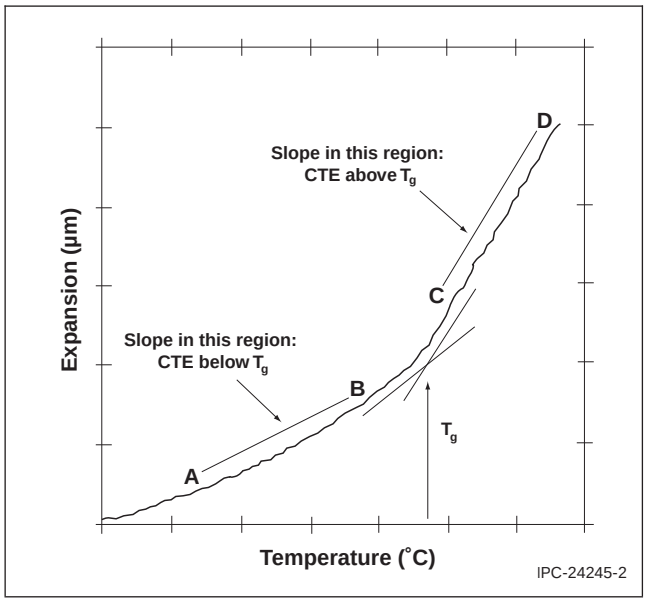

Figure 2 TMA Expansion Curve

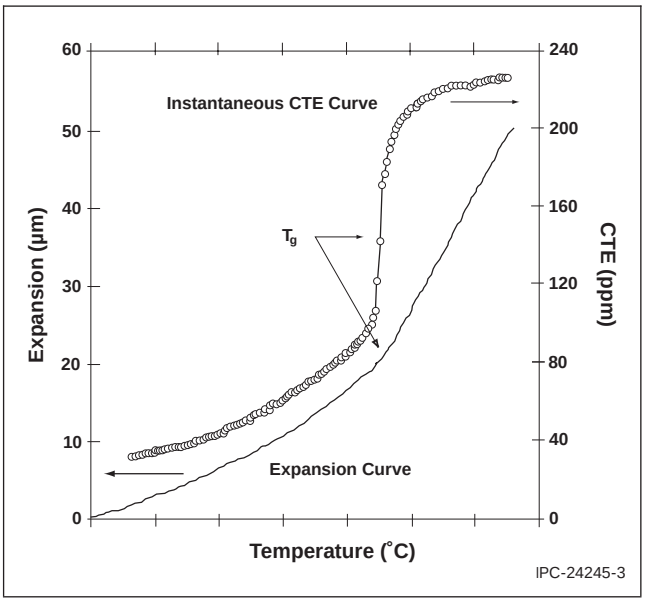

Figure 3 TMA Expansion Curve and Instantaneous CTE Curve

5.3.2 An idealized TMA curve has a linear section below the transition (expansion below T_g) and a linear section above the transition (expansion above T_g). These linear sections are used in calculating the T_g and CTE of the material.

With real samples, these ''linear'' sections are often curved so the standard CTE calculation (see 5.4.2) is the average CTE between the defined points (A-B and C-D in Figure 2). The instantaneous CTE provides CTE as a function of temperature and avoids this averaging effect (see Figure 3).

5.3.3 From the TMA plot, pick four temperatures and obtain the specimen thicknesses at these temperatures:

T_A – at least 10°C above $T_{initial}$ (to ensure thermal equilibrium) and no higher than 25°C above $T_{initial}$
T_B – on the linear portion of the graph below the T_g
T_C – on the linear portion of the graph above T_g
T_D – 300°C

Preferred temperatures for HDIS materials:
$T_{initial}$ = 30°C
T_A = 40°C
T_B = material dependent - below the T_g
T_C = material dependent - above T_g
T_D = 300°C

5.3.4 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 CTE.

5.4 Calculations

5.4.1 Glass Transition Temperature – T_g Construct a tangent line to the curve above and below the transition in the curve. The temperature where these tangents intersect is the TMA determined T_g for the material. If the tangent method fails to provide an adequate T_g, the instantaneous CTE can be calculated (see 5.4.3) and the midpoint of the step change in CTE may be taken as T_g (see Figure 3). For consistency, it is recommended that the TMA computer analysis software be used for this calculation (see Figure 2).

5.4.2 Mean Coefficient of Thermal Expansion – CTE The mean CTE shall be calculated over the specified regions and recorded in units of ppm/°C. For consistency it is recommended that the TMA computer analysis software be used for this calculation.

a. CTE below glass transition:

$$\alpha_{(B-A)} = \frac{(L_B - L_A)10^6}{L_o(T_B - T_A)}$$

For most materials, this will be in the range of 7 ppm to 50 ppm (reinforced) or 30 ppm to 150 ppm (unreinforced).

b. CTE above glass transition:

$$\alpha_{(D-C)} = \frac{(L_D - L_C)10^6}{L_o(T_D - T_C)}$$

For most materials, this will be in the range of 50 ppm to 100 ppm (reinforced) or 150 ppm to 500 ppm (unreinforced). Any reinforced materials, where the reinforcement has a negative CTE, will shrink rather than expand when heated above T_g of the resin.

Where:

T_A = Temperature at point A in Figure 2
T_B = Temperature at point B in Figure 2
T_C = Temperature at point C in Figure 2
T_D = Temperature at point D in Figure 2
L_0 = Initial Length or thickness
L_A = Length or thickness at point A in Figure 2
L_B = Length or thickness at point B in Figure 2
L_C = Length or thickness at point C in Figure 2
L_D = Length or thickness at point D in Figure 2

5.4.3 Instantaneous Coefficient of Thermal Expansion Curve (Optional) The instantaneous CTE expansion curve is the slope of the TMA expansion curve plotted as a function of temperature. Figure 3 shows a combined expansion curve and its resulting instantaneous CTE curve.

Instantaneous CTE (α_{Ti}) is calculated at each temperature (T_i) from the slope of the TMA expansion curve (dL_i/dT) at that temperature:

$$\alpha_{Ti} = \frac{1}{L_o}\left(\frac{dL_i}{dT}\right)$$

dL/dT is determined at each temperature (T_i) from the L vs. T curve by:

$$\left(\frac{dL_i}{dT}\right) = \frac{(L_{i+1} - L_i)}{(T_{i+1} - T_i)}$$

This calculation can be done in a spreadsheet that contains the L vs. T data. Some TMA computer analysis software performs this calculation for you. For an example of plot $\alpha\eta_{Ti}$ vs temperature, see Figure 3.

5.4.4 Percent Thermal Expansion (PTE) (Optional) The total percent of thermal expansion is calculated as follows:

$$\text{Percent TE} = \frac{(T_D - T_A)}{L_o} * 100$$

For consistency, it is recommended that the TMA computer analysis software be used for this calculation.

5.5 Report

5.5.1 Report the glass transition temperature of each specimen, rounding to the nearest whole number.

5.5.2 Report the CTE in ppm/°C above and below T_g and the temperature ranges over which the thermal expansion was determined. For Method B, report x and y CTE values.

5.5.3 Optionally report the PTE in percent and the temperature ranges over which the thermal expansion was determined.

5.6 Plot

5.6.1 Plot the expansion (µm) vs. temperature (°C) for the specimen. If using computer based analysis, include the T_g and CTE measurement start points and computer generated lines (see Figure 2).

5.6.2 Optionally plot the instantaneous CTE (µm/°C) vs. temperature (°C) for the specimen (see Figure 3).

5.6.3 Optionally plot the percent expansion vs. temperature (°C) for the specimen. If using computer-based analysis, include the PTE measurement start points on the plot.

6.0 *Notes*

6.1 Calibration of the TMA must be carried out according to the manufacturer's instructions for both probe expansion and specimen temperature.

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

- 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.

DSC measures the heat capacity of a specimen. TMA measures the expansion of a specimen and DMA (dynamic mechanical analysis) measures the stiffness of the specimen. The T_g determined from TMA, DSC, and DMA may vary significantly (up to 10°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 T_g and CTE values; it is recommended that this approach be used for consistency.

6.4 To improve the accuracy of this test:

Method A Increase the thickness to 0.76 mm or higher. Do not stack single layers of thinner materials to achieve the minimum thickness; this greatly increases test error.

Method B Be sure the specimen is mounted in the clamps correctly and that any clamp expansion is corrected properly by calibration of the probe. Method B clamps and specimens may have more difficulty reaching thermal equilibrium.

6.5 Load Selection Criteria

Method A The initial load is 2 g. The load may be adjusted for differences in material types or specimen configuration in order to assure intimate contact between the probe, specimen, and stage. Avoid an excess load (15 g), which may result in penetration or distortion of the specimen.

Method B The initial load is 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 that 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.