



IPC-TM-650 TEST METHODS MANUAL

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 μ m 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.

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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 $\pm$ 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 $\pm$ 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.

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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 initial specimen thickness (Method A) or length (Method B) prior to each heat cycle (L_0).

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 ($T_{initial}$)

- 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.
- 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.

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

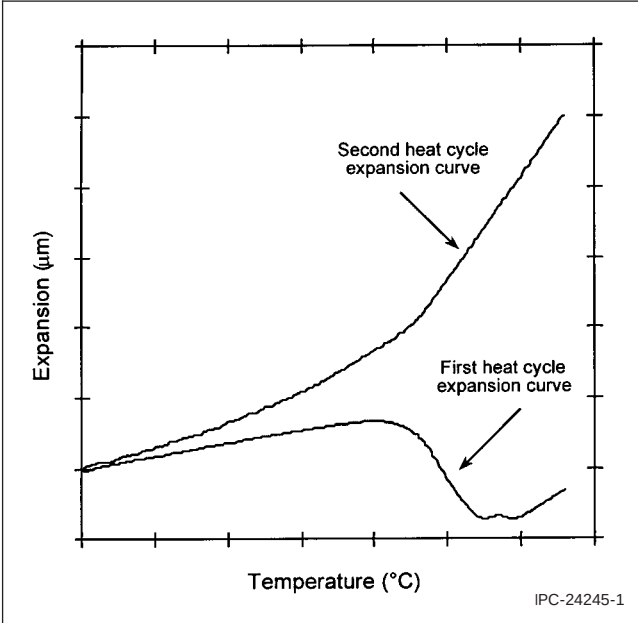


Figure 1 TMA Expansion Curves: First Heat Cycle and Second Heat Cycle

- 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

- 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.
- 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.