Association Connecting Electronics Industries

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Number **2.4.25**	
Subject **Glass Transition Temperature and Cure Factor by DSC**	
Date **11/17**	Revision **D**
Originating Task Group **Laminate/Prepreg Materials Subcommittee (3-11)**	

IPC-TM-650
TEST METHODS MANUAL

1 Scope This test method is designed to determine the glass transition temperature of dielectric materials used in printed boards by differential scanning calorimetry (DSC). It is suitable for prepreg, metallic clad or unclad laminate, and printed boards. It also provides a determination of relative degree of cure, or Cure Factor, for some types of materials.

2 Applicable Documents None

3 Test Specimens

3.1 Size and Configuration The specimen **shall** be a solid piece weighing between 10 to 40 mg. For very thin materials, multiple pieces may be used to achieve the specified weight. The specimen **shall** be of a size and configuration that fits within the sample pan of the DSC equipment. See 6.1 regarding use of a powdered specimen.

3.2 Quantity and Sampling The sampling **shall** be randomly taken from the material in question, and, unless otherwise specified, one specimen **shall** be tested, to be taken from the material in question.

4 Equipment/Apparatus

4.1 Differential scanning calorimeter capable of measuring and recording heat capacity of the applicable material.

4.2 Nitrogen gas supplied at a constant rate, suitable for purging and calibrating the DSC cell.

4.3 Equipment suitable for specimen preparation in accordance with 3.1, such as a punch press.

4.4 Standard aluminum sample pans and lids and crimping press.

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

4.6 Desiccator or drying cabinet capable of maintaining an atmosphere less than 30% RH at 23 ° C [73.4 °F].

5 Procedure

5.1 Specimen Preparation

5.1.1 Metallic clad laminates and printed boards **shall** be tested with metallic cladding left in place whenever possible.

5.1.2 For all laminates and printed boards, the sample **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 or drying cabinet for at least 1/2 hour prior to testing.

5.1.3 The specimen **shall** be prepared from the baked sample in accordance with 3.1. Edges **shall** be smoothed and burrs removed by light sanding, or equivalent, to achieve proper thermal conduction. Use care to minimize stress or heating of the specimen.

5.1.4 Place the specimen in a standard aluminum sample pan with an aluminum lid. Use of a lid and crimping is optional. For referee purposes, a cover lid crimped onto the sample pan **shall** be used. If the specimen is a powder, the pan **shall** be covered with a lid and crimped shut.

5.1.5 For referee purposes, a suitable reference **shall** be prepared by adding an equivalent weight of aluminum lids to the reference pan to match the weight of the sample. For example, if the sample weight is 8 mg, enough lids should be added to the reference pan to weigh 8 mg.

5.2 Test

5.2.1 Follow start up and operating procedures in accordance with instructions supplied by the test equipment manufacturer.

5.2.2 Conditioning Pre-Scan:

5.2.2.1 The point of the pre-scan is to erase previous thermal history and the effects on morphology of the sample.

5.2.2.2 Perform the pre-scan from at least 30 °C [54 °F] below the transition region to a temperature 10 °C [18 °F] above the transition region at a rate of 20 °C/min [36 °F/min]. Then quench-cool to at least 30 °C [54 °F] below the transition region, as rapidly as possible.

5.2.3 Analysis Scan.

5.2.3.1 Start the scan at a temperature that is at least 30 °C [54 °F] lower than the anticipated transition region. The heat rate **shall** be stabilized before the transition region is reached.

5.2.3.2 Unless otherwise specified, scan at a rate of 20 °C/min [36 °F/min].

5.2.3.3 When the transition has been observed, scan at least 30 °C [54 °F] beyond the transition region.

5.2.3.4 Record the results as T_{g1}.

5.2.4 Determination of Cure Factor.

5.2.4.1 The following steps **shall** be performed only if the Cure Factor is applicable and required by the governing specification (see Table 1). It does not apply to prepreg.

5.2.4.2 Continue the scan at a rate of 20 °C/min [36 °F/min] to a temperature per Table 1. The specimen is then held at the isothermal temperature for a time per Table 1.

5.2.4.3 The specimen is immediately cooled to initial conditions and a second glass transition scan carried out in accordance with 5.3.3. Record as T_{g2}.

5.3 Calculation

5.3.1 Determination of T_g The midpoint temperature T_m (°C) as described in Figure 1 reported as the T_g. T_g is the point on the thermal curve corresponding to 1/2 the heat flow difference between the extrapolated onset and extrapolated end. If suitable computer software is available, the automatic calculation of the glass transition temperature is allowable provided the value calculation is either the midpoint or the steepest deflection and not the onset temperature. See Figure 1.

Table 1 Resin Type Temperature Requirements

Resin Type	Isothermal[1] Temperature	Hold Time at Temperature
Difunctional and Tetrafunctional Epoxies	175 °C ± 2 °C	15 ± 0.5 minutes
Multifunctional and High Temperature Epoxies	190 °C ± 5 °C	15 ± 0.5 minutes
BT- Epoxies[2]	N/A	N/A
Polyimides[2]	N/A	N/A
Cyanate Esters[2]	N/A	N/A

1) Or in accordance with manufacturer's recommendations.

2) Certain materials are not compatible with the Cure Factor determination, as they will exhibit an increasing transition temperature with each exposure to a temperature above the cure level.

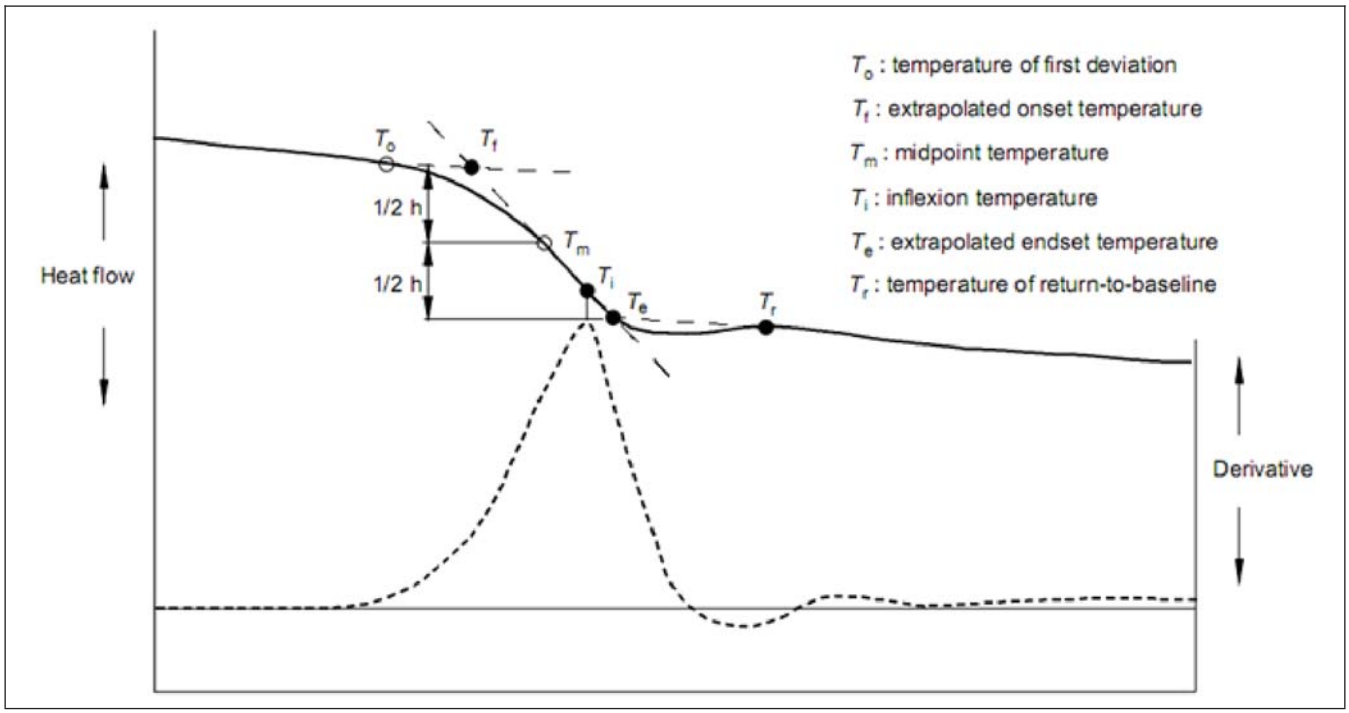

Figure 1 Typical DSC Plot

5.3.2 Determination of Cure Factor (Delta T_g) Cure Factor (or Delta T_g) is the absolute difference between the glass transition temperatures determined in the two scans, where:

Cure Factor (Delta T_g) = T_{g2} - T_{g1}

T_{g1} = T_g of first scan

T_{g2} = T_g of second scan

5.4 Report

5.4.1 The glass transition temperature (delta T_g) **shall** be reported for each specimen.

5.4.2 The Cure Factor **shall** be reported, if applicable, and specified for each specimen.

5.4.3 The scan rate, specimen preparation, isothermal temperature, hold time, and method of midpoint determination **shall** be reported if other than that specified in this method.

5.4.4 The specimen size, configuration, and preparation **shall** be reported.

6 *Notes*

6.1 Powdered Specimens Certain materials may be more appropriately tested using a specimen that is a powder prepared by grinding or filing the sample. Consult with the equipment's instructions and with the material manufacturer for more information.

6.2 Calibration of the instrument **shall** be carried out according to the manufacturer's instructions with at least one standard being indium.

6.2.1 Computer Determination of T_g If suitable computer software is available, the automatic calculation of the glass transition temperature is allowable, provided the value calculated is either the midpoint or the point of steepest deflection. and not the onset temperature.

Calibration of the instrument must be carried out according to the manufacturer's instructions, with at least one standard being indium.

6.3 The glass transition for a given material will be significantly different if measured by DSC versus TMA. The test equipment used should be noted beside the glass transition valve, i.e., 136.4 °C (DSC) or 132.6 °C (TMA).

6.4 Cure Factor is also described as Delta T_g.

6.5 Testing of single-sided or unclad laminates manufactured without metallic cladding on either side.

6.5.1 Single-sided or unclad laminates exhibit unreliable Cure Factor data, due to effects of moisture and other factors. It is recommended that Cure Factor requirements not be applied to these laminate configurations.

6.5.2 Single-sided or unclad laminates typically exhibit T_g approximately 8 °C to 15 °C lower than equivalent laminates that are clad on both sides. Accordingly, the specification requirements should take this into consideration. Reasons for the T_g ''loss'' include presence of moisture in the release films used in place of metallic cladding.