



# IPC-TM-650 TEST METHODS MANUAL

**1.0 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.

**2.0 Applicable Documents** None

## 3.0 Test Specimens

**3.1 Size and Configuration** The specimen shall be a solid piece weighing between 15 and 25 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.

## 4.0 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 punch press .

**4.4** Standard aluminum sample pans and lids, and crimping press.

**4.5** Air circulating oven capable of maintaining  $105 \pm 2^{\circ}\text{C}$  [ $221 \pm 3.6^{\circ}\text{F}$ ].

**4.6** Desiccator capable of maintaining an atmosphere less than 30% RH at  $23^{\circ}\text{C}$  [ $73.4^{\circ}\text{F}$ ].

## 5.0 Procedure

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Subject <b>Glass Transition Temperature and Cure Factor by DSC</b>	
Date <b>12/94</b>	Revision <b>C</b>
Originating Task Group <b>MIL-P-13949 Test Methods Task Group (7-11b)</b>	

## 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  $2 \pm 0.25$  hours at  $105 \pm 2^{\circ}\text{C}$  [ $221 \pm 3.6^{\circ}\text{F}$ ], then cooled to room temperature in a desiccator for at least 1/2 hour prior to testing.

**5.1.3** 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.

**5.2** 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.

## 5.3 Test

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

**5.3.2** Start the scan at a temperature that is at least  $30^{\circ}$  lower than the anticipated onset of  $T_g$ . The heat rate shall be stabilized before the onset temperature is reached.

**5.3.3** Unless otherwise specified, scan at a rate of  $20^{\circ}\text{C}/\text{min}$  [ $36^{\circ}\text{F}/\text{min}$ ].

**5.3.4** When the transition has been observed, scan at least  $30^{\circ}\text{C}$  [ $54^{\circ}\text{F}$ ] beyond the transition region.

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**5.3.5** The following steps shall be performed only if Cure Factor is applicable and required by the governing specification (see Table 1). It does not apply to prepreg. See 6.7.

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

**5.3.5.2** The specimen is immediately cooled to initial conditions and a second glass transition scan carried out in accordance with 5.3.2 through 5.3.4.

**5.4 Determination of  $T_g$**  The glass transition temperature is determined by a construction procedure on the heat flow curve.

**5.4.1** Construct a tangent line to the curve above the transition region and a second tangent line to the curve below the transition region.

**5.4.2** The temperature on the curve halfway between the two tangent lines, or 1/2 delta Cp, is the  $T_g$ .

#### **5.5 Determination of Cure Factor (Delta $T_g$ )**

**5.5.1** Cure Factor (or delta  $T_g$ ) is the absolute difference between the glass transition temperatures determined in the two scans, where:

$$CF (\text{Delta } T_g) = T_{gF} - T_{gI}$$

$$T_{gI} = \text{Initial } T_g$$

$$T_{gF} = \text{Final or Second } T_g$$

#### **5.6 Report**

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

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

**5.6.3** 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.

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

#### **6.0 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 Determination of $T_g$**

**6.2.1** Determination of  $T_g$  by midpoint. (To be determined)

**6.2.2 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.

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

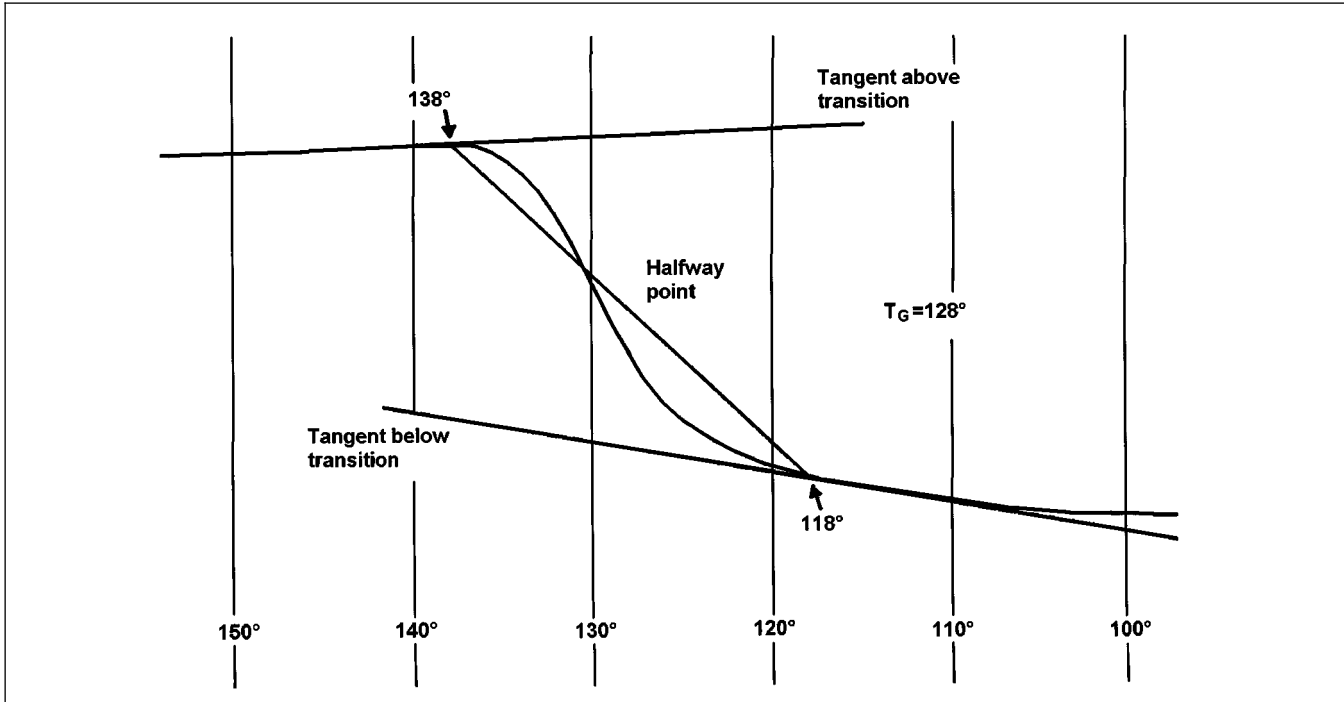
**Table 1**

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

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**Figure 1**

**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 value, i.e., 136.4°C (DSC) or 132.6°C (TMA).

**6.4** Cure Factor is also described as Delta T<sub>g</sub>.

**6.5** Some DSC curves exhibit spikes in the plot either just prior to, or after, the transition region. These events are due to anomalies of the material unrelated to the T<sub>g</sub> such as stress relaxation or moisture. No changes to the construction procedure (see 5.4) should be made in reaction to such deflections. Alternately, the cell may be quench-cooled and the procedure restarted. Such deflections will usually disappear with no other effect on the curve. Report this restart with the test result.

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

**6.6.1** 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.

**6.6.2** Single-sided or unclad laminates typically exhibit T<sub>g</sub> approximately 8° to 15°C lower than equivalent laminates that are clad on both sides, which specification requirements should take into account. Reasons for the T<sub>g</sub> "loss" include presence of moisture in the release films used in place of metallic cladding.