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IPC-TM-650
TEST METHODS MANUAL

Number	
2.4.24.3	
Subject	
Glass Transition Temperature of Organic Films – TMA Method	
Date	Revision
7/95	
Originating Task Group	
Deposited Dielectric Task Group (C-13a)	

1.0 Scope This test method establishes a procedure for determining the glass transition temperature of organic films using thermal mechanical analysis (TMA).

2.0 Applicable Documents

ASTM D 618 Standard Practice for Conditioning Plastics and Electrical Insulating Materials for Testing

3.0 Test Specimen The test specimen shall consist of a strip 15-20 mm long and 2 mm wide with a minimum thickness of 10 μm.

4.0 Apparatus or Material Perkin-Elmer TMA-7 with a film fixture in extension mode or equivalent equipment capable of handling films less than 25 μm thick.

5.0 Procedure

5.1 The test specimens should be conditioned at 23 ± 2°C and 50 ± 5% relative humidity for not less than 24 hours prior to testing. Refer to ASTM D 618.

5.2 Follow the manufacturer's recommendations for equipment startup and calibration.

5.2 Mount the test specimen in the film holder. The sample length (between the grips) should be between 11-13 mm.

5.3 Set the force at 30 mN.

5.4 Perform a prescan by heating at a rate of 20°C/min under inert atmosphere from ambient to 50°C beyond the apparent completion of the thermal activity to erase previous thermal history.

5.5 Hold the temperature for 10 min.

5.6 Cool to 50°C below the transition temperature observed in the prescan.

5.7 Hold the temperature for 10 min.

5.8 Reheat the specimen at a rate of 5°C/min until all desired transitions have been completed.

5.9 The glass transition is determined by a construction procedure on the transition region of the extension versus temperature curve (Figure 1).

5.10 Construct a tangent line to the curve above and below the transition.

5.11 The temperature at which the tangents intersect is the glass transition temperature.

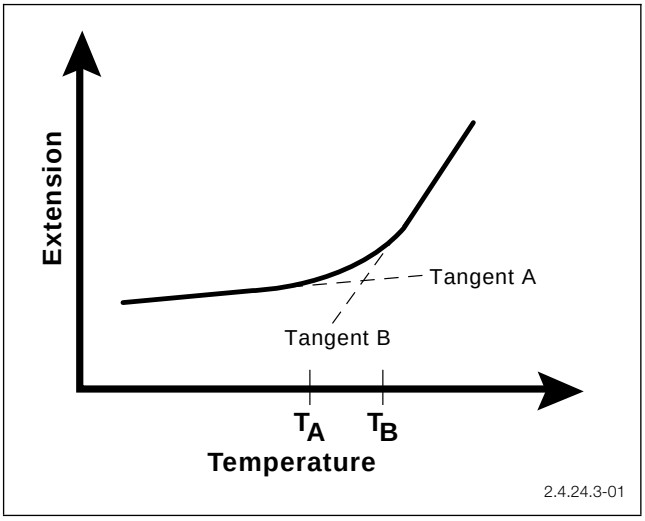

Figure 1

5.12 Report both the glass transition (intersection of tangents), e.g., 200°C (TMA-5°C/min), and the temperatures at the beginning of tangent A (T_A) and tangent B (T_B) (i.e., the transition range), e.g., transition range: 160-205°C.

6.0 Notes

6.1 Calibration of the instrument must be carried out according to the manufacturer's recommendations. Two calibrations are required, one to establish the baseline and the other to calibrate the TMA relative to a standard.

6.2 A quartz specimen of 11-13 mm in length (between the grips) is run at 5°C/min under inert gas purge (He) from –20 to 400°C to establish a baseline. The baseline is used to eliminate the effects of grip expansion on extension measurements. The coefficient of linear thermal expansion of quartz is 0.57×10^{-6}/°C (16-500°C)[1].

6.3 After the baseline is established, the TMA must be calibrated with at least one standard being 99.9999% pure aluminum which has a linear CTE of 24.9×10^{-6}/°C from 0-250°C. An aluminum specimen is run between -10 and 200°C and the coefficient of linear thermal expansion is calculated. If the measured value differs from the literature value, the specimen size is adjusted to correspond to the measured value, and the specimen is rerun. Once the measured and literature values are in agreement, this constant factor is used on subsequent specimen sizes.

6.4 The glass transition temperature for a given material will be significantly different depending on the method of analysis (i.e., DMA, DSC, or TMA). The glass transition determined by DMA is frequency dependent and increases with increasing frequency. The glass transition determined by DSC or TMA will depend on the heating rate. The test method used along with the frequency (DMA) or heating rate (DSC or TMA) should be noted beside the glass transition value, e.g., 135°C (DMA-10 Hz) or 141°C (DSC-5°C/min).

6.5 In general, DMA is more sensitive that DSC or TMA. This is especially important for high temperature polymers with weak transitions.

1. Lange's Handbook of Chemistry, 12th edition, J. A. Dean, ed., McGraw-Hill, New York (1979).