



IPC-TM-650 TEST METHODS MANUAL

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

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 22.5 mm long and 6.25 mm wide with a minimum thickness of 5 μm . The analysis is based on the assumption of a constant specimen geometry, therefore the test specimens must be stiff enough not to plastically deform during the experiment.

4.0 Apparatus or Material Rheometrics Solids Analyzer Model RSA-II with a film/fiber fixture or equivalent.

5.0 Procedure

5.1 The test specimens should be conditioned at $23 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ 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.

5.3 Mount the specimen in the film/fiber fixture. Make certain that the specimen is mounted perpendicular to the clamps. Hand tighten the clamps as much as possible to prevent specimen slippage during a run.

5.4 Operate at a frequency of 1 Hz (6.28 radians/sec). Heat the specimen in dry nitrogen at a rate of no faster than $2^\circ\text{C}/\text{min}$., or in steps of 5°C increments, in dry air.

5.5 When the transition has been observed, heat at least 50°C beyond the apparent completion of the thermal activity before returning to initial conditions.

5.6 The glass transition temperature is defined as the temperature corresponding to the maximum in the $\tan \delta$ vs. temperature curves at a frequency of 1 Hz. $\tan \delta$ is calculated from

$$\tan \delta = E''/E'$$

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where E'' is the loss modulus and E' is the storage modulus. A typical plot is shown in Figure 1.

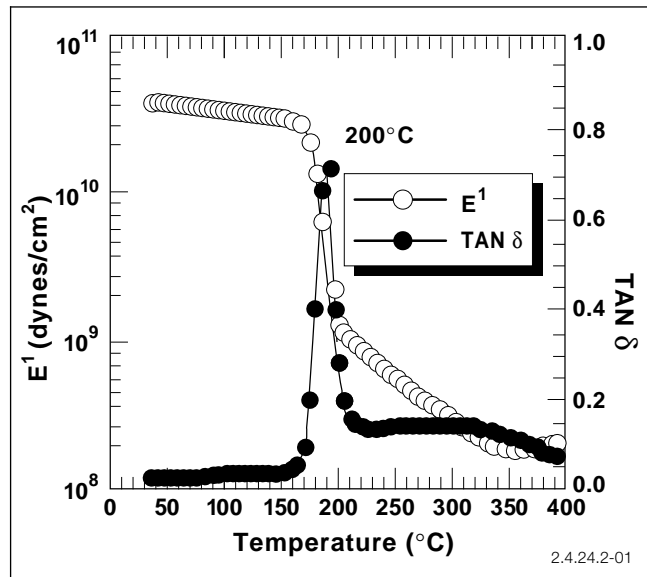


Figure 1

5.7 Report both the glass transition (maximum in $\tan \delta$), e.g., 200°C (DMA-1 Hz), and the temperature range over which the storage modulus (E') changes (i.e., the transition range), e.g., transition range: $160\text{-}205^\circ\text{C}$.

6.0 Notes

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

6.2 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-1 Hz) or 141°C (DSC- $5^\circ\text{C}/\text{min}$).

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

6.4 If the polymer decomposes before the glass transition is reached, report the decomposition temperature and indicate that it is a decomposition temperature and not a glass transition temperature.