Designation: E1824 – 09$^{\varepsilon 1}$

Standard Test Method for Assignment of a Glass Transition Temperature Using Thermomechanical Analysis: Tension Method[1]

This standard is issued under the fixed designation E1824; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Added research report information to Section 13 editorially in September 2010.

1. Scope

1.1 This test method covers a procedure for the assignment of a glass transition temperature of materials on heating using thermomechanical measurements.

1.2 This test method may be used as a complement to Test Method E1545 and is applicable to amorphous or to partially crystalline materials in the form of films, fibers, wires, etc. that are sufficiently rigid to inhibit extension during loading at ambient temperature.

1.3 The generally applicable temperature range for this test method is −100 to 600°C. This temperature range may be altered depending upon the instrumentation used.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 There is no ISO method equivalent to this method.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology
E1142 Terminology Relating to Thermophysical Properties
E1545 Test Method for Assignment of the Glass Transition Temperature by Thermomechanical Analysis
E1970 Practice for Statistical Treatment of Thermoanalytical Data
E2602 Test Method for the Assignment of the Glass Transition Temperature by Modulated Temperature Differential Scanning Calorimetry

3. Terminology

3.1 *Definitions:*

3.1.1 The following terms are applicable to this test method and can be found in Terminology E473 and Terminology E1142: *thermomechanical analysis (TMA), thermodilatometry, glass transition, glass transition temperature*.

4. Summary of Test Method

4.1 This test method uses thermomechanical analysis equipment (thermomechanical analyzer, dilatometer, or similar device) with the test specimen in tension to determine the change in dimension of a thin specimen observed when the material is subjected to a constant heating rate through the glass transition region. This change in dimension associated with the change from vitreous solid to amorphous liquid is observed as movement of a sensing probe in direct contact with the specimen and is recorded as a function of temperature. The intersection of the extrapolation of the slope of the probe displacement curve before and after the transition is used to determine a temperature that is assigned as the glass transition temperature.

5. Significance and Use

5.1 The glass transition is dependent on the thermal history, softening agents or additives of the material to be tested. For amorphous and semicrystalline materials the assignment of a glass transition temperature may lead to important information about thermal history, processing conditions, stability, progress of chemical reactions, and mechanical and electrical behavior.

5.2 Thermomechanical analysis provides a rapid means of detecting changes in hardness or linear dimensional change associated with the glass transition. Dimensional changes measured as a specimen is heated over the T_g region may include the interaction of several effects: an increase in the coefficient of expansion, a decrease in the modulus, which

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

under a constant stress leads to increased extension, stress relief leading to irreversible dimensional change (shrinkage in one dimension, expansion in another dimension), and physical aging effects which change the kinetics of the dimensional change.

5.3 This test method is useful for research and development, quality control, and specification acceptance testing; particularly of films and fibers.

6. Interferences

6.1 This test method may be used for materials having a glass transition at or below ambient temperature providing care is taken to avoid exposing the specimen to a tensile force prior to cooling the specimen below its glass transition. Applying a tensile load on a specimen that is above its glass transition will result in elongation of the specimen which may introduce orientation and residual stresses that will alter the specimen thermal history and may yield erroneous results during the heating cycle.

6.2 Specimens of thickness less than 0.2 mm may be difficult to handle.

6.3 Specimens of thickness greater than 5 mm may develop temperature nonuniformities of sufficient extent as to yield erroneously high values for an assigned glass transition temperature using this test method.

7. Apparatus

7.1 The essential equipment required to provide the minimum instrument capability for this test method includes:

7.1.1 A *Thermomechanical Analyzer (TMA) or Thermodilatometer*, consisting of:

7.1.1.1 *Rigid Specimen Holder,* of inert, low expansivity material (≤20 µm/m-°C), usually quartz, to center the specimen in the furnace and to fix the specimen to mechanical ground.

NOTE 1—Use of rigid specimen holders and tension probes constructed of lower thermal expansivity (≤5 µm/m-°C) materials or corrections for hardware expansivity may be necessary if very small changes in specimen dimensions are encountered with this test method.

7.1.1.2 *Rigid Tension Probe,* of inert, low expansivity material (≤ 5 µm/m-°C), usually quartz, which contacts the specimen with an applied in-plane tensile force.

7.1.1.3 *Sensing Element,* with a dynamic range of at least 5 mm, a linearity of 1 % or better, and sufficient sensitivity to measure the displacement of the rigid tension probe within ±1 µm resulting from changes in length of the specimen.

7.1.1.4 *Weight or Force Transducer,* to generate a constant force between 0 and 50 m*N* ± 2 % that is applied through the rigid tension probe to the specimen.

7.1.1.5 *Furnace and Temperature Controller,* capable of executing a temperature program of uniform controlled heating of a specimen at a constant rate of 5 ± 0.2°C/min between required temperature limits to ±0.5°C.

7.1.1.6 *Temperature Sensor,* that can be positioned reproducibly in close proximity to the specimen to measure its temperature between −100 and 600°C with a resolution of ±0.1°C.

7.1.1.7 *Means of Providing a Specimen Environment,* of an inert gas at a purge rate of 10 to 50 mL/min ± 5 %. The typical purge gas rate is usually given by the instrument manufacturer.

NOTE 2—Typically 99.99 % pure nitrogen, argon, or helium is employed when oxidation in air is a concern. Unless effects of moisture are to be studied, use of dry purge gas is recommended; especially for operation at subambient temperatures.

7.1.1.8 *Data Collection Device,* provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for thermomechanical analysis are dimension change, temperature and time.

7.1.2 *Rigid Specimen Clamps,* (clamps, grips, pins, or split shot) of inert, low expansivity material (≤ 20 µm/m-°C) that grip the specimen between the rigid specimen holder and the rigid tension probe without distortion (<1 %) or slippage (<1 %).

7.2 Auxiliary equipment considered useful in conducting this test method includes:

7.2.1 *Coolant System,* that can be coupled directly to the furnace/temperature controller to hasten recovery from elevated temperatures, to provide controlled cooling rates constant to ±1.0°C/min, and to sustain a subambient temperature to ±0.5°C.

7.2.2 *Calipers,* or other measuring device to determine specimen dimensions to ±0.01 mm.

7.2.3 *Balance,* to determine the specimen mass to ±0.1 mg.

8. Sampling

8.1 Analyze samples as received or after a prescribed pretreatment. If some treatment is applied to a specimen prior to analysis, note this treatment and any resulting changes in mass or appearance in the report. For samples with a glass transition below ambient, it may be desirable to form the glass with a known thermal history by using a controlled constant cooling rate to the starting temperature. Film samples may undergo stress relief related dimensional change that depends on whether the sample is prepared and measured parallel to the machine direction of manufacture or perpendicular to the machine direction.

9. Calibration

9.1 Perform temperature calibration in accordance with the apparatus manufacturer operator's manual using the same heating rate, purge, and temperature sensor position to be used with the test method.

10. Procedure

10.1 Attach a pair of rigid specimen clamps to a specimen with a minimum spacing of 5 mm between the contact points. Weigh the specimen and clamps and record this value.

NOTE 3—Use of between-clamp distances of less than 5 mm may impart erroneous results because of end effects introduced by the clamp pressure. Refer to the Precautions Section, if a thickness outside the range of 0.2 to 5 mm is to be used.

10.2 Suspend the specimen with clamps between the contact points of the specimen holder and the tension probe. BE SURE THE POSITION OF THE TEMPERATURE SENSOR IS