~~Designation:E 1824–96~~ Designation: E 1824 – 02

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

This standard is issued under the fixed designation E 1824; 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.

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 under tension under prescribed experimental conditions.

1.2 This test method may be used as a complement to Test Method E 1545E 1545 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 Computer or electronic-based instruments, techniques, or data treatment equivalent to this test method may also be used.

NOTE 1—Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this test method to determine the necessary equivalency prior to use.

~~1.5The values stated in SI units are to be regarded as the 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:*

E 473 Terminology Relating to Thermal Analysis[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

E 1545 Test Method for Glass Transition Temperatures by Thermomechanical Analysis[2]

3. Terminology

3.1 *Definitions:*

3.1.1 The following terms are applicable to this test method and can be found in Terminology E 473E 473 and Terminology E 1142E 1142: *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) in the tensile mode 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 regime. 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

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[1] This test method is under the jurisdiction of ASTM Committee ~~E-37~~E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

~~Current edition approved Sept. 10, 1996. Published December 1996.~~

Current edition approved May 10, 2002. Published July 2002. Originally published as E 1824–96. Last previous edition E 1824–96.

[2] *Annual Book of ASTM Standards*, Vol 14.02.

thermal history, processing conditions, stability, progress of chemical reactions, and mechanical and electrical behavior.

~~5.2Thermomechanical analysis provides a rapid means of detecting changes in hardness or linear dimensional change associated with the glass transition.~~

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 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 2—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 (≤ 20 µm/m-°C), usually quartz, which contacts the specimen with an applied in-plane tensile force.

7.1.1.3 *Sensing Element*~~, linear over a minimum range of 5 mm to measure the displacement of the rigid tension probe within ±1 µm resulting from changes in length of the specimen.~~, 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 3—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 *Recording Device*, either digital or analog, to record and display the changes in the rigid tension probe position with a *Y*-sensitivity of 0.1 µm and temperature with an *X*-sensitivity of 0.1°C.

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

~~8.1Analyze 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.~~