Designation: E 1545 – 00

Standard Test Method for Assignment of the Glass Transition Temperature by Thermomechanical Analysis[1]

This standard is issued under the fixed designation E 1545; 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 procedures for the assignment of the glass transition temperature of materials on heating using thermomechanical measurements under prescribed experimental conditions.

1.2 This test method is applicable to amorphous or to partially crystalline materials that are sufficiently rigid below the glass transition to inhibit indentation by the sensing probe.

1.3 The normal operating temperature range is from – 100 to 600°C. This temperature range may be extended 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 advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this test method to verify equivalency prior to use.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 This test method is related to ISO 11359-2. ISO 11359-2 additionally covers the determination of coefficient of linear thermal expansion not covered by this test method.

1.7 *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.* Specific precautionary statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 832 Practice for Rubber Conditioning for Low-Temperature Testing[2]

E 473 Terminology Relating to Thermal Analysis[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

E 831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis[3]

E 1142 Terminology Relating to Thermophysical Properties[3]

E 1356 Test Method for Glass Transition Temperatures by Differential Scanning Calorimetry or Differential Thermal Analysis[3]

E 1363 Test Method for Calibration of Thermomechanical Analyzers[3]

2.2 *IIPEC Standard:*

Test Method 2.4.24, Glass Transition Temperature TMA Method[4]

ISO 11359-2 Plastics—Thermomechanical Analysis (TMA) – Part 2: Determination of Coefficient of Linear Thermal Expansion and Glass Transition Temperature[5]

3. Terminology

3.1 *Definitions*—The following terms are applicable to this test method and can be found in Terminologies E 473 and E 1142: *thermomechanical analysis* (*TMA*), *thermomechanical measurement*, *thermodilatometry*, *glass transition*, *glass transition temperature*, and *linear thermal expansion.*

4. Summary of Test Method

4.1 This test method uses thermomechanical analysis equipment (thermomechanical analyzer, dilatometer, or similar device) to assign the change in dimension of a specimen observed when the material is subjected to a constant heating rate through its glass transition. This change in dimension associated with the change from vitreous solid to amorphous liquid is observed as movement of the 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

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

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

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

[4] Available from The Institute for Interconnecting and Packaging Electronic Circuits, 7380 N. Lincoln Ave., Lincolnwood, IL 60646-1705.

[5] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

displacement curve before and after the transition is used to determine the glass transition temperature.

5. Significance and Use

5.1 The glass transition is dependent on the thermal history of the material to be tested. For amorphous and semicrystalline materials the assignment of the 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 expansion associated with the glass transition.

5.3 This test method is useful for research and development, quality control, and specification acceptance.

6. Apparatus

6.1 *Thermomechanical Analyzer (TMA)*—The essential instrumentation required to provide the minimum thermomechanical analytical capability for this test method includes the following:

6.1.1 A rigid specimen holder, composed of inert low expansivity material ≤1 µm m^{-1} $°C^{-1}$, to center the specimen in the furnace and to fix the specimen to mechanical ground.

6.1.2 A rigid circular expansion probe, 2 to 6 mm in diameter, composed of inert low expansivity material ≤1 µm m^{-1}° C^{-1}, that contacts the specimen with an applied compressive force.

6.1.3 A sensing element linear over a minimum 2-mm range to measure the displacement in length of the specimen.

6.1.4 A weight or force transducer to generate a constant force of 0 to 50 mN that is applied through the rigid compression probe to the specimen.

6.1.5 A furnace capable of providing uniform controlled heating (cooling) of a specimen to a constant temperature or at a constant rate over the temperature range of –100 to 600°C.

6.1.6 A temperature controller capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of 5 ± 0.5°C/minute.

6.1.7 A temperature sensor that can be attached to, in contact with, or reproducibly placed in close proximity to the specimen to provide an indication of the specimen/furnace temperature to ± 0.1°C.

6.1.8 A means of sustaining an environment around the specimen of a dry inert purge gas of 45 to 55 mL/minute.

NOTE 2—Typically, 99.9+ % pure nitrogen, argon or helium is used. Unless effects of moisture are to be studied, dry purge gas is recommended and is essential for operation at subambient temperatures.

6.1.9 A recording device, either digital or analog, capable of recording and displaying any fraction of the specimen dimension signal, including signal noise, on the Y-axis versus any fraction of the temperature signal, including noise, on the X-axis.

6.2 Micrometer or other measuring device to determine specimen dimensions of up to 8 mm with a precision of ± 0.1 mm.

7. Hazards

7.1 This test method may be used for amorphous and semicrystalline materials having a glass transition that is at or below room temperature providing care is taken to avoid contacting the specimen with a loaded probe prior to cooling the specimen below its glass transition. Applying a loaded probe to a specimen that is above its glass transition may cause partial penetration by the probe which can lead to probe sticking upon cooling below the glass transition. This condition has been known to yield erroneous results during the heating cycle.

7.2 With some materials a transient may be observed between the pre-transition slope and the final slope (Run 1 of Fig. 1). This may occur due to settling, residual stresses within the specimen, or alteration of the specimen morphology. Refer to Note 6 for directions when this is encountered.

7.3 Specimens of thickness less than 0.2 mm may be very difficult to handle. Thin films (50 to 200 µm) on a substrate may be considered for this test method providing the substrate is mechanically stable in the temperature region of the film glass transition.

7.4 For specimens of thickness greater than 5 mm, temperature nonuniformities of sufficient extent can develop within the specimen as to yield erroneously high values of the glass transition temperature using this test method.

8. Sampling

8.1 Analyze samples as received or after pretreatment. If some treatment is applied to a specimen prior to analysis, note this treatment and any resulting change in mass in the report.

9. Calibration

9.1 Perform calibration in accordance with Test Method E 1363.

10. Procedure

10.1 Calibrate the thermomechanical analyzer in accordance with Test Method E 1363.

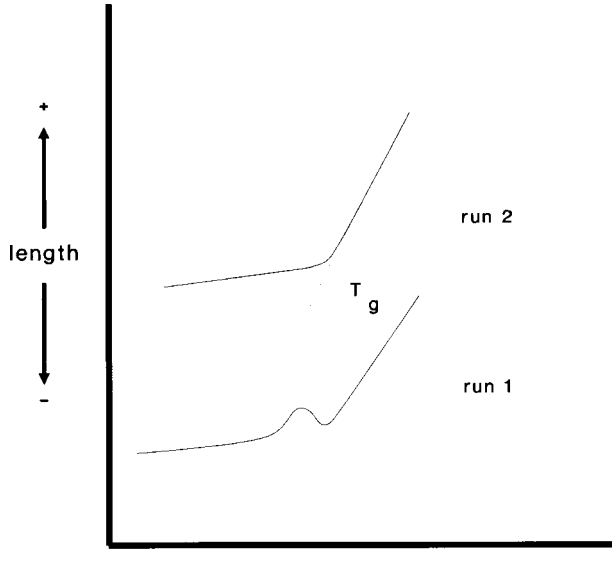

FIG. 1 Glass Transition Temperature from Expansion Mode