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Designation: ~~E1640 – 09~~ E1640 – 13

Standard Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis[1]

This standard is issued under the fixed designation E1640; 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 the assignment of a glass transition temperature ~~(Tg)~~(T_g) of materials using dynamic mechanical analyzers.

1.2 This test method is applicable to thermoplastic polymers, thermoset polymers, and partially crystalline materials which are thermally stable in the glass transition region.

1.3 The applicable range of temperatures for this test method is dependent upon the instrumentation used, but, in order to encompass all materials, the minimum temperature should be ~~about −150 °C.~~about −150°C.

1.4 This test method is intended for materials having an elastic modulus in the range of 0.5 MPa to 100 GPa.

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

1.6 This standard is similar to IEC 61006 except that standard uses the peak temperature of the loss modulus peak as the glass transition temperature while this standard uses the extrapolated onset temperature of the storage modulus change.

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.*

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4092 Terminology for Plastics: Dynamic Mechanical Properties

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1142 Terminology Relating to Thermophysical Properties

E1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E1545 Test Method for Assignment of the Glass Transition Temperature by Thermomechanical Analysis

E1867 Test Method for Temperature Calibration of Dynamic Mechanical Analyzers

E2254 Test Method for Storage Modulus Calibration of Dynamic Mechanical Analyzers

E2425 Test Method for Loss Modulus Conformance of Dynamic Mechanical Analyzers

2.2 *Other Standards:*

IEC 61006 Methods of Test for the Determination of the Glass Transition Temperature of Electrical Insulating Materials[3]

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in this document are defined in Terminology D4092 and E1142 including Celsius, dynamic mechanical analyzer, glass transition, glass transition temperature, loss modulus, storage modulus, tangent delta, and viscoelasticity.

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[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

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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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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~~3.1.2 *dynamic mechanical analyzer*—any of various commercial or experimental devices used to study the viscoelastic response of a specimen under a forced or free resonant oscillatory load. The force may be applied in torsion, flexure, or a combination of tension and compression.~~

4. Summary of Test Method

4.1 A specimen of known geometry is placed in mechanical oscillation at either fixed or resonant frequency and changes in the viscoelastic response of the material are monitored as a function of temperature. Under ideal conditions, during heating, the glass transition region is marked by a rapid decrease in the storage modulus and a rapid increase in the loss modulus and tangent delta. The glass transition of the test specimen is indicated by the extrapolated onset of the decrease in storage modulus which marks the transition from a glassy to a rubbery solid.

5. Significance and Use

5.1 This test method can be used to locate the glass transition region and assign a glass transition temperature of amorphous and semi-crystalline materials.

5.2 Dynamic mechanical analyzers monitor changes in the viscoelastic properties of a material as a function of temperature and frequency, providing a means to quantify these changes. In ideal cases, the temperature of the onset of the decrease in storage modulus marks the glass transition.

5.3 A glass transition temperature (T_g) is useful in characterizing many important physical attributes of thermoplastic, thermosets, and semi-crystalline materials including their thermal history, processing conditions, physical stability, progress of chemical reactions, degree of cure, and both mechanical and electrical behavior. T_g may be determined by a variety of techniques and may vary in accordance with the technique.

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

6. Interferences

6.1 Because the specimen size will usually be small, it is essential that each specimen be homogeneous ~~and/or~~or representative of the material as a ~~whole.~~whole, or both.

6.2 An increase or decrease in heating rates from those specified may alter results.

6.3 A transition temperature is a function of the experimental frequency, therefore the frequency of test must always be specified. (The transition temperature increases with increasing frequency.) Extrapolation to a common frequency may be accomplished using a predetermined frequency shift factor or assuming the frequency shift factor of about ~~8 °C~~8°C per decade of frequency.[4] Such extrapolation shall be reported.

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7. Apparatus

7.1 The function of the apparatus is to hold a specimen of uniform dimension so that the sample acts as the elastic and dissipative element in a mechanically oscillated system. Dynamic mechanical analyzers typically operate in one of several modes. See Table 1.

7.2 The apparatus shall consist of the following:

7.2.1 *Clamps,* a clamping arrangement that permits gripping of the specimen. Samples may be mounted by clamping at both ends (most systems), one end (for example, torsional pendulum), or neither end (free bending between knife edges).

7.2.2 *Oscillatory Stress (Strain),* for applying an oscillatory deformation (strain) or oscillatory stress to the specimen. The deformation may be applied and then released, as in freely vibrating devices, or continuously applied, as in forced vibration devices.

[4] ~~Ferry, D. "Viscoelastic Properties of Polymers," John Wiley & Sons, 1980.~~Ferry, D., *Viscoelastic Properties of Polymers*, John Wiley & Sons, 1980.

TABLE 1 Modes for Dynamic Mechanical Analyzers

Mode	Mechanical Response			
	Tension	Flexural	Torsional	Compression
Free/dec	...	...	X	...
Forced/res/CA	...	X	X	...
Forced/fix/CA	X	X	X	X
Forced/fix/CS	X	X	...	X

Free = free oscillation; dec = decaying amplitude; forced = forced oscillation; CA = constant amplitude; res = resonant frequency; fix = fixed frequency; CS = controlled stress.

7.2.3 *Detector,* for determining the dependent and independent experimental parameters, such as force (or stress), displacement (or strain), frequency, and temperature. Temperatures should be measurable with an accuracy of ~~±0.5 °C,~~ ±0.5°C, force to ±1 %, and frequency to ±0.1 Hz.

7.2.4 *Temperature Controller and Oven,* for controlling the specimen temperature, either by heating, cooling (in steps or ramps), or by maintaining a constant experimental environment. The temperature programmer shall be sufficiently stable to permit measurement of specimen temperature to ±0.5°C. The precision of the required temperature measurement is ~~±1.0 °C.~~±1.0°C.

7.2.5 *Data Collection Device,* to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals require for dynamic mechanical analysis are storage modulus, loss modulus, tangent delta, temperature and time.

NOTE 1—Some instruments suitable for this test may display only linear or logarithm storage modulus while others may display either linear ~~and/or~~or logarithm storage ~~modulus.~~ modulus, or both. Care must be taken to use the same modulus scale when comparing unknown specimens, and in the comparison of results from one instrument to another.

7.3 *Nitrogen, Helium* or other gas supplied for purging purposes.

7.4 *Calipers* or other length measuring device capable of measuring dimensions (or length ~~within) ± 0.01~~within) ±0.01 mm.

8. Precautions

8.1 Toxic and corrosive, or both, effluents may be released when heating some materials and could be harmful to personnel and to apparatus.

8.2 *Multiple Transitions*—Under some experimental conditions it is possible to have transitions secondary to the primary glass transition. Secondary transitions may be related to the glass transition of a second polymeric phase, melt processes, crystallization, chemical reactions, the motion of groups pendent to the main backbone or the crankshaft motion of the polymer backbone.

9. Samples

9.1 Samples may be any uniform size or shape, but are ordinarily analyzed in rectangular form. If some heat treatment is applied to the specimen to obtain this preferred analytical form, such treatment should be ~~noted in the report.~~reported.

9.2 Due to the numerous types of dynamic mechanical analyzers, sample size is not fixed by this test method. In many cases, specimens measuring between 1 × 5 × 20 mm and 1 × 10 × 50 mm are suitable.

NOTE 2—It is important to select a specimen size appropriate for both the material and the testing apparatus. For example, thick samples may be required for low modulus materials while thin samples may be required for high modulus materials.

10. Calibration

10.1 Calibrate the storage ~~modulus~~ modulus, loss modules, and temperature signals in accordance with Test Methods E1867 ~~and~~ , E2254, and E2425, respectively.

11. Procedure

11.1 Mount the specimen in accordance with procedure recommended by the manufacturer.

11.2 Measure the length, width, and thickness of the specimen to an accuracy of ±0.01 mm.

11.3 Maximum strain amplitude should be within the linear viscoelastic range of the material. Strains of less than 1 % are recommended and should not exceed 5 %.

11.4 Conduct tests at a heating rate of ~~1 °C/min~~ 1°C/min and a frequency of 1 Hz. Other heating rates and frequencies may be used but shall be reported.

NOTE 3—The glass transition temperature measured by dynamic mechanical measurements is dependent upon heating rate and oscillatory frequency. The experimental heating rate and the frequency of oscillation should be slow enough to allow the entire specimen to reach satisfactory thermal and mechanical equilibration. When the heating rate or oscillatory rate is high, the experimental time scale is shortened, and the apparent T_g is raised. Changing the time scale by a factor of 10 will generally result in a shift of about ~~8 °C~~ 8°C for a typical amorphous material. The effect of these variables on the temperature of the tangent delta peak may be observed by running specimens at two or more rates and comparing the results (see Appendix X1~~appendix).~~).

NOTE 4—Where possible in automated systems, a minimum of one data point should be collected for each °C increase in temperature. At low and high frequencies, use care in the selection of scanning rate and frequency rate; select test conditions and a data collection rate that will ensure adequate resolution of the mechanical response of the specimen. For example, select a heating rate that allows the specimen to complete at least one oscillation for ~~each° C~~each °C increase in temperature.

11.5 Measure and record the storage modulus, from ~~30 °C~~ 30°C below to ~~20 °C~~ 20°C above the suspected glass transition region.

12. Calculation

12.1 For the purpose of this test method the glass transition shall be taken as the extrapolated onset to the sigmoidal change in the storage modulus observed in going from the hard, brittle region to the soft, rubbery region of the material under test.

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NOTE 5—Storage modulus may be displayed on a linear or logarithmic scale. The reported glass transition temperature will differ depending upon the scale chosen. The scale type (for example, linear or logarithmic) shall be reported and must be the same for all parties comparing results.

12.1.1 Construct a tangent to the storage modulus curve below the transition temperature.

12.1.2 Construct a tangent to the storage modulus curve at the inflection point approximately midway through the sigmoidal change associated with the transitions.

12.1.3 The temperature at which these tangent lines intersect is reported as the glass transition temperature, T_g (see Fig. 1).

NOTE 6—Under special circumstances agreeable to all parties, other temperatures taken from the storage modulus, loss modulus, or tangent delta curve may be taken to represent the temperature range over which the glass transition takes place. Among these alternative temperatures are the peak of the loss modulus (T_l) or tangent delta (T_t) curves as illustrated in Fig. 2 and Fig. 3, respectively. These temperatures are generally in the order $T_g < T_l < T_t$.

12.2 For fixed frequency measurements at 1 Hz.

~~12.2.1 Report the mean value of duplicate determinations as T_g.~~

12.3 For measurements made at frequencies other than 1 Hz.

12.3.1 Using a predetermined frequency shift factor (k) (see Appendix X1~~appendix),~~), calculate the first approximation of the glass transition temperature (T_1') using equation 1.

$$T_l' = T + \frac{T^2}{k} \log \frac{F}{1\ \text{Hz}} \tag{1}$$

12.3.2 Calculate the glass transition temperature using equation 2:

$$T_1 = T + \frac{T\,T_1'}{k} \log \frac{F}{1\ \text{Hz}} \tag{2}$$

where:

~~k = Predetermined Frequency Shift Factor (see Appendix X1.1)~~
k = Predetermined Frequency Shift Factor (see Appendix X1)
F = Frequency of Measurement (Hz)
~~T = Glass Transition Temperature Observed at Frequency F (K)~~
T = Glass Transition Temperature Observed at Frequency F (K)
~~T_1' = First Approximation for the Glass Transition Temperature at 1 Hz (K)~~
T_1' = First Approximation for the Glass Transition Temperature at 1 Hz (K)
~~T_1 = Glass Transition Temperature at 1 Hz (K)~~
T_1 = Glass Transition Temperature at 1 Hz (K)

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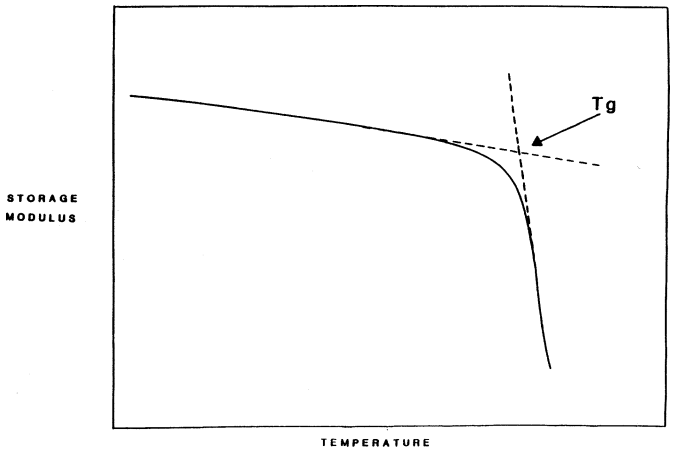

FIG. 1 Storage Modulus