Designation: E2347 – 16

Standard Test Method for Indentation Softening Temperature by Thermomechanical Analysis[1]

This standard is issued under the fixed designation E2347; 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 is applicable to materials that soften upon heating to a modulus less than 6.0 MPa. This test method describes the determination of the temperature at which the specific modulus of either 6.65 (Method A) or 33.3 MPa (Method B) (equivalent to Test Method D1525) of a test specimen is realized by indentation measurement using a thermomechanical analyzer as the test specimen is heated. This temperature is identified as the indentation softening temperature. The test may be performed over the temperature range of ambient to 300°C.

NOTE 1—This test method is intended to provide results similar to those of Test Method D1525 but is performed on a thermomechanical analyzer using a smaller diameter indenting probe. Equivalence of results to those obtained by Test Method D1525 has been demonstrated on a limited number of materials. Until the user demonstrates equivalence, the results of this Test Method shall be considered to be independent and unrelated to those of Test Method D1525.

1.2 This test method is not recommended for ethyl cellulose, poly (vinyl chloride), poly (vinylidene chloride) and other materials having a large measurement imprecision (see Test Method D1525 and 5.3 and Section 14).

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

1.4 There is no ISO standard equivalent to this test method.

1.5 *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]

D1525 Test Method for Vicat Softening Temperature of Plastics

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

E1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E2113 Test Method for Length Change Calibration of Thermomechanical Analyzers

E2206 Test Method for Force Calibration of Thermomechanical Analyzers

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in this test method are defined in Terminologies E473 and E1142 including *Celsius, complex modulus, modulus, strain, stress, storage modulus, thermal analysis,* and *thermomechanical analysis*.

3.1.2 *penetration softening temperature,* [°C], *n*—the temperature at which a test specimen has a modulus of either 6.65 or 33.3 MPa as measured in penetration.

4. Summary of Test Method

4.1 The modulus of a material may be determined by the indentation (penetration) of a circular, flat tipped probe. The relationship between modulus of a material (stress divided by strain) and penetration depth is given by:

$$E = 3\,F/(4\,D\,d) \quad (1)$$

where:

E = modulus, MPa,
F = force, N,

[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 standards Document Summary page on the ASTM website.

D = diameter of a circular, flat tipped probe, mm, and
d = penetration depth, mm.

NOTE 2—Note the identity Pa = N / m^2.

4.2 Some materials soften upon heating. For such materials, the modulus may be determined by penetration as the sample is heated. This test method identifies the temperature at which the modulus of the specimen is determined to be 6.65 MPa (Method A) or 33.3 MPa (Method B).

4.3 Specifically, a test specimen is tested in penetration using a circular, flat tipped probe. A known stress is applied to the center of a test specimen as it is heated at a constant rate from ambient temperature to the upper temperature limit for the material. The penetration (that is, deflection) of the test specimen is recorded as a function of temperature. The temperature at which the modulus of the specimen is determined to be 6.65 MPa (Method A) or 33.3 MPa (Method B) is determined to be the penetration softening temperature.

5. Significance and Use

5.1 Data obtained by this test method shall not be used to predict the behavior of materials at elevated temperatures except in applications in which the conditions of time, temperature, method of loading, and stress are similar to those specified in the test.

5.2 This test method is particularly suited for quality control and development work. The data are not intended for use in design or predicting endurance at elevated temperatures.

5.3 Ruggedness testing indicates that some materials, such as poly (vinyl chloride) exhibit substantially greater imprecision than that described in Section 14 for "well behaved" materials.

6. Apparatus

6.1 A thermomechanical analyzer consisting of:

6.1.1 *Rigid Specimen Holder,* 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 *Rigid Penetration Probe,* of inert, low expansivity material (<1 µm m^{-1} °C^{-1}) that contacts the specimen with an applied compression force (see Fig. 1). The tip shall be 0.1 to 1.0 mm in diameter, free of burrs and be perpendicular to the axis of the probe. The tip shall protrude at least 0.1 mm from the end of the probe.

6.1.3 *Deflection Sensing Element,* having a linear output over a minimum range of 5 mm to measure the displacement of the rigid penetration probe (see 6.1.2) to within ±0.1 µm.

6.1.4 *Programmable Force Transducer,* to generate a constant force (±2.5 %) between 0.05 and 1.0 N that is applied to the specimen through the rigid penetration probe (see 6.1.2).

NOTE 3—Other forces may be used but shall be reported.

6.1.5 *Temperature Sensor,* that can be positioned reproducibly in close proximity to the specimen to measure its temperature over the range of 25 to 300°C to ± 0.1°C.

6.1.6 *Temperature Programmer and Furnace,* capable of temperature programming the test specimen from ambient to 300°C at a linear rate of at least 2.0 ± 0.2°C/min.

6.1.7 *Means of Providing a Specimen Environment,* of inert gas at a purge rate of 50 mL/min ± 5 %.

NOTE 4—Typically, inert purge gas that inhibits specimen oxidation are 99.9+ % pure nitrogen, helium or argon. Dry gases are recommended for all experiments unless the effect of moisture is part of the study.

6.1.8 *Data Collection Device,* to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required are a change in linear dimension to a sensitivity of ±0.1 mm, and temperature to a sensitivity of ±1 µm.

6.1.9 *Calipers, Micrometer,* or other length measuring device capable of a length measurement of up to 2 mm with a precision of ±1 µm.

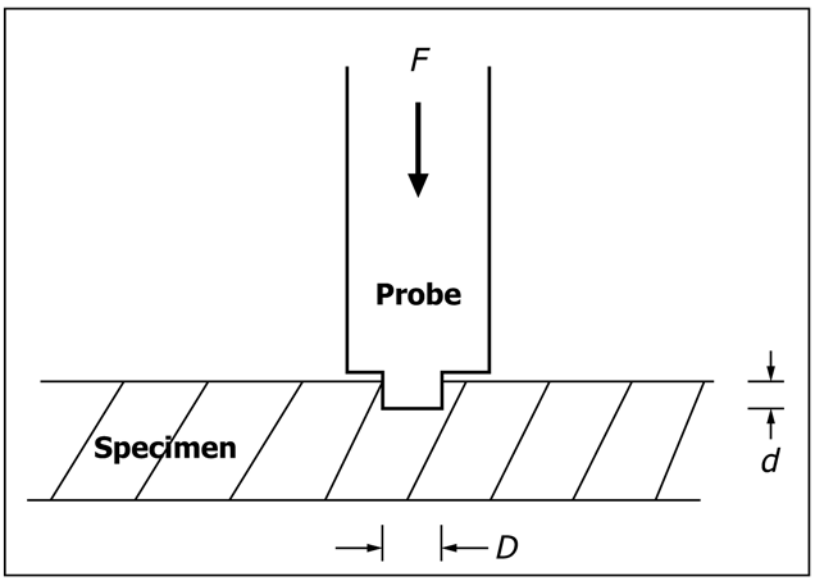

FIG. 1 Penetration Probe

7. Hazards

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

8. Sampling, Test Specimens, and Test Units

8.1 Because the specimen size is small, care shall be taken to ensure that each specimen is homogeneous and representative of the sample as a whole.

8.2 The specimen may be cut from sheets, plates or molded shapes, or may be molded to the desired finished dimensions.

8.3 A typical test specimen is a rectangle 7 to 8 × 7 to 8 mm or a circle 7 to 8 mm in diameter with a thickness of 1 to 3 mm.

8.4 This test method assumes that the material is isotropic. Should specimens be anisotropic, such as in reinforced composites, the direction of the reinforcing agent shall be reported relative to the compression (specimen) dimensions.

9. Preparation of Apparatus

9.1 Perform any setup or calibration procedures recommended by the apparatus manufacturer in the operations manual.

10. Calibration and Standardization

10.1 Calibrate the temperature display of the apparatus according to Test Method E1363 using a heating rate of 2.0 ± 0.2°C/min.

10.2 Calibrate the deflection display of the apparatus according to Test Method E2113.

10.3 Calibrate the mechanism for applying force to the test specimen according to Test Method E2206.

11. Procedure

11.1 Measure the diameter of the circular penetration tip of the penetration probe to ±1 µm and record this value as *D*.

11.2 *Method A:*

11.2.1 Set the value of Force (*F*) at 0.15 ± 0.004 N.

11.2.2 Proceed with steps 11.3.2 to 11.3.4.6.

11.3 *Method B:*

11.3.1 Set the value of Force (*F*) to 0.75 ± 0.01 N.

11.3.2 *Perform Scouting Experiment:*

11.3.2.1 Using Eq 2 and an estimated value of d_o = 0, estimate the deflection (*d′*) to be used as the experimental endpoint to three significant figures.

11.3.2.2 Center the test specimen on the stage with a surface perpendicular to the loading nose of the penetration probe.

11.3.2.3 Load the penetration probe onto the center of the test specimen with the force determined in 11.2.1 (Method A) or 11.3.1 (Method B). Set the deflection signal to zero at ambient temperature.

11.3.2.4 Heat the test specimen at 2.0 ± 0.2°C min^{-1} from ambient temperature until the deflection *d′* (determined in 11.3.2.1) is obtained while recording specimen deflection and temperature. Once the deflection value is achieved, terminate the temperature program and remove the load from the test specimen. Cool the apparatus to ambient temperature.

11.3.2.5 Record the temperature at the deflection value *d′* as the estimated indentation softening temperature (*T′*).

11.3.2.6 For ease of interpretation, record the thermal curve with penetration displayed on the *Y*-axis and temperature on the *X*-axis as illustrated in Fig. 2.

11.3.3 *Determine the Baseline:*

11.3.3.1 With no sample present, place the tip of the penetration probe onto the center of the sample stage. Load the probe with the force determined in 11.2.1 or 11.3.1. Set the deflection scale signal to be zero at ambient temperature.

11.3.3.2 Heat the sample area at 2.0 ± 0.2°C min^{-1} from ambient temperature to a temperature 5°C higher than *T′* determined in 11.3.2.5. Once the temperature program is complete, remove the load from the probe and cool the apparatus to ambient temperature.

11.3.3.3 Measure the deflection of the baseline at temperature *T′* and record it as d_o.

NOTE 5—d_o is positive for a baseline that expands with temperature and negative if the baseline contracts.

11.3.4 *Test Specimen:*

11.3.4.1 Using Eq 2 and the value for d_o from 11.3.3.3, determine to three significant figures the deflect (*d*) to be used as the experimental endpoint.

11.3.4.2 Center the test specimen on the stage with a surface perpendicular to the loading nose of the penetration probe.

11.3.4.3 Load the penetration probe onto the center of the test specimen with the force determined in 11.2.1 (Method A) or 11.3.1 (Method B). Set the deflection signal to zero at ambient temperature.

NOTE 6—During heating, the test specimen may expand (see Fig. 2). Nonetheless, the deflection value is taken from the original dimension of the test specimen measured at ambient conditions. This corresponds with the conditions of D1525. Intralaboratory studies show that using the original dimension compared to the maximum dimension produces a 0.7°C increase in the value for *T*. This is within experimental error (see Section 14).

11.3.4.4 Using the appropriate softening temperature determined in 11.3.2.5 start the temperature program 50°C below this temperature and heat the test specimen at 2.0 ± 0.2°C min^{-1} from ambient temperature until the deflection *d* (determined in 11.3.4.1) is obtained while recording specimen deflection and temperature. Once the deflection value is achieved, terminate the temperature program and remove the load from the test specimen. Cool the apparatus to ambient temperature.

11.3.4.5 Record the temperature at the deflection value *d* as the indentation softening temperature (*T*).

11.3.4.6 For ease of interpretation, record the thermal curve with penetration displayed on the *Y*-axis and temperature on the *X*-axis as illustrated in Fig. 2.

12. Calculation

12.1 Calculate the deflection value as follows:

$$d = (3\ F/4\ D\ E) - d_o \qquad (2)$$

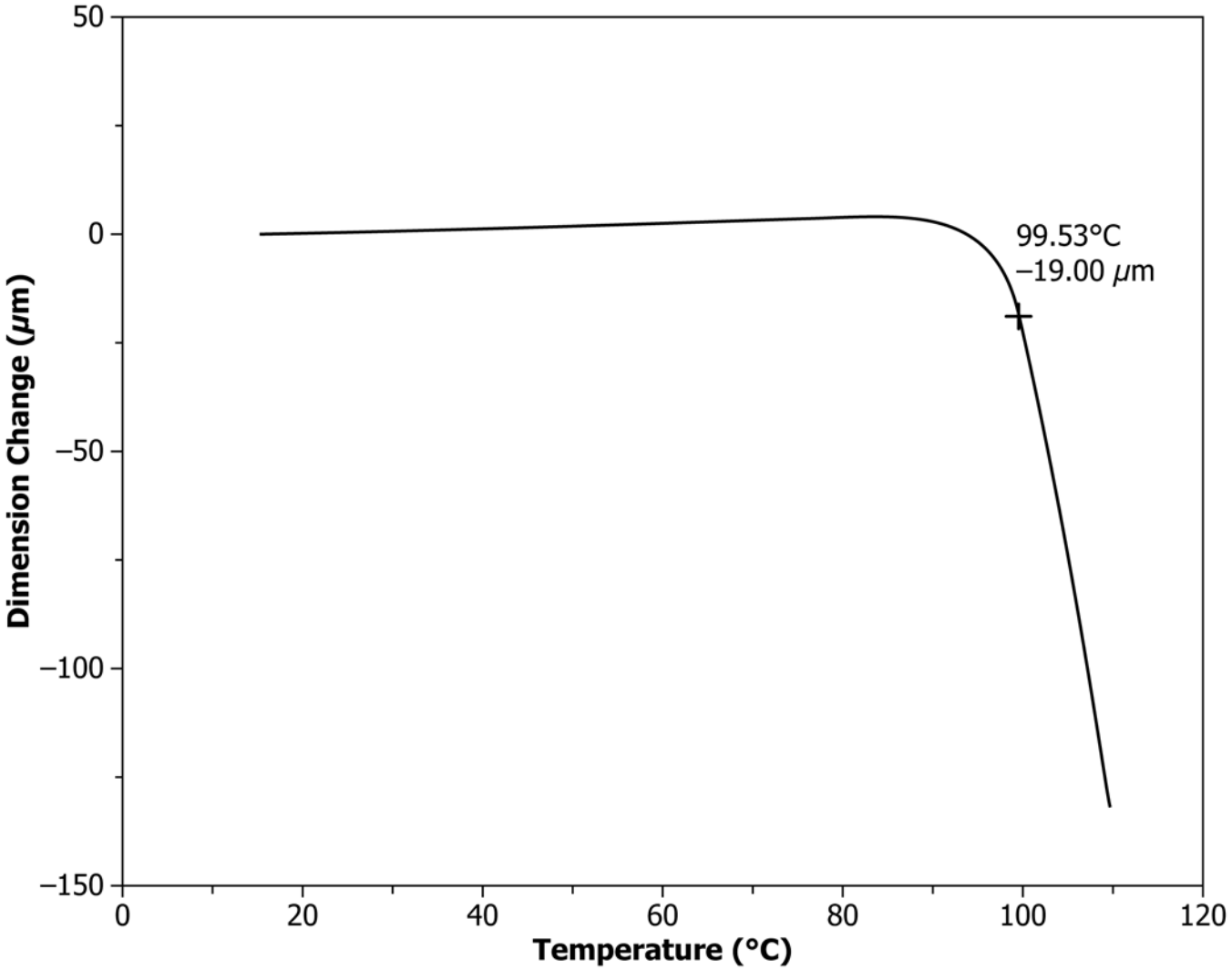

FIG. 2 Penetration Curve of Polystyrene

where:

E = modulus, MPa,
F = force, N,
D = diameter of a circular, flat tipped probe, mm,
d = penetration depth, mm, and
d_o = baseline depth at temperature T, mm.

NOTE 7—Note the identity Pa = N / m^2.

12.1.1 For example, if:

E = 6.65 MPa,
F = 0.15 N,
D = 0.889 mm, and
d_o = 0.0003 mm.

13. Report

13.1 Report the following information:

13.1.1 Complete identification and description of the material tested including source, manufacturer code and any thermal or mechanical pretreatment.

13.1.2 Description of the instrument used, including model number and location of the temperature sensor.

13.1.3 Details of the procedure used to calculate the penetration softening temperature including strain and resulting force, stress and resultant strain, as well as specimen dimensions.

13.1.4 Heating rate and temperature range.

13.1.5 A copy of all original records that are presented.

13.1.6 The penetration softening temperature (T), and

13.1.7 The specific dated version of this test method used.

14. Precision and Bias

14.1 An interlaboratory study was conducted in 2005 in which polystyrene was tested using Method A (6.65 MPa modulus) and Method B (33.3 MPa modulus). Twelve laboratories participated in the test using six instrument models from three manufacturers.[3]

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1034. Contact ASTM Customer Service at service@astm.org.

14.2 *Precision:*

14.2.1 Within laboratory variability may be described using the repeatability value (r) obtained by multiplying the repeatability standard deviation by 2.8. The repeatability value estimates the 95 % confidence limit, That is, two results from the same laboratory should be considered suspect (at the (95 % confidence level) if they differ by more than the repeatability value.

14.2.2 The within laboratory repeatability standard deviation obtained for Method A (6.65 MPa modulus) was 1.1°C with 36 degrees of experimental freedom.

14.2.3 The within laboratory repeatability standard deviation obtained for Method B (33.3 MPa modulus) was 1.1°C with 36 degrees of experimental freedom.

14.2.4 The between laboratory variability may be described using the reproducibility value (R) obtained by multiplying the reproducibility standard deviation by 2.8. The reproducibility value estimates the 95 % confidence limit. That is, results obtained from two different laboratories, operators or apparatus should be considered suspect (at the 95 % confidence level) if they differ by more than the reproducibility value.

14.2.5 The between laboratory reproducibility standard deviation for Method A (6.65 MPa modulus) was 3.2°C.

14.2.6 The between laboratory reproducibility standard deviation for Method B (33.3 MPa modulus) was 5.2°C.

14.3 *Bias:*

14.3.1 Bias is the difference between the mean value obtained and an acceptable reference value for the same material. To the knowledge of the committee, no acceptable reference material is available for indentation softening temperature. Therefore, bias was unable to be determined.

14.3.2 The mean value for polystyrene characterized by Method A (6.65 MPa modulus) was found to be 100.0°C with 36 degrees of experimental freedom.

14.3.3 The mean value for polystyrene characterized by Method B (33.3 MPa modulus) was found to be 96.3°C with 36 degrees of experimental freedom.

14.4 *Comparison of Results to That of Other Methods:*

14.4.1 Test Method D1525 reports a repeatability standard deviation of ±0.24°C for polystyrene for the VICAT softening temperature of 97.3°C using Method A.

15. Keywords

15.1 penetration temperature; softening; strain; stress; temperature; thermomechanical analysis (TMA); vicat temperature