Designation: E2769 – 22

Standard Test Method for Elastic Modulus by Thermomechanical Analysis Using Three-Point Bending and Controlled Rate of Loading[1]

This standard is issued under the fixed designation E2769; 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 describes the use of linear controlled-rate-of-loading in three-point bending to determine the elastic modulus of isotropic specimens in the form of rectangular bars using a thermomechanical analyzer (TMA).

NOTE 1—This method is intended to provide results similar to those of Test Methods D790 or D5934 but is performed on a thermomechanical analyzer using smaller test specimens. Until the user demonstrates equivalence, the results of this method shall be considered independent and unrelated to those of Test Methods D790 or D5934.

1.2 This test method provides a means for determining the elastic modulus within the linear region of the stress-strain curves (see Fig. 1). This test is conducted under isothermal temperature conditions from –100 °C to 300 °C.

1.3 Typical test specimens are in the form of thin strips 0.5 mm in thickness, 1.5 mm in width, and 6 mm in length. The size of the test specimen is limited by the distance between the supports used in the three-point bending mode of operation, commonly 0.5 cm.

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 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

D5934 Test Method for Determination of Modulus of Elasticity for Rigid and Semi-Rigid Plastic Specimens by Controlled Rate of Loading Using Three-Point Bending (Withdrawn 2009)[3]

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

E1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E2092 Test Method for Distortion Temperature in Three-Point Bending by Thermomechanical Analysis

E2113 Test Method for Length Change Calibration of Thermomechanical Analyzers

E2206 Test Method for Force Calibration of Thermomechanical Analyzers

3. Terminology

3.1 *Definitions*—Definitions of technical terms used in this standard are defined in Terminologies E473 and E1142 including *anisotropic, Celsius, expansivity, isotropic, proportional limit, storage modulus, strain, stress, thermodilatometry, thermomechanical analysis,* and *yield point*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *elastic modulus, n*—the ratio of stress to corresponding strain within the elastic limit on the stress-strain curve (see Fig. 1) expressed in Pascal units.

4. Summary of Test Method

4.1 A specimen of rectangular cross section is tested in three-point bending (flexure) as a beam. The beam rests on two

[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] The last approved version of this historical standard is referenced on www.astm.org.

***A Summary of Changes section appears at the end of this standard**

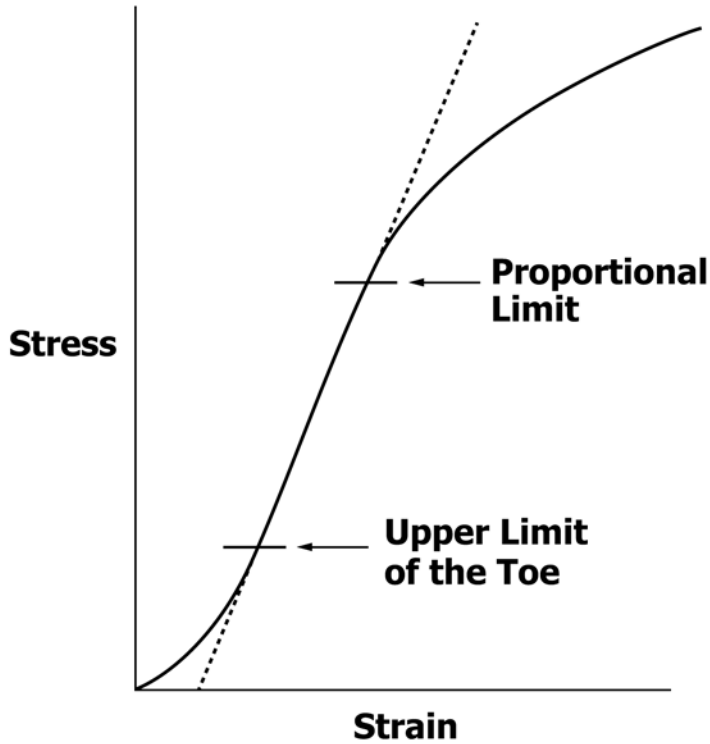

FIG. 1 Stress-Strain Curve (Linear Region)

supports and is loaded midway between the supports by means of a loading nose. A linearly increasing load (stress) is applied to the test specimen of known geometry while the resulting deflection (strain) is measured under isothermal conditions. The elastic modulus is obtained from the linear portion of the display of resultant strain versus applied stress.

5. Significance and Use

5.1 This test method provides a means of characterizing the mechanical behavior of materials using very small amounts of material including thermoplastic and thermoset polymers, composites, and metals.

5.2 The data obtained may be used for research and development and establishment of optimum processing conditions. The data are not intended for use in design or for predicting performance.

NOTE 2—This test method may not be suitable for anisotropic materials.

6. Interferences

6.1 Since small test specimen geometries are used, it is essential that the specimens be representative of the material being tested.

6.2 This test method is not applicable for strains greater than 3 %.

7. Apparatus

7.1 The function of the apparatus is to hold a rectangular test specimen (beam) so that the material acts as the elastic and dissipative element in a mechanically driven linear displacement system. Displacements (deflections) are generated using a controlled loading rate applied to a specimen in a three-point bending configuration.

7.2 *Thermomechanical Analyzer*—The essential instrumentation required to provide the minimum thermomechanical analytical or thermodilatometric capability for this method includes:

7.2.1 A rigid *specimen holder* of inert low expansivity material ≤30 µm m^{-1} K^{-1} to center the specimen in the furnace and to fix the specimen to mechanical ground.

7.2.2 A rigid *flexure fixture* of inert low expansivity material ≤30 µm m^{-1} K^{-1} to support the test specimen in a three-point bending mode (see Fig. 2). The radius of the supports shall not be greater than 1 mm.

7.2.3 A rigid *knife-edge compression probe* of inert low expansivity material ≤30 µm m^{-1} K^{-1} that contacts the specimen with an applied compressive force (see Fig. 1). The radius of the knife-edge shall not be larger than 1 mm.

7.2.4 *Deflection sensing element*, having a linear output over a minimum range of 5 mm to measure the displacement of the rigid compression probe (see 7.2.3) to within ±0.1 µm.

7.2.5 *Programmable weight or force transducer* to generate a force program of 0.1 N min^{-1} over the range of 0.01 to 1.0 N that is applied to the specimen through the rigid compression probe (see 7.2.3).

7.2.6 *Temperature sensor*, that can be reproducibly positioned in close proximity to the specimen to measure its temperature with the range between –100 °C and 300 °C to within ±0.1 °C.

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

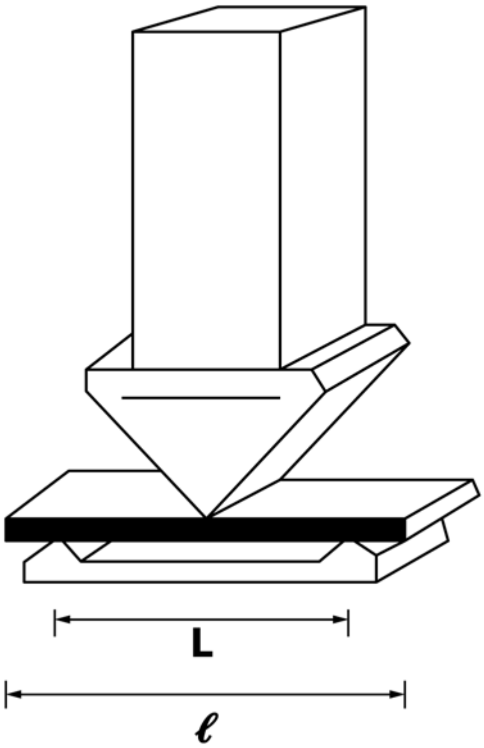

FIG. 2 Flexure Support Geometry

7.2.7 *Temperature programmer and furnace* capable of temperature programming the test specimen from –100 °C to 300 °C at a linear rate of at least 20 ± 1 °C min^{-1} and holding isothermally to within ±1 °C.

7.2.8 *Means of sustaining an environment around the specimen* of inert gas at a purge rate of 50 mL min^{-1} ± 5 %.

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

7.2.9 A *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 change, applied force, temperature and time.

7.2.10 While not required, it is convenient to have the capability for continuous calculation and display of stress and strain resulting from the measurements of dimension change and force.

7.3 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

7.3.1 *Cooling capability* to provide isothermal subambient temperatures.

7.4 *Micrometer*, calipers, film gage or other length-measuring device capable of measuring length of 0.01 mm to 20 mm with a precision of ±0.001 mm (±1 µm).

NOTE 5—Propagation of uncertainties shows that the largest source of error in this determination is the accuracy with which the test specimen thickness is measured. Care should be taken to ensure the best precision and accuracy in this measurement.

7.5 A high modulus (>2 GPa) beam *reference material*, 0.5 mm in thickness or greater of approximately the same width and length as the test specimen.

8. Hazards

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

9. Test Specimens

9.1 The test specimens used in this test method are ordinarily in the form of rectangular beams with aspect ratios of 1:3:12 for thickness or specimen depth (d), width (b), and length (l), depending upon the modulus of the sample and length of the support span (L).

NOTE 6—Other specimen and support dimensions may be used but care must be taken that the support length to specimen thickness ratio (L/d) be greater than 10.

NOTE 7—The specimen shall be long enough to allow overhanging on each end of at least 10 % of the support span, that is $l \geq 1.2\ L$.

NOTE 8—For precise results, the surfaces need to be smooth and parallel. Twisting of the specimen will diminish precision.

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

9.3 Replicate determinations are required. Sufficient test specimens for replicated determinations shall be prepared for each sample.

10. Calibration

10.1 Calibrate the temperature measurement system of the apparatus according to Test Method E1363 using a heating rate of 1 ± 0.1 °C min^{-1}.

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

10.3 Calibrate the force display of the apparatus according to Test Method E2206.

11. Conditioning

11.1 Polymeric test specimens shall be conditioned at 23 ± 2 °C and 50 ± 10 % relative humidity for not less than 40 h prior to test according to Procedure A of Practice D618, unless otherwise specified and reported.

12. Procedure

12.1 Measure the test length (L) of the test specimen as the distance between the two support points of the flexure fixture to three significant figures (see Fig. 2).

NOTE 9—For many apparatus, this will be 5.0 mm.

12.2 Measure the width (b) and thickness (d) of the specimen midway along its length to three significant figures (see Fig. 3). (See Note 5).

12.3 Center the specimen on the supports of the flexure fixture, with the long axis of the specimen perpendicular to the loading nose and supports (see Fig. 2).

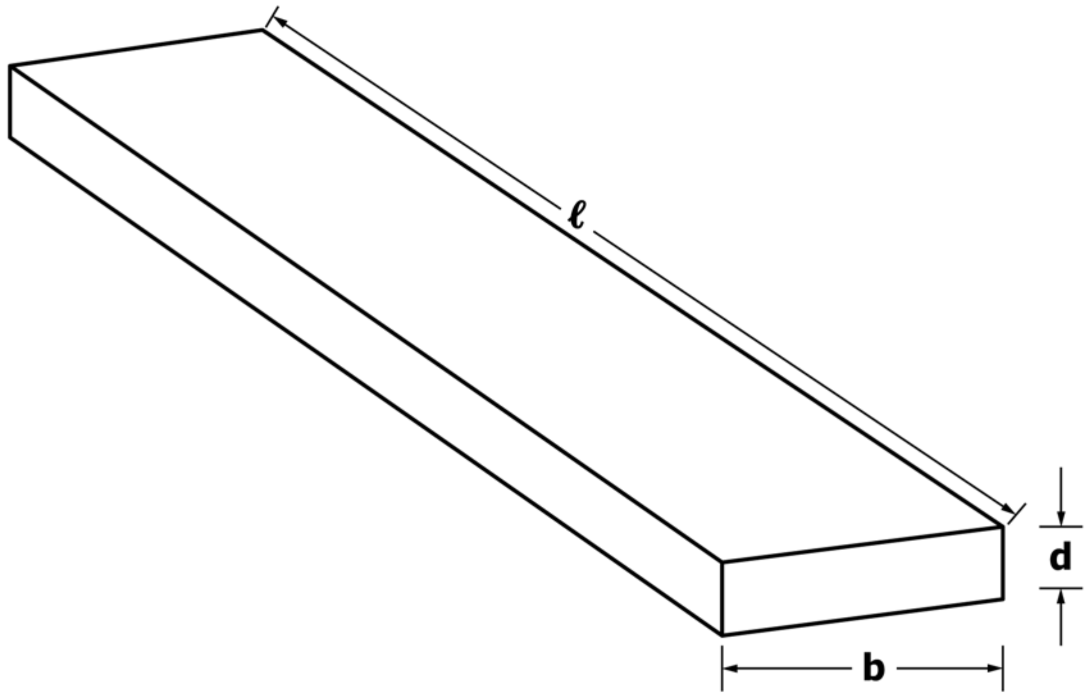

FIG. 3 Test Specimen Geometry

NOTE 10—The typical rectangular test beam is tested flat wise on the support span, with the applied force through its thinnest dimension.

12.4 Place the furnace around the test specimen and program the temperature to the desired isothermal test temperature ±1 °C and equilibrate for 3 min.

12.5 Preload the test specimen with 0.01 N ± 1 % of full scale. Set the displacement-axis signal to be zero.

12.6 Apply a linearly increasing force at a rate of $0.1\ N\ min^{-1} \pm 1\ \%$ up to the maximum applicable load while recording the applied force (or calculated stress) and specimen displacement (or calculated strain) as a function of time. Terminate the test if the maximum strain reaches 30 mm/m (3 %) or the proportional limit, the yield force, the rupture force or the maximum force of the analyzer has been reached, whichever occurs first. Once maximum force is achieved, terminate the force program and remove the load from the test specimen. Cool the apparatus to ambient temperature.

NOTE 11—This method is not applicable for strains higher than 3 %.

NOTE 12—If the specimen fails or ruptures, then use another specimen and repeat the test using forces that do not exceed the linear region as defined by the failed or ruptured specimen.

12.7 Perform a baseline determination similar to sections 1.3 – 12.6 except that the test specimen is a high modulus beam of the same nominal dimensions as the test specimen.

12.8 For ease of interpretation, display the thermal curves from sections 12.6 and 12.7 with stress or force on the Y-axis and strain or deflection on the X-axis. The same X- and Y- axis scale sensitivities shall be used for both thermal curves.

12.9 Using the same Y-axis scale sensitivity, subtract the baseline curve of 12.7 from the test specimen curve of 12.6.

12.10 *Method A*—Using the resultant curve from 12.9, prepare a display of stress (see Eq 1) on the Y-axis and strain (see Eq 2) on the X-axis such as that in Fig. 1.

12.11 Determinate the slope of the linear portion of the curve (that is, between the "upper limit of the toe" and the "proportional limit"). Report this slope as the elastic modulus (E) in bending according to Eq 3.

12.12 *Method B*—Using the resultant curve from 12.9, prepare a display of applied force on the Y-axis (or derived stress) and deflection (or derived strain) on the X-axis. Determine the linear portion of the curve (that is, between the "upper limit of the toe" and the "proportional limit") Determine and report the value of elastic modulus (E) at an identified point within this linear region using Eq 3.

13. Calculation

13.1 The elastic modulus is the ratio of stress with respect to strain within the elastic limit of the stress-strain curve (Fig. 1). It is calculated using Eq 3.

$$\text{stress} = \sigma = \frac{(3\ F\ L)}{(2\ b\ d^2)} \tag{1}$$

where:

σ = stress, MPa,
b = beam width, mm,
d = beam thickness, mm,
D = beam displacement, mm,
E = elastic modulus, MPa,
F = force, N,
L = support span, mm, and
ε = strain, dimensionless.