Standard Test Method for Plastics: Dynamic Mechanical Properties: In Torsion[1]

This standard is issued under the fixed designation D 5279; 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 use of dynamic mechanical instrumentation for gathering and reporting the viscoelastic properties of thermoplastic and thermosetting resins and composite systems in the form of rectangular specimens molded directly or cut from sheets, plates, or molded shapes. The torsional data generated may be used to identify the thermomechanical properties of a plastics material or composition.

1.2 This test method is intended to provide means for determining the modulus as a function of temperature of plastics using nonresonant forced-vibration techniques, as outlined in Practice D 4065. Plots of the elastic (storage), loss (viscous), and complex moduli and tan delta, as a function of frequency, time, or temperature are indicative of significant transitions in the thermomechanical performance of the polymeric material system.

1.3 This test method is valid for a wide range of frequencies, typically from 0.01 to 100 Hz.

1.4 Apparent discrepancies may arise in results obtained under differing experimental conditions. These apparent differences from results observed in another study can usually be reconciled without changing the observed data by reporting in full (as described in this test method) the conditions under which the data were obtained.

1.5 Test data obtained by this test method are relevant and appropriate for use in engineering design.

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

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.*

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 4065 Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics[3]

D 4092 Terminology Relating to Dynamic Mechanical Measurements of Plastics[3]

3. Terminology

3.1 For definitions applicable to this test method, refer to Terminology D 4092.

4. Summary of Test Method [4]

4.1 This test method covers the determination of the shear modulus of plastics using dynamic mechanical techniques. A test specimen of rectangular cross section is tested in dynamic torsion. The specimen is gripped longitudinally between two clamps. The specimen of known geometry is placed in mechanical torsional displacement at either a fixed frequency, or variable frequencies at either isothermal conditions, or with a linear temperature increase. The elastic or loss modulus, or both, of the polymeric material system are measured in torsion.

5. Significance and Use

5.1 This test method provides a simple means of characterizing the thermomechanical behavior of plastics materials using very small amounts of material. The data obtained may be used for quality control, research and development, and establishment of optimum processing conditions.

5.2 Dynamic mechanical testing provides a sensitive method for determining thermomechanical characteristics by measuring the elastic and loss moduli as a function of frequency, temperature, or time. Plots of moduli and tan delta of a material versus temperature provide graphical representations indicative of functional properties, effectiveness of cure (thermosetting resin system), and damping behavior under specified conditions.

5.3 This test method can be used to assess

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 08.02.

[4] The particular method for measurement of the elastic and loss moduli and tan delta depends upon the individual instrument's operating principles.

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

5.3.1 The modulus as a function of temperature,

5.3.2 The modulus as a function of frequency,

5.3.3 The effects of processing treatment, including orientation,

5.3.4 Relative resin behavioral properties, including cure and damping,

5.3.5 The effects of substrate types and orientation (fabrication) on elastic modulus, and

5.3.6 The effects of formulation additives that might affect processability or performance.

6. Interferences

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

7. Apparatus

7.1 The function of the apparatus is to hold a rectangular test specimen so that the material acts as the elastic and dissipative element in a mechanically driven torsional system. Dynamic mechanical instruments operate at a forced, constant amplitude, and either at a fixed frequency, or variable frequencies.

7.2 The apparatus shall consist of the following:

7.2.1 *Fixed Member*—A fixed or essentially stationary member carrying one grip.

7.2.2 *Movable Member*—A movable member carrying a second grip.

7.2.3 *Grips*—Grips for holding the test specimen between the fixed member and the movable member. The grips shall be mechanically aligned, that is, they shall be attached to the fixed and movable member, respectively, in such a manner that they will move into alignment as soon as any load is applied, so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly.

7.2.3.1 The test specimen shall be held in such a way that slippage relative to the grips is minimized.

7.2.4 *Deformation (Strain Device)*—A device for applying a continuous linear deformation (strain) to the specimen. In the force-displacement device the deformation (strain) is applied and then released. (See Table 1 of Practice D 4065.)

7.2.5 *Detectors*—Devices for determining dependent and independent experimental parameters, such as force (stress), deformation (strain), frequency, and temperature. Temperature should be measurable with a precision of ±1°C, frequency to ±1 %, and force to ±1 %.

7.2.6 *Temperature Controller and Oven*— A device for controlling the temperature, either by heating (in steps or ramps), cooling (in steps or ramps), maintaining a constant specimen environment, or a combination thereof. A temperature controller should be sufficiently stable to permit measurement of environmental chamber temperature to within 1°C.

7.3 Nitrogen or other inert gas supply for purging purposes.

8. Test Specimens

8.1 The specimens may be cut from sheets, plates or molded shapes, or may be molded to the desired finished dimensions. Typically, the rectangular test specimen is 76 by 13 by 3 mm. Rectangular test specimens of other dimensions can be used but should be clearly identified in the report section. The distance between grips is approximately 64 mm.

9. Calibration

9.1 Calibrate the instrument using procedures recommended by the manufacturer.

10. Conditioning

10.1 *Conditioning*—Condition the test specimens at 23.0 ± 2.0°C and 50 ± 5 % relative humidity for not fewer than 40 h prior to test in accordance with Procedure A of Practice D 618 for those materials requiring conditioning.

11. Procedure

11.1 Use an untested specimen for each measurement. Measure the width and thickness of the specimen to the nearest 0.03 mm at the center of the specimen.

11.2 Clamp the test specimen between the movable and stationary members; use shim stock, if necessary, to minimize slippage within the clamp.

11.3 Preload the test specimen so that there is a positive force. Monitor the normal force to ensure adequate preloading.

11.4 Measure to the nearest 0.03 mm the jaw separation between the movable and stationary members.

11.5 Select the desired frequency (or frequencies) for dynamic-torsional displacement.

11.6 Select the torsional-displacement amplitude.

11.7 Temperature sweep.

11.7.1 Temperature increases should be controlled to 1 to 2°C/min for linear increases or 2 to 5°C/min with a minimum of 1-min thermal-soak time for step increases. This will allow characterizing of the modulus from the glassy region, through the glass-transition region, up to the softening or glassy-rubbery state.

11.8 The tan-delta peak will coincide with a change in G' (elastic or storage) modulus through the glass-transition region. Another indication of the glass-transition is a maximum value of the G'' (loss or viscous) modulus.

12. Calculations

12.1 The equations listed in Practice D 4065 are used to calculate the important rheological properties measured in forced, nonresonant dynamic displacement:

where:
G' = storage (elastic) modulus in torsion,
G'' = loss (viscous) modulus in torsion,
G^* = complex modulus in torsion, and
d^* = tan delta.

13. Report

13.1 Report the following information:

13.1.1 Complete identification of the material tested, including type, source, manufacturer's code, number, form, principal dimensions, and previous history,

13.1.2 Description and direction of cutting and loading specimen, including preload force, 13.1.3 Conditioning procedure,

13.1.4 Description of the instrument used for the test,

13.1.5 Description of the calibration procedure,

13.1.6 Identification of the sample atmosphere by gas composition, purity, and rate used,

13.1.7 Width and thickness of specimen,

13.1.8 Jaw separation distance,

13.1.9 Frequency of dynamic displacement,

13.1.10 Amplitude of displacement,

13.1.11 Thermal gradient; heating rate,

13.1.12 Number of specimens tested,

13.1.13 Equations used to calculate values,

13.1.14 Table of data and results, including moduli and tan delta as a function of temperature, and

13.1.15 A plot of the modulus (moduli) and tan delta as a function of temperature. (See Fig. 1.)

14. Precision and Bias

14.1 The repeatability standard deviation has been determined for the following materials . Laboratory A evaluated a polyurethane sample and the values in Table 1 were obtained with the same test method in the same laboratory by the same operator using the same equipment in the shortest practical period of time using test specimens taken at random from a single quantity of homogeneous material. Laboratory B tested the same material and obtained the results shown in Table 1.

TABLE 1 DMRT-Torsion, Elastic Modulus, G' (E^8 Pascals) or (E^9 dynes/cm^2) at Selected Temperatures

Laboratory	–40°C	0°C	40°C	80°C
A				
Mean	3.773	0.2038	0.03624	0.03532
Standard Deviation	0.008	0.0067	0.00032	0.00087
B				
Mean	3.568	0.2263	0.03206	0.02758
Standard Deviation	0.160	0.0461	0.00142	0.00023

15. Keywords

15.1 dynamic mechanical rheological properties; elastic; loss; storage; tan delta; torsional shear modulus; viscoelastic behavior; viscous

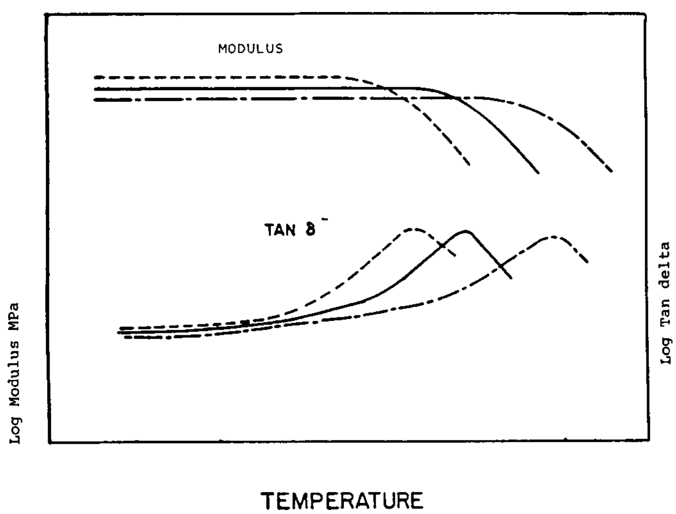

FIG. 1 Dynamic Mechanical Modulus in Torsion as a Function of Temperature

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 5279–99:
Revised Precision and Bias statement.
(*2*) Added Summary of Changes.

D 5279–01:
(*1*) Title was changed.
(*2*) Footnote 2 from the previous revision was deleted.