Designation: D5024 – 15

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

This standard is issued under the fixed designation D5024; 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 outlines the use of dynamic mechanical instrumentation for determining and reporting the viscoelastic properties of thermoplastic and thermosetting resins as well as composite systems in the form of cylindrical specimens molded directly or cut from sheets, plates, or molded shapes. The compression data generated may be used to identify the thermomechanical properties of a plastics material or composition using a variety of dynamic mechanical instruments.

1.2 This test method is intended to provide a means for determining the thermomechanical properties (as a function of a number of viscoelastic variables) for a wide variety of plastic materials using nonresonant, forced-vibration techniques as outlined in Practice D4065. Plots of the elastic (storage) modulus, loss (viscous) modulus, complex modulus, 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 Due to possible instrumentation compliance, the data generated are intended to indicate relative and not necessarily absolute property values.

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

1.7 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.8 *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 known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D4000 Classification System for Specifying Plastic Materials

D4065 Practice for Plastics: Dynamic Mechanical Properties: Determination and Report of Procedures

D4092 Terminology for Plastics: Dynamic Mechanical Properties

3. Terminology

3.1 *Definitions*—For definitions applicable to this test method refer to Terminology Standard D4092.

4. Summary of Test Method

4.1 This test method covers the determination of the compressive modulus of both solid and cellular plastics using dynamic mechanical techniques. A test specimen of cylindrical cross section is tested in dynamic compression. The specimen may be secured using appropriate grip fixtures or simply positioned between two parallel, flat plates or disks. The specimen of known geometry is placed in mechanical, linear displacement at fixed frequencies with a linear temperature variation or at variable frequencies at isothermal conditions. The elastic moduli or loss moduli, or both, of the polymeric material system are measured in compression.

NOTE 2—The particular method for measurement of the elastic and loss moduli and tan delta depends upon the individual instrument's operating principles.

5. Significance and Use

5.1 This test method provides a simple means of characterizing the thermomechanical behavior of plastic compositions using very small amounts of material. The data obtained can be

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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.

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

used for quality control and/or research and development purposes. For some classes of materials, such as thermosets, it can also be used to establish 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 these variables provide graphical representation 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:

5.3.1 Modulus as a function of temperature,

5.3.2 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,

5.3.6 The effects of formulation additives which might affect processability or performance,

5.3.7 The effects of annealing on modulus and glass transition temperature,

5.3.8 The effect of aspect ratio on the modulus of fiber reinforcements, and

5.3.9 The effect of fillers, additives on modulus and glass transition temperature.

5.4 Before proceeding with this test method, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters, or combination thereof, covered in the relevant ASTM materials specification shall take precedence over those mentioned in this test method. If there are no relevant ASTM material specifications, then the default conditions apply.

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 maintain a cylindrical test specimen of a polymeric material system so that the material acts as the elastic and dissipative element in a mechanically driven linear-displacement system. These dynamic mechanical instruments generally operate in a forced, constant strain amplitude testing mode at a fixed frequency.

7.2 The apparatus shall consist of the following:

7.2.1 *Fixed Member*—A fixed or essentially stationary member carrying one flat plate, disc, or clamp.

7.2.2 *Movable Member*—A movable member carrying a second flat plate, disc, or clamp.

7.2.3 *Flat Plate or Parallel Discs and Clamps*—The fixtures are used to hold, support, and compress the test specimen between the fixed member and the movable member. These fixtures 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 freely into alignment as soon as any load is applied, so that the minor axis of the test specimen will coincide with the direction of the applied load through the center line of the fixture assembly.

7.2.3.1 The test specimen shall be held in such a way that slippage relative to the flat plates is prevented as much as possible.

7.2.4 *Linear Deformation (strain)*—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 I of Practice D4065).

7.2.5 *Detectors*—A device or 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 *d* + 1°C, frequency to ±1 %, strain 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 if appropriate.

8. Test Specimens

8.1 The test specimens may be cut from molded shapes, or may be molded to the desired finished dimensions. Typically, the cylindrical test specimen is up to 25 mm (1.0 in.) in diameter by 5 mm (0.2 in.) in height (thickness). Cylindrical specimens of other dimensions can be used but should be clearly identified in the report.

9. Calibration

9.1 Calibrate the instrument using procedures recommended by the manufacturer.

10. Conditioning

10.1 Condition the test specimen at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 10 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D618 unless otherwise specified by contract or relevant ASTM material specification.

11. Procedure

11.1 Use an untested specimen for each measurement. Measure the diameter and height (thickness) of the specimen to the nearest 0.03 mm (0.001 in.) at the center of the specimen.

11.2 Compress the test specimen between the movable and stationary members.

11.3 Pre-load 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 (0.001 in.) the jaw separation between the movable and stationary fixtures.

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

11.6 Select the linear displacement amplitude within the linear elastic region of the material being tested. If the linear elastic region is not known, perform a strain sweep at ambient temperature to determine an appropriate amplitude.

11.7 *Temperature Sweep:*

11.7.1 Temperature variation should be controlled to 1 to 2°C/min for linear increases and 2 to 5°C/min with a minimum of 3-min thermal soak time for step increases. This will allow proper characterizing of the measured variables from the glassy region, through the glass-transition region, up to the softening or leathery-rubbery state.

11.7.2 The tan delta peak shall coincide with the dramatic change in modulus through the glass transition region.

12. Calculation

12.1 The equations listed in Practice D4065 are used to calculate the following important rheological properties measured in forced, nonresonant dynamic displacement where:

E' = storage (elastic) modulus in bending,
E'' = loss (viscous) modulus in bending,
E^* = complex modulus in bending, and
$Tan\delta$ = 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 processing, or thermal history, or both.

13.1.2 Description and direction of cutting and loading specimen, including pre-load 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 if appropriate,

13.1.7 Diameter and height (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; heat rate if appropriate,

13.1.12 Number of specimens tested,

13.1.13 Table of data and results, including moduli and tan delta as a function of temperature, frequency, strain, or time (as appropriate), and

13.1.14 A plot of the modulus (moduli) and tan delta as a function of temperature (see Fig. 1), frequency, strain, or time (as appropriate).

TABLE 1 Specimen (Elastic Modulus, G', E6 Pa)

Temperature	A	B	C	D	E
50°C	3238	8512	3303	2847	2633
75°C	2771	1159	2242	2061	1953
100°C	1805	1560	1578	1635	1544

14. Precision and Bias

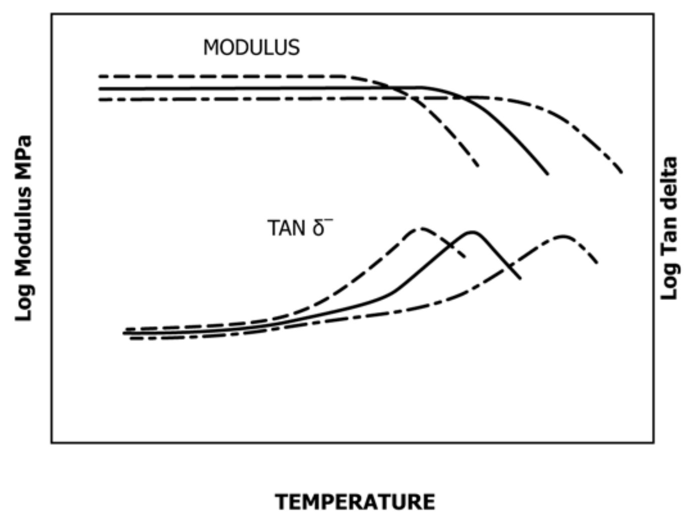

FIG. 1 Dynamic Mechanical Properties in Compression (At Varying Frequencies)

14.1 One technician in a single laboratory conducted an analysis of the repeatability of the dynamic mechanical compressive properties of a plasticized (flexible) PVC homopolymer compound. The specimen was treated to a thermal gradient of 4°C/min using a dynamic frequency of six radians/s.

15. Keywords

15.1 compression; dynamic mechanical rheological properties; elastic; linear displacement; loss; storage modulus; tan delta; viscoelastic behavior

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D5024 - 07) that may impact the use of this standard. (July 1, 2015)

(1) Revised Note 1, 4.1, 10.1, 12.1, and 13.1.11.

(2) Added 5.3.7, 5.3.8, and 5.3.9.