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Designation: D 5024 – 95a01

An American National Standard

# Standard Test Method for Measuring the <u>Plastics</u>: Dynamic Mechanical Properties of Plastics in <u>Properties</u>: In Compression<sup>1</sup>

This standard is issued under the fixed designation D 5024; 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 ( $\epsilon$ ) 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 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 means for determining the modulus as a function of temperature of a wide variety of plastics materials 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 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-is 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 similar or equivalent ISO standard.

#### 2. Referenced Documents

2.1 ASTM Standards:

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing<sup>2</sup>

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

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<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D=20 on Plastics and is the direct responsibility of Subcommittee D20.10 on Mechanical Properties. Current edition approved-Oet: September 10, 1995: 2001. Published-December 1995. November 2001. Originally published as D 5024 – 89. Last previous edition D 5024 – 95a.

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D 4000 Classification System for Specifying Plastic Materials<sup>3</sup>

D 4065 Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics<sup>3</sup>

D 4092 Terminology Relating to Dynamic Mechanical Measurements on Plastics<sup>3</sup>

#### 3. Terminology

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

#### 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 is gripped between two flat plates or parallel disks. The specimen of known geometry is placed in mechanical linear tensile displacement at fixed frequencies at either isothermal conditions or with a linear temperature increase. 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 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 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, and,
- 5.3.6 The effects of formulation additives which might affect processability or performance.

5.4 For many materials, there may be a specification that requires the use of this test method, but with some procedural modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Table 1 of Classification System D 4000 lists the ASTM materials standards that currently exist.

#### 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 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 operate in a forced, constant strain amplitude, fixed frequency testing mode.

7.2 The apparatus shall consist of the following:

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

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

7.2.3 *Flat Plate or Parallel Discs*—The plates are used to hold, support, and compress 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 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 pull through the center line of the flat plate 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 D 4065).

<sup>&</sup>lt;sup>2</sup> Dynamic Mechanical Instrumentation is available from Rheometrics, Inc., Piscataway, NJ 08854 and The Perkin-Elmer Corp., 761 Main Avenue, Norwalk, CT 06859-0256, (203) 762-1000.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 08.0+2.

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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 measureable with a precision of  $d + 1^{\circ}$ C, frequency to  $\pm 1$ %, and force to  $\pm 1$ %.

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 test specimens may be cut from sheets, plates, or 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  $23 \pm 2^{\circ}$ C (73.4  $\pm 3.6^{\circ}$ F) and  $50 \pm 5$  % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618 for those tests where conditioning is required.

#### 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 pre-loading.
- 11.4 Measure to the nearest 0.03 mm (0.001 in.) the jaw separation between the movable and stationary flat plates.
- 11.5 Select the desired frequency (or frequencies) for dynamic linear displacement.
- 11.6 Select the linear displacement amplitude.
- 11.7 Temperature Sweep:

11.7.1 Temperature increases should be controlled to 1 to  $2^{\circ}$ C/min for linear increases and 2 to  $5^{\circ}$ C/min with a minimum of 3-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 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 D 4065 are used to calculate the following important rheological properties measured in forced, nonresonant dynamic displacement:

- 12.1.1 Storage (elastic) modulus in compression, E',
- 12.1.2 Loss (viscous) modulus in compression, E",
- 12.1.3 Complex modulus in compression,  $E^*$ , and
- 12.1.4 Tan delta, *d*\*.

#### 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 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,
- 13.1.7 Diameter or 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,
- 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

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

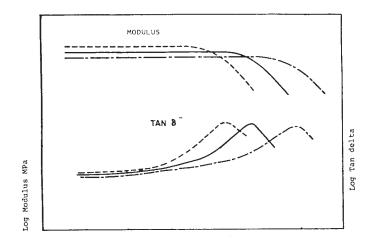
#### 14. Precision and Bias

14.1 The precision and bias of this test method are under investigation by

<u>14.1</u> One technician in a task group single laboratory conducted an analysis of Section D20.10.15. Anyone wishing to participate in this work may contact the Chairman, Section D20.10.15, ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428. 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 (almost 1 Hz).

#### 15. Keywords

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



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

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TABLE 1 S	Specimen (	Elastic	Modulus,	<b>G</b> ',	<b>E6</b>	Pa)	1
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Temperature	<u>A</u>	B	<u>C</u>	D	E
50°C	<u>3 238</u>	8 512	3 303	2 847	2 633
75°C	2 771	1 159	2 242	2 061	1 953
100°C	1 805	1 560	1 578	1 635	1 544

#### 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* 5024 – 01:

(1) Title changed.

(2) Precision and Bias statement added.

(3) Summary of Changes section added.

(4) Deleted Footnote 2 from previous revision.

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