Designation: D4065 – 06

StandardPractice for Plastics: Dynamic Mechanical Properties: Determination and Report of Procedures [1]

This standard is issued under the fixed designation D4065; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This practice is for general use in gathering and reporting dynamic mechanical data. It incorporates laboratory practice for determining dynamic mechanical properties of plastic specimens subjected to various oscillatory deformations on a variety of instruments of the type commonly called dynamic mechanical analyzers or dynamic thermomechanical analyzers.

1.2 This practice is intended to provide means of determining the transition temperatures, elastic, and loss moduli of plastics over a range of temperatures, frequencies, or time, by free vibration and resonant or nonresonant forced vibration techniques. Plots of elastic and loss moduli are indicative of the viscoelastic characteristics of a plastic. These moduli are functions of temperature or frequency in plastics, and change rapidly at particular temperatures or frequencies. The regions of rapid moduli change are normally referred to as transition regions.

1.3 The practice is primarily useful when conducted over a range of temperatures from −160°C to polymer degradation and is valid for frequencies from 0.01 to 1000 Hz.

1.4 This practice is intended for materials that have an elastic modulus in the range from 0.5 MPa to 100 GPa [73 psi to 1.5×10^7 psi].

1.5 Discrepancies in results are known to arise when obtained under differing experimental conditions. Without changing the observed data, reporting in full (as described in this practice) the conditions under which the data were obtained will enable apparent differences observed in another study to be reconciled. An assumption of this technique is that testing is conducted in the region of linear viscoelastic behavior.

1.6 Different modes of deformation, such as tensile, bending and shear, are used, as listed in the referenced test methods.

1.7 Test data obtained by this practice are relevant and appropriate for use in engineering design.

1.8 The values stated in SI units are to be regarded as standard. The values given in brackets are for information only.

1.9 *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 practice to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 8.

NOTE 1—This practice is equivalent to ISO 6721–1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D4000 Classification System for Specifying Plastic Materials
D4092 Terminology for Plastics: Dynamic Mechanical Properties
D4440 Test Method for Plastics: Dynamic Mechanical Properties Melt Rheology
D5023 Test Method for Plastics: Dynamic Mechanical Properties: In Flexure (Three-Point Bending)
D5024 Test Method for Plastics: Dynamic Mechanical Properties: In Compression
D5026 Test Method for Plastics: Dynamic Mechanical Properties: In Tension
D5279 Test Method for Plastics: Dynamic Mechanical Properties: In Torsion
D5418 Test Method for Plastics: Dynamic Mechanical Properties: In Flexure (Dual Cantilever Beam)
E1867 Test Method for Temperature Calibration of Dynamic Mechanical Analyzers
E2254 Test Method for Storage Modulus Calibration of Dynamic Mechanical Analyzers
E2425 Test Method for Loss Modulus Conformance of Dynamic Mechanical Analyzers

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.10 on 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.

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

2.2 *ISO Standard:*
ISO 6721–1 Plastics— Determination of Dynamic Mechanical Properties, Part 1, General Principles[3]

3. Terminology

3.1 *Definitions*—For definitions of terms relating to this practice, see Terminology D4092.

4. Summary of Practice

4.1 A specimen of known geometry is placed in mechanical oscillation either at fixed or natural resonant frequencies. Elastic or loss moduli, or both of the specimen are measured while varying time, temperature of the specimen or frequency of the oscillation, or both the latter. Plots of the elastic or loss moduli, or both, are indicative of viscoelastic characteristics of the specimen. Rapid changes in viscoelastic properties at particular temperatures, times, or frequencies are normally referred to as transition regions.

NOTE 2—The particular method for measurement of elastic and loss moduli depends upon the operating principle of the instrument used.

4.2 D5023, D5024, D5026, D5279, and D5418 describe specific methods for determining dynamic mechanical properties.

5. Significance and Use

5.1 Dynamic mechanical testing provides a method for determining elastic and loss moduli as a function of temperature, frequency or time, or both. A plot of the elastic modulus and loss modulus of material versus temperature provides a graphical representation of elasticity and damping as a function of temperature or frequency.

5.2 This procedure can be used to locate transition temperatures of plastics, that is, changes in the molecular motions of a polymer. In the temperature ranges where significant changes occur, elastic modulus decreases rapidly with increasing temperature (at constant or near constant frequency) or increases with increasing frequency (at constant temperature). A maximum is observed for the loss modulus, as well as for the tan delta curve, in the transition region.

5.3 This procedure can be used, for example, to evaluate by comparison to known reference materials or control materials:
5.3.1 Degree of phase separation in multicomponent systems,
5.3.2 Filler type, amount, pretreatment, and dispersion, and
5.3.3 Effects of certain processing treatment.

5.4 This procedure can be used to determine the following:
5.4.1 Stiffness of polymer composites, especially as a function of temperature,
5.4.2 Degree of polymer crystallinity, and
5.4.3 Magnitude of triaxial stress state in the rubber phase of rubber modified polymers.

5.5 This procedure is useful for quality control, specification acceptance, and research.

5.6 Procedural modifications in material specifications take precedence to this practice. Therefore, consult the appropriate material specification before using this practice. Table 1 of Classification System D4000 lists the ASTM materials standards that currently exist.

6. Interferences

6.1 Since small quantities of specimen are used, it is essential that the specimens be homogeneous or representative, or both.

7. Apparatus

7.1 The function of the apparatus is to hold a plastic specimen of uniform cross section, so that the specimen acts as the elastic and dissipative element in a mechanically oscillated system. Instruments of this type are commonly called dynamic mechanical or dynamic thermomechanical analyzers. They typically operate in one of seven oscillatory modes: (*1*) freely decaying torsional oscillation, (*2*) forced constant amplitude, resonant, flexural oscillation, (*3*) forced constant amplitude, fixed frequency, compressive oscillation, (*4*) forced constant amplitude, fixed frequency, flexural oscillation, (*5*) forced, constant amplitude, fixed frequency, tensile oscillation, (*6*) forced constant amplitude, fixed frequency, torsional oscillation and (*7*) forced constant amplitude, fixed frequency, or variable frequency dual cantilever.

7.2 The apparatus shall consist of the following:
7.2.1 *Clamps*—A clamping arrangement that permits gripping of the sample.
7.2.2 *Oscillatory Deformation (Strain)*—A device for applying an oscillatory deformation (strain) to the specimen. The deformation (strain) shall be applied and then released, as in free-vibration devices, or continuously applied, as in forced-vibration devices (see Table 1).
7.2.3 *Detectors*—A device or devices for determining dependent and independent experimental parameters, such as force (stress or strain), frequency, and temperature. Temperature shall be measurable with an accuracy of ±1°C, frequency to ±1 %, and force to ±1 %.
7.2.4 *Temperature Controller and Oven*—A device for controlling the specimen temperature, either by heating (in steps or ramps), cooling (in steps or ramps), or maintaining a constant specimen environment. Any temperature programmer should be sufficiently stable to permit measurement of sample temperature to ±0.5°C.

7.3 Nitrogen or other gas supply for purging purposes.

7.4 Calipers or other length-measuring device capable of measuring to an accuracy of ±0.01 mm.

8. Hazards

8.1 *Precautions:*
8.1.1 Certain materials, when heated near their decomposition point, can release potentially toxic, or corrosive effluents, or both that can be harmful to personnel or to the apparatus.
8.1.2 Buckling of the clamped specimen due to thermal expansion during the test.

[3] Available from American National Standards Institute, 25 W. 43rd St., New York, NY 10036.

9. Test Specimens

9.1 Specimens are of any uniform size or shape but are ordinarily analyzed in rectangular form. If some heat treatment is applied to the specimen to obtain this preferred analytical form, this treatment shall be noted in the report.

9.2 Due to the numerous types of dynamic mechanical instruments, specimen size is not fixed by this practice. In many cases, a specimen of 0.75 by 9.4 by 50 mm [0.03 by 0.38 by 2.0 in.] is found to be usable and convenient.

NOTE 3—It is important to select a specimen size consistent with the modulus of the material under test and capabilities of the measuring apparatus. For example, while thick specimens of low modulus materials are suitable for measurement, thin specimens of high modulus materials are required.

9.3 Unless otherwise specified in the appropriate material specification, condition the specimen at a set temperature of 23°C [73°F] that is maintained ±2°C [±4°F] and at a set relative humidity of 50 % that is maintained ±5 % for not less than 40 h prior to test in accordance to Procedure A of Practice D618, for those tests where conditioning is required. If other specimen conditioning is used, it should be noted in the report.

10. Calibration

10.1 Using the same heating rate or schedule to be used for specimens, calibrate the instrument temperature axis, using the instrument manufacturer's procedures with either or both of the following substances. Refer to E1867, E2254, and E2425 for additional details on calibration.

Standard	Transition Temperature, °C	Type of Transition
Water	0.0	fusion
Indium	156.6	fusion

11. Procedure

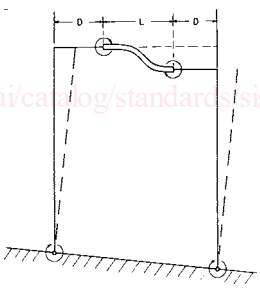
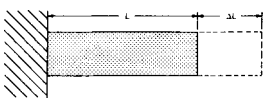
11.1 Measure the length, width, and thickness of the specimen to an accuracy of ±1 %.

11.2 Maximum strain amplitude shall be within the linear viscoelastic range of the material. Strains of less than 1 % are recommended.

11.3 If temperature is to be the independent variable:

11.3.1 The test frequency shall be from 0.01 to 500 Hz, fixed or changing as the dependent variable.

TABLE 1 Summary of Techniques and Calculations Used to Determine Dynamic Mechanical Properties

Technique	Input Excitation	Mode of Oscillation	Frequency Range, Hz	Specimen Size, mm	Calculations		
					Oscillating Strain	Elastic Component	Damping Component
Dynamic mechanical analyzer	Sinusoidal/ fixed or resonance frequency	Forced constant amplitude-fixed or resonance frequency flexural oscillation 	0.001 to 60 Hz	t = 0.01–1.6 b = 0.02–13 L = 18, 25, or 33	$\pm 3tA\,(2D+L)/L^2R$	Rectangular: $E' = \frac{4\pi^2 f^2 I-H}{2b(L/2+D)^2}\,[L/t]^3$ Circular: $E' = 4\pi^2 f^2 I\text{-}H/3r^4\,(2D+L)^2\,[2L^3]$	Tan δ = JV/f^2
Visco-elastometer[A]	Sinusoidal fixed frequency	Forced constant amplitude-fixed frequency-tensile oscillation (see Fig. 4)	3.5, 11, 35, 110	L = 7 cm T = 0.05 cm B = 0.4 cm	$\Delta L/L$	Rectangular cross section: $E' = NL:/\,bt\Delta\,L\,\cos\delta$ Circular cross section:	$E'' = NL/\,tb\Delta L\cdot\sin\delta$ Tan δ directly read
					$\Delta L/L$	$E' = NL\cos\delta/\pi r^2\,\Delta L$	$E'' = NL\sin\delta/\pi r^2\,\Delta L$ Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal fixed or variable frequency	Forced constant amplitude; fixed or variable frequency-tensile oscillation (see Fig. 5)	0.0016 to 80	t = 0.025–1.0 b = 12.7 L = 63.5	$\Delta L/L$	Rectangular cross section: $E' = NL\cos\delta/bt\,\Delta/\,L$ Circular cross section:	$E'' = NL\sin\delta/tb\Delta\,L$ Tan δ directly read