Designation: ~~D4440 – 08~~ D4440 – 15

Standard Test Method for Plastics: Dynamic Mechanical Properties Melt Rheology[1]

This standard is issued under the fixed designation D4440; 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 in determining and reporting the rheological properties of thermoplastic resins and other types of molten polymers. It may be used as a method for determining the complex viscosity and other significant viscoelastic characteristics of such materials as a function of frequency, strain amplitude, temperature, and time. Such properties may be influenced by fillers and other additives.

1.2 It incorporates a laboratory test method for determining the relevant rheological properties of a polymer melt subjected to various oscillatory deformations on an instrument of the type commonly referred to as a mechanical or dynamic spectrometer.

1.3 This test method is intended to provide a means of determining the rheological properties of molten polymers, such as thermoplastics and thermoplastic elastomers over a range of temperatures by nonresonant, forced-vibration techniques. Plots of modulus, viscosity, and tan delta as a function of dynamic oscillation (frequency), strain amplitude, temperature, and time are indicative of the viscoelastic properties of a molten polymer.

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

1.5 This test method is intended for homogenous and heterogeneous molten polymeric systems and composite formulations containing chemical additives, including fillers, reinforcements, stabilizers, plasticizers, flame retardants, impact modifiers, processing aids, and other important chemical additives often incorporated into a polymeric system for specific functional properties, and which could affect the processability and functional performance. These polymeric material systems have molten viscosities typically less than 10^6 Pa·s (10^7 poise).

1.6 Apparent discrepancies may arise in results obtained under differing experimental conditions. Without changing the observed data, reporting in full (as described in this test method) the conditions under which the data was obtained may enable apparent differences observed in another study to be reconciled.

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

1.8 The values stated in SI units are to be regarded as the standard. The values given in parentheses 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 standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—This test method is equivalent to ISO 6721, Part 10.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standard:*[3]

ISO 6721, Part 10 ~~Plastics— Determination~~ Plastics—Determination of Dynamic Mechanical Properties, Part 10, Complex Shear Viscosity Using a Parallel-Plate Oscillatory Rheometer

[1] This test method 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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology Standard D4092.

4. Summary of Test Method

4.1 A known amount of thermoplastic polymer (molten powder or pellet, or solid preform disk) is placed in mechanical oscillation at a fixed or varying frequency at isothermal conditions or over a linear temperature increase or a time-temperature relation simulating a processing condition. Storage (elastic) modulus, G' or loss (viscous) modulus, G'', or both, or the corresponding dynamic viscosity functions $n' = g''/w$ and $n'' = g'/w$, of the polymeric material specimen are measured in shear as a function of frequency, strain, temperature, or time.

5. Significance and Use

5.1 This test method provides a simple means of characterizing the important rheological properties and viscosity of thermoplastic polymers using very small amounts of material (approximately 25 to 50 mm in diameter by 1 to 3 mm in thickness ... approximately 3 to 5 g). Data ~~may be~~are generally used for quality control, research and development, and establishment of optimum processing conditions.

5.2 Dynamic mechanical testing provides a sensitive method for determining molten polymer properties by measuring the elastic and loss moduli as a function of frequency, strain, temperature, or time. Plots of viscosity, storage, and loss moduli, and tan delta as a function of the aforementioned process parameters provide graphical representation indicative of molecular weight, molecular weight distribution, effects of chain branching, and melt-processability for specified conditions.

5.3 Values obtained in this test method can be used to assess the following:

5.3.1 Complex viscosity of the polymer melt as a function of dynamic oscillation,

5.3.2 Processing viscosity, minimum as well as changes in viscosity as a function of experimental parameters,

5.3.3 Effects of processing treatment,

5.3.4 Relative polymer behavioral properties, including viscosity and damping, and

5.3.5 Effects of formulation additives that might affect processability or performance.

5.4 Before proceeding with this test method, ~~reference should be made~~ refer to the specification ~~of~~for 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 the test method. If there are no relevant ASTM material specifications, then the default conditions apply.

6. Interferences

6.1 Since small quantities of polymer are used, it is essential that the specimens be homogeneous and representative.

6.2 Toxic or corrosive effluents, or both, ~~may~~ have the potential to be released when heating the polymer specimen to its molten state and could be harmful to personnel or to the instrumentation.

6.3 Entrapped air/gas ~~may~~ has the potential to affect the results obtained using powder or pellet-type samples.

7. Apparatus

7.1 The function of the apparatus is to hold a molten polymer of known volume and dimensions so that the material acts as the elastic and dissipative element in a mechanically driven oscillatory system, as outlined in Practice D4065. These instruments operate in one or more of the following modes for measuring rheological behavior in dynamic oscillatory shear: (*1*) forced constant amplitude, fixed frequency, (*2*) forced constant amplitude, varying frequency, and (*3*) forced varying amplitude, fixed frequency.

7.2 The apparatus shall consist of the following:

7.2.1 *Test Fixtures*—A choice of either polished cone and plate (having a known cone angle) or parallel plates having either smooth, polished, or serrated surfaces. Variations of this tooling, such as bottom plates with concentric overflow rims, can be used as necessary.

7.2.2 *Oscillatory Deformation (Strain)*—A device for applying a continuous oscillatory deformation (strain) to the specimen.

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 should be measurable~~ Measure temperature with a precision of ±1°C, frequency to ±1 %, strain 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, or a combination thereof. Fig. 1 illustrates several time-temperature profiles. A temperature programmer ~~should be~~that is sufficiently stable to permit measurement of sample temperature to 1°C.

7.3 *Nitrogen,* or other gas supply for purging purposes, if appropriate.

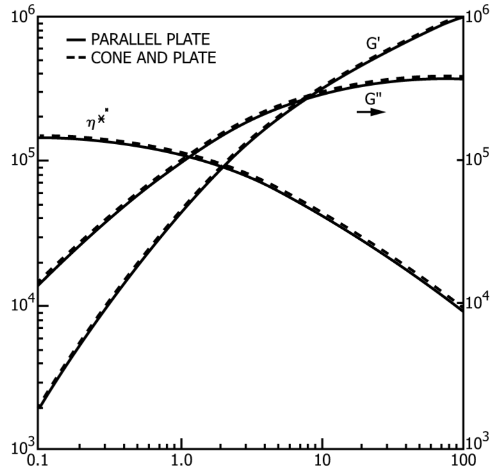

FIG. 1 Rheological Properties of a Polymer Melt

8. Test Specimens

8.1 The molten polymer composition ~~should~~shall be both homogeneous and representative.

8.2 Due to various geometries that might be used for dynamic mechanical characterization of molten polymeric systems, size is not fixed by this test method; however, sample geometry (diameter and thickness) ~~should~~shall be reported for any series of comparisons.

8.3 Serrated tooling ~~might be used~~is an option for materials exhibiting interfacial slippage due to high modulus (as when approaching a solidified state).

9. Calibration

9.1 Calibrate the instrument using procedures recommended by the manufacturer.

10. Procedure

10.1 Lower the upper test fixture so that it is just touching the bottom fixture. Zero the gap indicator dial.

10.2 If a dynamic temperature sweep (linear heating rate or ramp temperature scan) is required for the specimen, then the gap setting must be corrected for the thermal expansion of the support fixtures during testing.

10.2.1 Determine the thermal expansion of the fixtures at the temperature sweep conditions to be used during testing. Record the gap-setting reading at the time and temperature corresponding to computer calculation of the viscoelastic properties, while maintaining a fixed normal force between the test fixtures.

10.2.2 Plot the gap-separation reading, due to thermal expansion of the fixtures, as a function of temperature.

10.2.3 Adjust the upper test fixture during the test in order to maintain a fixed sample thickness, if necessary.

10.3 Apply an adequate amount of polymer material onto the test fixture. Be certain that there is sufficient material to cover the bottom plate uniformly.

10.4 Bring down the upper test fixture so that it is touching the polymeric material.

10.4.1 A gap setting from 1 to 3 mm is a good operating range for parallel plate geometry. This gap setting is arbitrary and dependent on the type of material being characterized. A gap setting of 0.5 mm would be a minimum. However, when large platens and low-viscosity materials are being used, the recommended minimum gap setting is 0.25 mm.

~~10.4.2 Cone and plate experiments should be conducted at an isothermal temperature. Any change in the temperature setting will require adjustment of the gap at the new temperature.~~

10.4.2 Remove excess material flush to the test fixtures using a razor blade, spatula, knife, or hot soldering iron, as appropriate.

10.5 *Isothermal Evaluations at Elevated Temperature:*

10.5.1 In cases where the specimen is introduced directly into the test chamber at elevated temperatures, preheat and stabilize the chamber to the desired temperature prior to introducing the test specimen.

10.5.2 Cone and plate experiments are generally conducted at an isothermal temperature. Any change in the temperature settings require adjustment of the gap at the new temperature.

10.6 ~~*Isothermal Evaluations at Elevated Temperature:*~~ *Ramped or Simulated Process Program Heating—*

~~10.5.1 In cases where the specimen can be introduced directly into the test chamber at elevated temperatures, preheat and stabilize the chamber to the desired temperature prior to introducing the test specimen.~~ For materials that are to be characterized