Designation: 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 of Dynamic Mechanical Properties, Part 10, Complex Shear Viscosity Using a Parallel-Plate Oscillatory Rheometer

3. Terminology

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

[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.

Current edition approved Jan. 15, 2015. Published February 2015. Originally approved in 1984. Last previous edition approved in 2008 as D4440 - 08. DOI: 10.1520/D4440-15.

[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**

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 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, refer to the specification 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, 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 has the potential to affect the results obtained using powder or pellet-type samples.

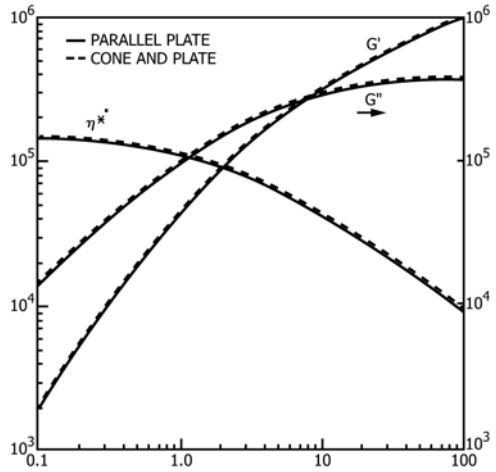

FIG. 1 Rheological Properties of a Polymer Melt

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. 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 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.

8. Test Specimens

8.1 The molten polymer composition 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) shall be reported for any series of comparisons.

8.3 Serrated tooling 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 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 *Ramped or Simulated Process Program Heating*—For materials that are to be characterized starting at a low temperature, and controlled for either a linear ramp or step-and-hold function, apply the material to the test tooling and the test chamber closed and heated at the desired rate. It is recommended that temperature be monitored during this heat-up. Thermal gradients of 3 to 5°C/min are recommended for measuring the rheological properties. For both isothermal and simulated processing conditions, discontinue measurements when the polymeric composition exhibits deterioration, degradation, or decomposition since the degradation of the polymer will affect the test results.

10.7 Maximum strain amplitude shall be within the linear viscoelastic range of the material. Automated strain sweeps conducted to determine the strain sensitivity of the polymeric material are recommended. This is especially helpful for characterizing the effects of fillers and for monitoring crystallization as the molten polymer slowly cools down.

NOTE 2—There are several devices capable of testing in the non-linear viscoelastic region for obtaining long chain branching information. Higher strains can be used as long as the device is capable of repeatable measurements and a harmonic analysis is applied to the resulting signal.

10.8 Duplicate measurements are recommended.

11. Calculation

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

11.1.1 Storage (elastic) modulus, G',
11.1.2 Loss (viscous) modulus, G",
11.1.3 Tan delta, δ,
11.1.4 Complex modulus, G*,
11.1.5 Complex viscosity, η*, and
11.1.6 Dynamic viscosity, n', n".

11.2 Plot the moduli, tan delta, and viscosity as a function of either frequency, strain amplitude, temperature, or time, as required. An example of typical data representation is shown in Fig. 1.

12. Report

12.1 Report the following information:

12.1.1 Complete identification and description of the material tested including the name, stock or code number, date made, form, source, etc., if available.

12.1.2 Description of the instrument used for the test.

12.1.3 Dimensions of the sample geometry and type of tooling.

12.1.4 Description of the calibration procedure.

12.1.5 Identification of the sample atmosphere by gas composition, purity, and rate used, if appropriate.

12.1.6 Details of conditioning the specimen prior to test.

12.1.7 The temperature used in the analysis, the thermal gradient if any, and the time for the specimen to reach equilibrium.

12.1.8 Table of data and results, including the moduli, complex viscosity, and tan delta as a function of the dynamic oscillation (frequency), percent strain, temperature, or time, as appropriate.

12.1.9 Number of specimens tested.

12.1.10 A plot of the rheological behavior versus experimentally controlled independent variable(s) for multiple studies.

12.1.11 Frequency of test or frequency range.

12.1.12 Strain amplitude or range.

TABLE 1 Precision Results

Material	Grand Average	Standard Deviations		Limits	
		s_r	s_R	r	R
Oscillation Rate 10 rad/s, Temperature 190°C					
A	77608	1992	2835	5576	7939
B	6768	109	357	304	1001
C	5409	156	951	436	2664
D	14525	266	441	744	1235
E	23952	315	937	882	2624
Oscillation Rate 10 rad/s, Temperature 210°C					
A	69644	1330	2122	3724	5942
B	4875	80	656	224	1837
C	3759	210	286	587	801
D	10845	331	708	926	1982
E	19042	232	1500	651	4200
Oscillation Rate 100 rad/s, Temperature 190°C					
A	15793	338	606	947	1697
B	2788	42	157	117	439
C	2452	111	333	311	933
D	5279	74	153	206	428
E	7099	87	271	245	760
Oscillation Rate 100 rad/s, Temperature 210°C					
A	14961	292	502	818	1405
B	2172	27	272	77	761
C	1855	87	105	243	293
D	4270	99	263	277	737
E	6099	67	448	189	1255

12.1.13 Date of test.

13. Precision and Bias[4]

13.1 Table 1 is based on a round robin conducted in 2007 in accordance with Practice E691, involving five materials tested by eight laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the average of three individual determinations. Each laboratory obtained three test results for each material. (**Warning—**The explanation of "*r*" and " *R*" are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 is not to be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method need to apply the principles outlined in Practice E691 to generate data specific to their materials and laboratory (or between specific laboratories). The principles of 13.2 through 13.2.3 would then be valid for such data.)

13.2 Concept of "*r*" and " *R*" in Table 1: If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing three specimens for each test result, then:

13.2.1 *Repeatability*—Two results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material. "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

13.2.2 *Reproducibility*—Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the "*R*" value for that material. " *R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

13.2.3 Any judgment in accordance with 13.2.1 or 13.2.2 would have an approximate 95 % (0.95) probability of being correct.

13.3 There are no recognized standards by which to estimate the bias of this method.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1246.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4440 - 08) that may impact the use of this standard. (January 15, 2015)

(1) Editorial changes were made to 5.1, 5.4, 7.2.3, 7.2.4, and 8.2.
(2) Moved 10.4.2 to become 10.5.2. This moves an isothermal statement to an isothermal segment of the procedure. Subsection 10.4.3 became 10.4.2
(3) A new 10.6 was created by moving 10.5.2 out from under 10.5. Subsection 10.6 became 10.7 and subsection 10.7 became 10.8
(4) Note 2 was added to the new 10.7.
(5) Use of the permissive words "may", "should", and "can" were revised, and in some cases making the language mandatory. The revised sections were 5.1, 5.4, 6.2, 6.3, 7.2.3, 7.2.4, 8.1, 8.2, 8.3, 10.4.2, 10.5.1, 10.5.2, 10.6, 13.1