



Standard Practice for Rheological Measurement of Polymer Melts Using Dynamic Mechanical Procedures¹

This standard is issued under the fixed designation D 4440; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice covers the use of dynamic mechanical instrumentation² for use in gathering and reporting the rheological properties of thermoplastic resins. It may be used as a practice for determining the complex viscosity and 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 practice 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 mechanical or dynamic spectrometer.

1.3 This practice is intended to provide 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 practice is valid for a wide range of frequencies, typically from 0.01 to 100 Hz.

1.5 This practice 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 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 practice) the conditions under which the data were obtained will enable apparent differences observed in another study to be reconciled.

1.7 Test data obtained by this practice is 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 4000 Classification System for Specifying Plastic Materials³

D 4065 Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics³

D 4092 Terminology Relating to Dynamic Mechanical Measurements on Plastics³

3. Terminology

3.1 *Definitions:* Definitions are in accordance with Terminology D 4092.

4. Summary of Practice

4.1 A known amount of thermoplastic resin (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) G' or loss (viscous) moduli, 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 practice provides a simple means of characterizing the important rheological properties and viscosity of thermoplastic resins 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 used for quality control, research and development, and establishment of optimum

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² 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.

³ *Annual Book of ASTM Standards*, Vol 08.02.

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 practice 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 resin behavioral properties, including viscosity and damping, and

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

5.4 For many materials, there may be a specification that requires the use of this practice, 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 practice. Table 1 of Classification System D 4000 lists the ASTM materials standards that currently exist.

6. Interferences

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

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

6.3 Entrapped air/gas may affect the results obtained using powder or pellet 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 D 4065. 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 surface. 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 with a precision of $\pm 1^\circ\text{C}$, frequency to $\pm 1\%$, and force to $\pm 1\%$.

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 sufficiently stable to permit measurement of sample temperature to 1°C .

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

8. Test Specimens

8.1 The molten polymer composition should 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 practice; however, sample geometry (diameter and thickness) should be reported for any series of comparisons.

8.3 Serrated tooling might be used for materials exhibiting interfacial slippage due to high modulus.

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 touching the bottom fixture with approximately the same normal force as will be experienced during testing. Zero the gap indicator dial.

10.2 If a dynamic temperature sweep (linear heating rate or ramp temperature scan) were 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.

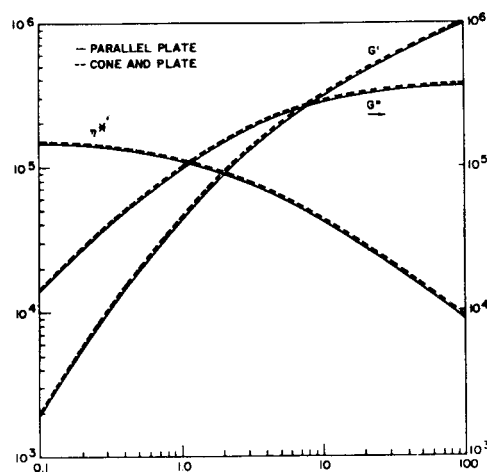


FIG. 1 Rheological Properties of a Polymer Melt

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

10.2.3 Raise the upper test fixture during the test in order to maintain a fixed sample thickness.

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 run at only one temperature. Any change in the setting will require setting the required gap at the new temperature level.

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

10.5 *Isothermal Evaluations at Elevated Temperature:*

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.

10.5.2 *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, the material should be applied to the test tooling and the test chamber closed and heated at the desired rate. Temperature should 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, continue measurements until the polymeric composition exhibits deterioration, degradation, or decomposition, since the degradation of the polymer may affect the test results.

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

10.7 Duplicate measurements are recommended.

11. Calculation

11.1 The following equations listed in Practice D 4065 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. 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.

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.

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.

12.1.9 Number of specimens tested.

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

12.1.11 Frequency of test or frequency range.

12.1.12 Strain amplitude or range.

12.1.13 Date of test.

13. Precision and Bias

13.1 A statement on precision will be developed after a series of round-robin evaluations of typical polymer melts have been conducted.

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