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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Measuring the Cure Behavior of Thermosetting Resins Using Dynamic Mechanical Procedures[1]

This standard is issued under the fixed designation D 4473; 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 practice covers the use of dynamic-mechanical-oscillation instrumentation[2] for gathering and reporting the thermal advancement of cure behavior of thermosetting resin. It may be used for determining the cure properties of unsupported resins and resins supported on substrates and subjected to various oscillatory deformations. These deformations may be in shear or the dynamic compression of supported resins using a dynamic mechanical instrument.

1.2 This practice is intended to provide means for determining the cure behavior of supported and unsupported thermosetting resins over a range of temperatures by free vibration and resonant and nonresonant forced-vibration techniques, in accordance with Practice D 4065. Plots of modulus, cure behavior, tan delta, and damping index as a function of time/temperature are indicative of the thermal advancement or cure characteristics of a resin.

1.3 This practice is valid for a wide range of frequencies, typically from 0.01 to 100 Hz. However, it is strongly recommended that low-frequency test conditions, generally below 1.5 Hz, will generate more definitive cure-behavior information.

1.4 This practice is intended for resin/substrate composites that have an uncured effective elastic modulus in shear greater than 0.5 MPa.

1.5 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 practice) the conditions under which the data were obtained.

1.6 Due to possible instrumentation compliance, especially in the compressive mode, the data generated may indicate relative and not necessarily absolute property values.

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.

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.* Specific precautionary statements are given in Note 5.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 4000 Classification System for Specifying Plastic Materials[3]

D 4065 Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics[3]

D 4092 Terminology Relating to Dynamic Mechanical Measurements of Plastics[3]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[4]

3. Terminology

3.1 *Definitions*—For definitions applicable to this practice refer to Terminology D 4092.

4. Summary of Practice

4.1 A known amount of thermosetting liquid or resin-impregnated substrate is placed in mechanical oscillation at fixed or natural resonant frequencies at either isothermal conditions, with a linear temperature increase or a time-temperature relation simulating a processing condition. The elastic or loss modulus, or both, of the composite specimen are measured in shear as a function of time. The point in time when tan delta is maximum, and the elastic modulus levels after an increase, is calculated as the gel time of the resin under the conditions of the test.

NOTE 2—The particular method for measuring the elastic and loss

[1] This practice is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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[2] Dynamic Mechanical Instrumentation is available from Rheometrics, Inc., Piscataway, NJ 08854 and The Perkin-Elmer Corp., 761 Main Ave., Norwalk, CT 06859-0256, (203) 762-1000.

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

[4] *Annual Book of ASTM Standards*, Vol 14.02.

moduli and tan delta depends upon the individual instrument's operating principles.

5. Significance and Use

5.1 This practice provides a simple means of characterizing the cure behavior of thermosetting resins using very small amounts of material (fewer than 3 to 5 g). 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 cure characteristics by measuring the elastic and loss moduli as a function of temperature or time, or both. Plots of cure behavior and tan delta of a material versus time provide graphical representation indicative of cure behavior under a specified time-temperature profile.

5.3 This practice can be used to assess the following:

5.3.1 Cure behavior, including rate of cure, gel, and cure time.

5.3.2 Processing behavior, as well as changes as a function of time/temperature.

NOTE 3—The presence of the substrate prevents an absolute measure, but allows relative measures of flow behavior during cure.

5.3.3 The effects of processing treatment.

5.3.4 Relative resin behavioral properties, including cure behavior and damping.

5.3.5 The effects of substrate types on cure.

NOTE 4—Due to the rigidity of a supporting braid, the gel time obtained from dynamic mechanical traces will be longer than actual gel time of the unsupported resin measured at the same frequency. This difference will be greater for composites having greater support-to-polymer rigidity ratios.[5]

5.3.6 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 representative of the polymeric material being tested.

6.2 The result is a response of the thermal advancement or cure behavior of the resin in combination with any substrate used to support the resin.

7. Apparatus

7.1 The function of the apparatus is to hold a neat (unmodified) resin or uncured supported composite formulation or coated substrate of known volume and dimensions. The material acts as the elastic and dissipative element in a mechanically driven oscillatory shear or dynamic compression system. These dynamic mechanical instruments operate in one or more of the following modes for measuring cure behavior in torsional shear or dynamic compression:

7.1.1 Forced, constant amplitude, fixed frequency,

7.1.2 Forced, constant amplitude, resonant oscillation,

7.1.3 Freely decaying oscillation.

7.2 The apparatus shall consist of the following:

7.2.1 *Test Fixtures*, a choice of the following:

7.2.1.1 *Polished Cone and Plate (Having a Known Cone Angle)*—Usually a 25 or 50-mm diameter cone and plate or parallel plates are recommended for neat resins. Variations of this tooling, such as bottom plates with concentric overflow rims, may be used as necessary.

7.2.1.2 *Parallel Plates*, having either smooth, polished, or serrated surfaces are recommended for neat resins or prepregs having less than 6 % volatiles.

7.2.1.3 *Clamps*—A clamping arrangement that permits gripping of the composite sample.

7.2.2 *Oscillatory Deformation (Strain Device)*—A device for applying a continuous oscillatory deformation (strain) to the specimen. The deformation (strain) may be applied and then released, as in free-vibration devices, or continuously applied, as in forced-vibration devices (see Table 1 of Practice D 4065).

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 ±1°C, frequency to ±1 %, and force to ±1 %.

7.2.4 *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. Fig. 1 illustrates typical time-temperature profiles. A temperature controller should be sufficiently stable to permit measurement of sample temperature to within 1°C.

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

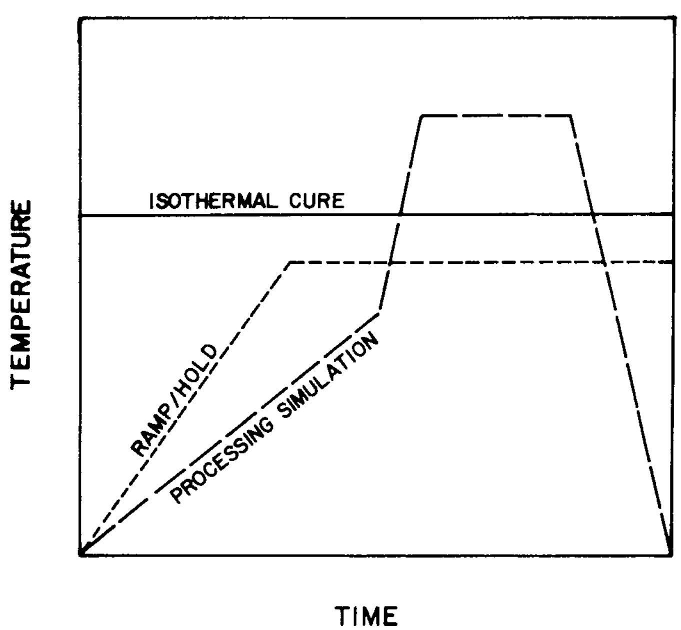

FIG. 1 Typical Temperature Profile

[5] Hedvat, S., *Polymer Engineering and Science*, Vol 21, No. 3, February 1981.