INTERNATIONAL

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Standard Test Method for Cure Behavior of Thermosetting Resins by Dynamic Mechanical Procedures using an Encapsulated Specimen Rheometer[1]

This standard is issued under the fixed designation D7750; 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 method covers the use of dynamic mechanical instrumentation for determination and reporting of the thermal advancement of cure behavior of thermosetting resin on an inert filler or fiber in a laboratory. It may also be used for determining the cure properties of resins without fillers or fibers. These encapsulated specimens are deformed in torsional shear using dynamic mechanical methods.

1.2 This method is intended to provide means for determining the cure behavior of thermosetting resins on fibers over a range of temperatures from room temperature to 250 °C by forced-constant amplitude techniques (in accordance with Practice D4065). Plots of complex modulus, complex viscosity, and damping ratio or tan delta as a function of time or temperature, or both, quantify the thermal advancement or cure characteristics of a resin or a resin on filler or fiber.

1.3 Test data obtained by this method is relevant and appropriate for optimizing cure cycles.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4.1 *Exception*—The Fahrenheit temperature measurement in 10.1 is provided for information only and is not considered standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3878 Terminology for Composite Materials

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

D4473 Test Method for Plastics: Dynamic Mechanical Properties: Cure Behavior

D6507 Practice for Fiber Reinforcement Orientation Codes for Composite Materials

D7028 Test Method for Glass Transition Temperature (DMA Tg) of Polymer Matrix Composites by Dynamic Mechanical Analysis (DMA)

E380 Practice for Use of the International System of Units (SI) (the Modernized Metric System) (Withdrawn 1997)[3]

3. Terminology

3.1 *Definitions:* For most definitions applicable to this method refer to Terminology D4092.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Encapsulated Sample Parallel Plate Rheometer*—Dynamic Mechanical Analyzer apparatus that holds the specimen under pressure within a confined cavity. The apparatus is designed to contain the resin within the specimen throughout the progress of the cure.

4. Summary of Test Method

4.1 A small circular specimen is assembled from uncured thermoset resin materials that correspond to a representation of

[1] This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor 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] The last approved version of this historical standard is referenced on www.astm.org.

a composite part. This specimen is placed in mechanical oscillation at a fixed frequency at either isothermal conditions, a linear temperature increase or a time-temperature relation simulating a processing condition. The lower plate oscillates and transmits torque from the lower plate through the sample into the upper plate. The resulting torque measured at the upper plate is converted to shear modulus using equations that compensate for the shape and size of the sample. The shear modulus is separated into a component that is in phase with the applied strain or elastic shear modulus and a component that is 90° out of phase with the applied strain or loss shear modulus. The elastic and loss modulus of the specimen are measured as a function of time. During cure, the elastic shear modulus will initially decrease as the temperature is increased due to a decrease in the viscosity of the resin in the sample. When cure occurs in the sample, the elastic shear modulus increases.

5. Significance and Use

5.1 This method provides a simple means of characterizing the cure behavior of a thermosetting resin specimen that is a representation of a composite part. The diameter of the specimen is approximately 38 mm and the thickness ranges from 2.6 mm to 3.2 mm. This corresponds to a sample volume of approximately 3 cm^3 to 4 cm^3. The data may be used for quality control, research and development, and verifying the cure within processing equipment including autoclaves.

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 provide graphical representation indicative of cure behavior under a specified time-temperature profile. The presence of fibers within the resin may change the dynamic properties measured within a material. However, it is still possible to compare different resins with the same fiber structure and obtain the relative difference due to the resin cure properties.

5.3 This method can be used to assess the following:

5.3.1 Cure behavior, as well as changes as a function of temperature or time, or both,

5.3.2 Processing behavior, as well as changes as a function of temperature or time, or both,

5.3.3 The effects of processing treatments,

5.3.4 Relative resin behavioral properties, including cure behavior, damping and impact resistance,

5.3.5 The effects of reinforcement on cure; the reinforcement can be a fiber or a filler,

5.3.6 The effects of materials used to bond the resin and reinforcement,

5.3.7 The effect of formulation additives that might affect processability or performance.

5.4 This provides a method to assess the cure properties of a thermosetting resin containing woven fiber or other reinforcing materials.

5.5 This method is valid for a wide range of oscillation frequencies typically from 0.002 Hz to 50 Hz.

NOTE 1—It is recommended that low-frequency test conditions, generally 1 Hz to 2 Hz, be used to generate more definitive cure-behavior information. Slower frequencies will miss important cure properties. Faster frequencies will reduce sensitivity to cure.

6. Interferences

6.1 Apparent discrepancies in results may arise when using different 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 method) the conditions under which the data were obtained. One essential condition within this method that must be noted is the presence of pressure within the specimen chamber which ensures good precision.

6.2 In many cases, the specimens made with this method will be significantly smaller than the parts in production. It is essential that specimens be made from representative samples of uncured material used to make parts. This will ensure that the data is representative of the part cure.

6.3 The result is a response to the thermal advancement or cure behavior of the resin. The cure behavior is also influenced by the reinforcement and materials used to enhance the bond between the resin and reinforcement.

6.4 The data will represent the cure of the system at the measured temperature. Parts are often significantly thicker than the specimen. There may be a significant difference between the temperature versus time profile on the inside and the outside of those composite parts. Thermocouples can be used to measure both temperatures during a cure process of a thick part. The measured temperature versus time data can be used to define temperature versus time profiles for dynamic cure specimens using the procedures of this standard. The results can be used to compare the cure response for the inside and the outside of the composite part.

7. Apparatus

7.1 The function of the apparatus is to hold a resin specimen with inert reinforcement, such as fibers, under pressure and yet prevent the escape of resin. Thereby, the reinforcement to resin ratio will remain constant throughout the test. The material acts as the elastic and dissipative element in a mechanically driven oscillatory shear system. This dynamic mechanical instrument operates in torsional shear using one of the following modes for measuring cure behavior:

7.1.1 Forced, constant amplitude, fixed frequency,

7.1.2 Forced, variable amplitude, fixed frequency.

7.2 The apparatus shall consist of the following:

7.2.1 Parallel plates with serrated or radial grooved surfaces. The diameter of the dies shall be 40 mm ± 2 mm. The depth of the grooves shall be limited to 1.0 mm or less to keep a constant reinforcement to resin ratio.

7.2.2 *Encapsulated Specimen Cavity*—The specimen shall be encapsulated by the two parallel plates and a series of mechanical components at the outer plate diameter designed to contain the specimen under pressure without loss of resin. These components shall include an O-ring inserted at the outer diameter of the specimen (Fig. 1). The O-ring meets ASTM International size No. 2-127.