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# Standard Test Method for Measurement of Resin Content and Other Related Properties of Polymer Matrix Thermoset Prepreg by Combined Mechanical and Ultrasonic Methods<sup>1</sup>

This standard is issued under the fixed designation D 5300; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes measurement procedures for determining physical properties of polymer matrix thermoset prepreg such as resin content, fiber volume, fiber areal weight, and thickness per ply. It provides a systematic process for measuring these physical properties using a combination of mechanical and ultrasonic methods.

1.2 The procedures are applicable to quality/process control, manufacturing acceptance and incoming inspection of prepreg composite materials.

1.3 The test method consists of a procedure for obtaining reproducible configurations for measurement of ultrasonic properties and associated geometrical properties of thermoset prepreg specimens and three calculation procedures which utilize these data to determine thickness per ply, resin content and fiber areal weight, attached as Annex A1, A2, and A3 respectively.

1.4 *This standard does not purport to address all of the safety problems, 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:

- C 613 Test Method for Resin Content of Carbon and Graphite Prepregs by Solvent Extraction<sup>2</sup>
- D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals<sup>3</sup>
- D 2584 Test Method for Ignition Loss of Cured Reinforced Resin<sup>4</sup>
- D 3529 Test Method for Resin Solids Content of Carbon Fiber-Epoxy Prepreg<sup>2</sup>
- D 3531 Test Method for Resin Flow of Carbon Fiber-Epoxy Prepreg<sup>2</sup>
- D 3800 Test Method for Density of High-Modulus Fibers<sup>2</sup>
- E 1065 Guide for Evaluating Characteristics of Ultrasonic Search Units<sup>5</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-30 on High Modulus Fibers and Their Composites and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor Properties.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 15.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 15.05.

<sup>4</sup> Annual Book of ASTM Standards, Vol 08.02.

<sup>5</sup> Annual Book of ASTM Standards, Vol 03.03.

E 1324 Guide for Measuring Some Electronic Characteristics of Ultrasonic Examination Instruments<sup>5</sup>

### 2.2 Federal Specification:

GGG-G-15C Gage Block and Accessories (Inch and Metric)<sup>6</sup>

### 2.3 Other Standards:

SPI Prepreg 1 (revised February 1962) Test Method for Resin and Volatile Content of Preimpregnated Inorganic Reinforcements<sup>7</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *ultrasonic calibration factor (Uc)*—a mathematical quantity defined as the square root of the product of ultrasonic wave transit time multiplied by the distance traveled in the material.

3.1.2 *slowness (S)*—the reciprocal of an ultrasonic wave propagation velocity. Slowness is measured in time per unit of distance, such as microseconds per millimeter.

3.1.3 *material time-of-flight ( $t_m$ )*—transit time of the ultrasonic wave through the material with respect to a selected reference in microseconds (usually the reference will be the transit time with the ultrasonic heads in contact with each other, that is, with no specimen between them).

3.1.4 *consolidation*—the process by which individual prepreg plies are compacted under pressure to produce inter-ply adhesion and eliminate voids.

3.1.5 *optimal consolidation point<sup>8</sup> ( $t_c$ )*—the point on the compressive deformation curve at which the deformation mechanism shifts from primarily void collapse to primarily matrix flow.

3.1.6 *ultrasonic heads*—the assembly which holds the ultrasonic search units.

## 4. Summary of Test Method

4.1 The test method consists of a procedure for measuring mechanical and ultrasonic properties for a given fiber-matrix system under a specified consolidation pressure to generate a material-specific calibration database. These data are used with the measured values for a specimen to calculate the property of interest.

<sup>6</sup> Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

<sup>7</sup> Available from Society of the Plastics Industry, 1275 K Street, NW, Suite 400, Washington, D.C. 20005.

<sup>8</sup> D. R. Smith, et al, *International SAMPE Technical Conference Series*, Vol 22, 1990, pp. 237–250.

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4.2 Multi-ply prepreg specimens are prepared and the specified consolidation force applied. A series of ultrasonic pulses are sent through the specimen and response waveforms are recorded. A simultaneous series of thickness measurements are also taken by mechanical means and recorded. The optimum consolidation point is determined from the thickness deformation curve. This provides a reproducible configuration from which physical properties of the thermoset prepreg specimen can be determined.

4.3 Thickness per ply is determined by direct measurement of thickness at the optimal consolidation point. Refer to Annex A1 for details.

4.4 Resin content is calculated by determining the thickness, time-of-flight, inverse velocity and ultrasonic calibration factor at the optimal consolidation point for the test specimen. These values are used to derive resin content from a pre-established material-specific calibration curve. Refer to Annex A2 for details.

4.5 The fiber volume fraction and fiber areal weight are calculated from the resin content data together with specimen area, weight and density of fiber and resin. Refer to Annex A3 for details.

## 5. Significance and Use

5.1 This test method is designed to provide a consistent technique for measurement of ultrasonic properties of multi-ply thermoset prepreg materials.

5.2 The mechanical properties of structures made with advanced composites can be sensitive to differences in the fiber/resin ratio of the material used. Depending on the material system and the application, keeping fiber/resin ratio within specification in the order of a few percent can be critical. Variations of the raw material (prepreg) can arise from changes in fiber areal weight, density of resin and fibers used, and inconsistencies in the manufacturing process.

5.3 The consistent measurement of ultrasonic properties of thermoset prepreg requires a reproducible configuration. The determination of the optimal consolidation point achieves this reproducibility. During the mechanical compression of prepreg composite material, the deformation is believed to have at least two simultaneously occurring mechanisms. The first component is a collapse and closure of the compressible inclusions such as voids. The second is the flow and extrusion of the matrix. When compression begins, the dominant mechanism of compressive deformation is void collapse. After void collapse is essentially completed, the dominant mechanism of compressive deformation is flow of the matrix, which occurs at a different rate from void collapse. The selected transition point (optimal consolidation point) provides the reference point from which other measurements may be taken for comparing physical properties of the multi-ply prepreg material.

5.4 Resin content and other related properties are used as follows:

5.4.1 Thickness per ply is used by designers and manufacturers of composites to ensure the part conforms to designed geometry and strength.

5.4.2 Resin content and fiber volume of prepreg materials are utilized in evaluating the physical and mechanical properties of composite materials.

5.4.3 Resin content of prepregs is also used to determine

the bleeder schedule during the curing process.

5.5 The thickness and ultrasonic wave propagation characteristics of prepreg depend on the fiber and matrix of which it is composed. Therefore, a material-specific calibration database must be prepared for each fiber/matrix combination.

## 6. Interferences

6.1 Conditions that could interfere with the accuracy of the measurements described in this test method include the following:

6.1.1 The prepreg must not be aged, or partially cured, and it must not exceed the recommended "out-time" (time out of refrigerated storage), so as to allow for proper consolidation under pressure at room temperature.

6.1.2 The total thickness of the multi-ply prepreg specimens must be greater than 0.76 mm (0.03 in.).

6.1.3 The fiber areal weight of the test and calibration prepreg specimens must not vary by more than  $\pm 3\%$  (this is a typical user's specification).

6.1.4 Temperature at which the measurements are made must be held constant within  $\pm 2.4^\circ\text{C}$  ( $\pm 5^\circ\text{F}$ ).

## 7. Apparatus

7.1 An experimental configuration<sup>9</sup> composed of a mechanical loading apparatus, an ultrasonic pulser-receiver, a digitizer and a computer.

7.1.1 A mechanical apparatus for applying load to the specimens shall include a displacement gauge and holders for the ultrasonic transducers above and below the specimen. The ultrasonic heads shall be made adjustable so that a maximum ultrasonic signal between ultrasonic transducers can be obtained. When the ultrasonic signal level is maximized, the heads are locked in place. The displacement gauge shall be capable of setting and maintaining a parallel alignment to an accuracy of  $\pm .2\text{ mm/m}$  ( $\pm .0002\text{ in./in.}$ ) throughout the useful range of travel while a controlled pressure is applied and simultaneously measuring displacement.

7.1.2 The load application and measurement shall be capable of variation from 44.48 N to 1,334.4 N (10 lbf to 300 lbf), accurate to  $\pm 1.11\text{ N}$  ( $\pm 0.25\text{ lbf}$ ).

7.1.3 The displacement measurement device shall be capable of  $0.5\text{ }\mu\text{m}$  ( $1.97 \times 10^{-5}\text{ in.}$ ) resolution.

7.1.4 The ultrasonic pulser/receiver shall have broad band characteristics (up to 30 MHz.).

7.1.5 Two broadband (for example, 70 %) ultrasonic search units (the transducers) shall be used with the pulser/receiver. Typical sizes of the ultrasonic search units used for this test method are circular with a diameter of between 1.27 cm (0.50 in.) and 3.17 cm (1.25 in.). Once chosen, this diameter must be kept constant for calibration and unknown measurements. Guide E 1065 procedures should be used to establish the transducer characteristics for matching purposes.

7.1.6 Digitizer capable of minimum time equivalent sampling rate of 200 MHz.

<sup>9</sup> The Ultrasonic Resin Analyzer (URA) available from Xxsys Technologies Inc., 4025 Avati Drive, San Diego, CA 92117 has been found to be satisfactory for performing these measurements.