Designation: D3530 – 97 (Reapproved 2015)

Standard Test Method for Volatiles Content of Composite Material Prepreg[1]

This standard is issued under the fixed designation D3530; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of the volatiles content, in weight percent of composite material prepregs. This standard focuses on composites with thermosetting resins that tend to lose a few percent of the matrix mass when heated due to loss of both retained water and low molecular weight matrix constituents that volatilize during heating.

1.2 Use of this test method is limited to maximum temperature of circulating air ovens (approximately 300°C).

1.3 Use of this test method is limited to temperatures below which the matrix flows from the reinforcement.

1.4 The values stated in SI units are to be regarded as 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3878 Terminology for Composite Materials

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases (Withdrawn 2015)[3]

2.2 *NFPA Standard:*

NFPA 86 Standard for Ovens and Furnaces[4]

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology D883 defines terms relating to plastics. Practice E177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over other documents.

3.1.1 *prepreg, n*—the admixture of fibrous reinforcement and polymeric matrix used to fabricate composite materials. Its form may be sheet, tape, or tow. See Terminology D3878.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Volatiles Content, n*—the amount of volatiles present in a prepreg expressed as a weight percent.

3.3 *Symbols:*

3.3.1 M_i—the initial mass of the sample.

3.3.2 M_f—the mass of the sample after oven exposure.

3.3.3 V_c—the weight percent volatiles content.

4. Summary of Test Method

4.1 Specimens of prepreg are weighed and then exposed to elevated temperature, equal to the nominal cure or consolidation temperature of the material, in an air circulating oven to remove the volatiles. The exposed samples are reweighed and the percent change in weight expressed as volatiles content.

5. Significance and Use

5.1 This test method is used to obtain the volatiles content of composite material prepreg. Knowledge of the volatiles content is useful in developing optimum manufacturing processes.

5.2 The volatiles content is determined after exposure to the nominal cure or consolidation temperature.

6. Interferences

6.1 *Airflow*—The amount of measured volatiles may be increased or decreased by changing the velocity of airflow.

[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.

Current edition approved Nov. 1, 2015. Published December 2015. Originally approved in 1976. Last previous edition approved in 2008 as D3530/D3530M – 97(2008)ε2. DOI: 10.1520/D3530_D3530M-97R15.

[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.

[4] Available from National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02269-9101.

Since airflow in most ovens is not linear in each part, a velometer should be used to measure airflow where samples are placed. Samples should be placed only in positions of known airflow so that results may be repeatable. Use of baffles has been found to even airflow between samples.

6.2 *Sample Exposure*—The geometric shape and positioning of the sample have an effect on the measured volatiles content. Samples placed horizontally in a rack will not be exposed to the same amount of airflow as samples hung vertically. A ribbon wound in a 150 mm diameter hoop may give slightly different results than the same ribbon wound in a 50 mm diameter hoop. A thinner sample will be exposed to more airflow at its surface than a thicker sample.

6.3 *Time of Exposure*—For any given temperature, sample placement, and airflow, the sample will lose volatiles at a set initial rate, that decreases over time. After some time period, volatiles lost in the test will approach the true volatiles content of the sample. If the time period is not sufficient to show a true volatiles content of the material, the volatiles content is representative of only the condition of the test.

6.4 *Time of Ambient Exposure*—Volatiles content varies due to prolonged exposure of temperatures exceeding prepreg glass transition temperature or exposure to humidity. This change is associated with matrix crosslinking and change in tack.

7. Apparatus

7.1 *Cutting Blade*—Die cutter that provides fixed specimen area to within a tolerance of 0.5 % is recommended. Single edge blade is acceptable.

7.2 *Cutting Template*—When a die cutter is not used, a cutting template is required. The cutting template shall have grooves within a tolerance of 0.4 mm parallel true position from center line. This allows a 2 % area error.

7.3 *Analytical Balance*—The analytical balance shall be capable of reading to within ±0.1 mg.

7.4 *Circulating Air Oven,* capable of tolerance within ±3°C. Removable baffles should be placed in the oven so that airflow is not directly aimed at the specimen.

7.5 *Rack,* that allows air circulation and from which hooks may be suspended.

7.6 *Timer,* capable of reading 20 ± 1 min.

7.7 *Desiccator.*

8. Hazards

8.1 Some materials contain flammable or toxic solvents as part of the matrix. These materials could build up to dangerous concentrations of vapor in the oven. NFPA 86 provides guidelines on amount of flammable materials that may be safely placed in an oven.

9. Test Specimens

9.1 A minimum of three specimens shall be tested for each sample.

9.2 The specimen size shall be a minimum of 1600 mm^2 by material thickness. Ribbon, braid, and fabric forms that do not drip resin shall be suspended from the rack. Ribbon shall be looped in close proximity so that individual strands are not clumped together. Other forms may be placed horizontally on the rack.

10. Calibration and Standardization

10.1 All measuring equipment shall have certified calibrations that are current at the time of use of the equipment. The calibration documentation shall be available for inspection.

11. Conditioning

11.1 Store carbon fiber-epoxy prepreg at low temperatures as recommended by the manufacturer (typically approximately −18°C). Allow sealed packages of material to warm as recommended by manufacturer or controlling specification before seal is opened to ensure that the material does not absorb moisture from the atmosphere.

12. Procedure

12.1 Weigh each of the three specimens on an analytical balance to 0.1 mg. Record the mass of each specimen as M_i.

12.2 Coat any surfaces of the rack or clip that may contact the specimen with a suitable release agent to prevent loss of specimen weight due to resin adherence.

12.3 Place the samples on the rack or suspended from the rack (braids, ribbons, and fabric) as quickly as possible, place the rack into a preheated oven, and set at the nominal cure or consolidation temperature as recommended by the manufacturer or controlling specification. The specimens should be placed so that the maximum surface area is exposed to the circulating heat.

12.4 Set the timer for 15 min.

NOTE 1—Other times may be used, but a timed study shall be performed showing that at least 90 % of the maximum volatiles content is obtained at the recommended time.

12.5 Remove the specimens from the rack and place them in a desiccator.

12.6 Remove the specimens from the desiccator after they have cooled to ambient (a minimum of 5 min). Weigh specimens within 1 min of removal from desiccator. Record this mass as M_f.

13. Calculation

13.1 Calculate the volatiles content V_c, weight percent, of each specimen as follows:

$$V_c = \frac{M_i - M_f}{M_i} \times 100 \quad (1)$$

where:

M_i = initial weight of prepreg specimen, g, and
M_f = weight of specimen after removed from oven, g.

14. Report

14.1 Report the following information or references pointing to other documents containing the information to the maximum extent applicable:

14.1.1 Reporting of items that are beyond the control of a given test laboratory, such as material details shall be the responsibility of the requestor,

14.1.2 Complete identification of the material according to Guide E1309 including fiber type, surface treatment, fiber manufacturer, resin system, resin manufacturer, prepreg manufacturer, prepreg form, prepreg areal weight, and prepreg matrix content,

14.1.3 The time and temperature to remove the volatiles,

14.1.4 Specific ambient exposure conditions and time at each condition, and

14.1.5 The volatiles content for each of the three specimens expressed as weight percent, the average volatiles content.

15. Precision and Bias

15.1 *Precision*—The data required for the development of a precision statement is not available for this test method. Committee D30 is currently planning a round-robin test for this test method in order to determine precision.

15.2 *Bias*—Bias cannot be determined for this test method as no acceptable reference standard exists.

16. Keywords

16.1 composite materials; prepreg; volatiles content