Designation: D3529M – 10

Standard Test Methods for Constituent Content of Composite Prepreg[1]

This standard is issued under the fixed designation D3529M; 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 Department of Defense.

1. Scope

1.1 This test method covers the determination of the fiber content, fiber areal weight, matrix solids content and matrix content of composite material prepregs. Optionally, the matrix solids content can also be determined after a volatiles content has been established. Volatiles content, if appropriate and required, is determined by means of Test Method D3530/D3530M.

1.2 Procedure A of this test method applies to composite prepreg of primarily thermosetting matrices that can be extracted in organic solvent. The reinforcement and filler must be substantially insoluble in the selected extraction reagent. This procedure may also be used for the same purposes to extract other matrix material types.

1.3 Procedure B of this test method uses ignition loss of a composite prepreg matrix and applies to organic matrix composite systems containing reinforcing fibers that do not change mass when exposed to the matrix combustion method.

1.4 This test method assumes a two-part material system (plus volatiles) and does not distinguish between hybrid reinforcements or matrices. Use with hybrid composites is limited to determination of total reinforcement or total matrix content.

1.5 Matrix solids determination for Procedures A or B uses Test Method D3530/D3530M to determine volatiles content.

1.6 Alternate techniques for determining constituent content include Test Methods C613/C613M (resin content by Soxhlet extraction) and D3171 (used principally for consolidated laminates).

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

1.8 *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 information is given in Notes 2-8 .

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C613/C613M Test Method for Constituent Content of Composite Prepreg by Soxhlet Extraction

D883 Terminology Relating to Plastics

D3171 Test Methods for Constituent Content of Composite Materials

D3530/D3530M Test Method for Volatiles Content of Composite Material Prepreg

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

2.2 *NFPA Standard:*

NFPA 86 Standard for Ovens and Furnaces[3]

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 the other documents.

3.1.1 *fiber content*, *n*—the amount of fiber present in a composite or prepreg expressed either as percent by weight or percent by volume. This is sometimes stated as a fraction, that is, fiber volume fraction. See Terminology D3878.

3.1.2 *matrix content*, *n*—the amount of matrix present in a composite or prepreg expressed either as percent by weight or percent by volume. For polymer matrix composites this is resin content. See Terminology D3878.

3.1.3 *prepreg*, *n*—the admixture of fibrous reinforcement and polymeric matrix used to fabricate composite materials. Its form may be sheet, tape, or tow. For thermosetting resins, it has

[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, 2010. Published December 2010. Originally approved in 1976. Last previous edition approved in 2008 as D3529/D3529M – 97(2008). DOI: 10.1520/D3529M-10.

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

been partially cured to a controlled viscosity called "B stage". See Terminology D3878.

3.1.4 *reinforcement content*, *n*—the amount of reinforcement present in a composite or prepreg expressed either as percent by weight or percent by volume.

3.1.4.1 *Discussion*—If fiber is the sole reinforcement, then fiber content is equal to reinforcement content. See Terminology D3878.

3.1.5 *resin content*, *n*—see *matrix content*. See Terminology D3878.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dry resin content*, *n*—a prepreg resin content calculated by subtracting the average mass loss due to volatiles from the initial test mass.

3.2.2 *fiber areal weight*, *n*—the mass per unit area of the fiberous reinforcement of a composite material.

3.2.3 *matrix solids content*, *n*—see *dry resin content*.

3.2.4 *wet resin content*, *n*—a prepreg resin content that includes any volatiles present in the uncured resin. This term is used interchangeably with matrix content and resin content in this test method.

3.2.5 *volatiles content*, *n*—the amount of volatiles present in a prepreg expressed as percent by weight.

3.3 *Symbols:*

3.3.1 *A*—specimen area, m^2.

3.3.2 *FAW*—fiber areal weight; the fiber mass, W_f, divided by the specimen area, *A*, in g/m^2.

3.3.3 *FC*—fiber content of specimen, weight percent.

3.3.4 M_c—mass of container, g.

3.3.5 *MC*—matrix content of specimen, weight percent.

3.3.6 M_f—final mass of specimen and container at end of test, g.

3.3.7 M_I—initial mass of the specimen, g.

3.3.8 *MS*—matrix solids content of specimen, weight percent.

3.3.9 V_c—average volatiles content test result from Test Method D3530/D3530M, weight percent.

4. Summary of Test Methods

4.1 Prepreg is sampled and specimens of a specific area are weighed and the matrix removed by appropriate procedure. For Procedure A, the specimen is subjected to suitable solvent that affects the matrix (but not the reinforcement) until the matrix is dissolved and removed. The remaining reinforcement is then dried and weighed. For Procedure B, the specimen is placed in a muffle furnace for a specific time and at a specific temperature (at which the reinforcement remains essentially unchanged) until the matrix can be entirely removed as ash residue. The remaining reinforcement is weighed. For either procedure the change in mass is expressed, both matrix content and fiber content, as a percentage of the specimen initial mass.

4.2 When matrix solids content is requested, adjacent samples are tested for volatiles content according to Test Method D3530. The average mass loss due to volatiles is subtracted from the average resin content result and the result expressed as a percentage of initial specimen mass as matrix solids content. The matrix solids content is applicable mainly to thermosetting matrices.

5. Significance and Use

5.1 This test method can be used to obtain the fiber content, fiber areal weight, matrix content or matrix solids content. Knowledge of these results is useful in specifying material systems and in developing optimum manufacturing processes. Where volatiles content is consistent and not critical to process, matrix content shall be used.

5.2 This test method is limited to reinforcement constituents that do not change mass upon exposure to the matrix removal procedure and limited to matrix constituents that are fully removed by the matrix removal procedure. In the event of experiencing either limitation, an agreement should be reached with the end-user as to how to proceed.

6. Interferences

6.1 *Procedure A*:

6.1.1 *Extent of Cure in Thermosetting Systems*—In Procedure A, the efficiency of matrix dissolution for thermosetting matrix materials is directly related to the extent of cure of the resin system. A resin that has started to cross link (such as B-staged resins) will be increasingly more difficult to extract as the cure advances. If Procedure A is ineffective in removing the matrix, and Procedure B is inappropriate for the fiber, then another test method, such as one of the methods of Test Method D3171, should be consulted.

6.1.2 *Reagent Selection*—The proper reagent, in a suitable quantity, must be selected for the constituents under test. The reagents listed in Section 8 are provided for consideration, particularly with regard to thermosetting materials, but cannot be assured to perform well on all material systems within the scope of this test method.

6.1.3 *Ability of Solvent to Reach Matrix*—Solvents are more efficient if the material is spread as a film, rather than a solid mass. It is recommended that the sample be consolidated as little as possible before introduction into the solvent. A solvent that is satisfactory for a given form may not be adequate with the same material of a different form.

6.1.4 *Solvent Strength*—Solvent strength, or the ability to rapidly dissolve the matrix, may be enhanced by increasing the amount of solvent, increasing agitation, increasing temperature, or by placing the sample under vacuum. These are offered as physical alternatives to use of more hazardous solvents, or combined solvent systems.

6.2 *Procedure B*:

6.2.1 *Applicability of Ignition Loss Approach*—In Procedure B a reinforced resin matrix composite specimen will be exposed to a temperature in excess of 500°C in an air environment for up to 6 h. This test method does not provide a measure of resin content for samples containing reinforcing materials that lose weight under the conditions of the test or contain resins or fillers that do not decompose to volatile materials released by ignition.

6.2.2 *Length of Test*—The sample type and configuration will determine the length of test exposure. The products of matrix combustion will disappear more readily in configurations having a large surface area of resin matrix exposure to the oxidizing atmosphere.