Designation: D3171 – 22

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

This standard is issued under the fixed designation D3171; 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 These test methods determine the constituent content of composite materials by one of two approaches. Test Method I physically removes the matrix by digestion or ignition or carbonization by one of eight procedures, leaving the reinforcement essentially unaffected and thus allowing calculation of reinforcement or matrix content (by weight or volume) as well as percent void volume. Test Method II, applicable only to laminate materials of known fiber areal weight, calculates reinforcement or matrix content (by weight or volume), and the cured ply thickness, based on the measured thickness of the laminate. Test Method II is not applicable to the measurement of void volume.

1.1.1 These test methods are primarily intended for two-part composite material systems. However, special provisions can be made to extend these test methods to filled material systems with more than two constituents, though not all test results can be determined in every case.

1.1.2 The procedures contained within have been designed to be particularly effective for certain classes of polymer or metal matrices. The suggested applications are discussed in Section 4, as well as at the start of each procedure.

1.1.3 Test Method I assumes that the reinforcement is essentially unaffected by the digestion or ignition medium or carbonization. A procedure for correction of the results for minor changes in the reinforcement is included. Procedures A through F are based on chemical removal of the matrix, while Procedure G removes the matrix by igniting the matrix in a furnace. Procedure H carbonizes the matrix in a furnace.

1.1.4 Test Method II assumes that the fiber areal weight of the reinforcement material form is known or controlled to an acceptable tolerance. The presence of voids is not measured. Eq 15 and 16 assume zero void content to perform the calculation.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.* See Section 9 for additional information.

1.4 *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]

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Relating to Plastics

D1505 Test Method for Density of Plastics by the Density-Gradient Technique

D3878 Terminology for Composite Materials

D5229/D5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials

E12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases (Withdrawn 1996)[3]

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

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology D883 defines terms relating to plastics. Terminology E12 defines terms relating to

[1] These test methods are under the jurisdiction of ASTM Committee D30 on Composite Materials and are the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

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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.

specific gravity. Practice E177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over other documents.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *density, $\rho^{23°C}$*—the weight per unit volume measured in air, of the impermeable portion of a material at 23 °C.

3.2.1.1 *Discussion*—The definitions of specific gravity and density are essentially equivalent to the definitions of apparent specific gravity and apparent density in Terminology E12, because no correction is made for buoyancy of the material in air. However, this difference is insignificant for most engineering purposes.

3.2.2 *specific gravity, $SG^{23°C}$*—the ratio of the weight in air of a unit volume of the impermeable portion of a material at 23 °C referenced to the standard unit volume weight of water at 23 °C.

3.3 *Symbols:*

A = area of the specimen.
A_r = calculated mass of one layer of reinforcement/unit area.
CR_m = carbonization ratio of the neat resin.
ρ_c = density of the composite specimen.
ρ_m = density of the cured matrix.
ρ_r = density of the reinforcement or fiber.
h = thickness of the specimen.
h_p = cured ply thickness, mm.
m_c = mass of the dry crucible for neat resin carbonization.
m_{cr} = mass of the dry crucible with carbonized neat resin.
m_d = final mass of the neat resin residue after carbonization.
m_i = initial mass of the neat resin specimen.
M = mass of the specimen.
M_c = mass of the dry crucible or sintered glass filter.
M_{cr} = mass of the dry crucible or sintered glass filter with reinforcement residue.
M_d = final residue mass of the composite specimen.
M_i = initial mass of composite specimen before digestion, combustion, or carbonization.
M_f = final mass of composite specimen after digestion, combustion, or carbonization.
M_m = mass of the resin matrix in the composite specimen.
N_p = number of plies in the laminate.
V_m = volume percent of matrix in specimen.
V_r = volume percent of reinforcement in the specimen.
V_v = void volume percent in the specimen.
W_m = weight percent of matrix in the specimen.
W_r = weight percent of reinforcement in the specimen.

4. Summary of Test Method

4.1 *Test Method I*—The matrix portion of a material specimen of known mass is removed in a hot liquid medium (for dissolution) or furnace (for combustion). When dissolving in a hot liquid medium, the remaining residue, containing the reinforcement, is then filtered, washed, dried, cooled, and weighed. The weight percent of the reinforcement is calculated, and from this value, and if densities of both the composite and the reinforcement are known, the volume percent is calculated. An additional calculation for void volume may be made if the density of the matrix is known or determined.

4.1.1 A correction for weight change of the reinforcement or retention of the matrix may be made (13.1.2 and 13.1.3), if this change is sufficiently reproducible under the conditions of the test and has the same value for the reinforcement or matrix alone as for the constituents in the composite.

4.1.1.1 *Procedure A,* for matrices such as epoxy resin, steel, copper, or others digestible by concentrated nitric acid.

NOTE 1—Many reinforcements are attacked by nitric acid. If reinforcement is attacked, an alternative method is recommended, depending on the matrix. See Annex A1.

4.1.1.2 *Procedure B,* for matrices such as epoxy, phenolic, polyamide, or thermoplastic resin, or others digestible by an aqueous mixture of sulfuric acid and hydrogen peroxide. See Annex A2.

4.1.1.3 *Procedure C,* for matrices such as epoxy resin and others digestible by a mixture of ethylene glycol and potassium hydroxide. See Annex A3.

NOTE 2—Procedure C is especially applicable to anhydride-cured epoxy systems containing aramid or carbon reinforcement.

4.1.1.4 *Procedure D,* for matrices such as aluminum, brass, or others digestible by sodium hydroxide solution. See Annex A4.

4.1.1.5 *Procedure E,* for matrices such as steel, titanium, copper, aluminum, or others digestible by hydrochloric acid. See Annex A5.

4.1.1.6 *Procedure F,* a version of Procedure A for microwave-aided heating. See Annex A6.

4.1.1.7 *Procedure G,* for reinforcements such as glass, or ceramic that are not affected by high-temperature environments, or reinforcements such as carbon where temperature is adequately controlled so that reinforcement does not char. See Annex A7.

4.1.1.8 *Procedure H,* for any reinforcements, particularly carbon, that are not affected at high temperature in a nitrogen atmosphere, and any resin matrix systems. The correction for the retention of the matrix (13.1.3) is not necessary for this procedure. See Annex A8.

4.2 *Test Method II*—The thickness of a relatively flat panel made with reinforcement of known and consistent areal weight is measured. By the thickness of the panel, the reinforcement and matrix content is calculated.

5. Significance and Use

5.1 A constituent content of a composite material must be known in order to analytically model the material properties (mechanical, physical, thermal, or electrical) of the composite which are affected by the reinforcement or matrix. Also, knowledge of the constituent content is required for evaluation of the quality of a fabricated material and the processes used during fabrication.

5.2 The void volume of a composite material may significantly affect some of its mechanical properties. Higher void volumes usually mean lower fatigue resistance, greater susceptibility to moisture penetration and weathering, and increased