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Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[1]

This standard is issued under the fixed designation D5229/D5229M; 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.

ε[1] NOTE—The appearance of some of the section references was updated and other editorial changes were made throughout in February 2019.

INTRODUCTION

Consistent evaluation and comparison of the response of polymer matrix composites to moisture absorption can only be performed when the material has been brought to a uniform through-the-thickness moisture profile. The procedures described in Test Method D570 and Practices D618 do not guarantee moisture equilibrium of the material. A similar, but more rigorous, procedure for conditioning to equilibrium is described by this test method, which can also be used with fluid moisture other than water, and which, additionally, can provide the moisture absorption properties necessary for the analysis of single-phase Fickian moisture diffusion within such materials.

1. Scope

1.1 This test method covers a procedure for the determination of moisture absorption or desorption properties in the through-the-thickness direction for single-phase Fickian solid materials in flat or curved panel form. Also covered are procedures for conditioning test coupons prior to use in other test methods; either to an essentially moisture-free state, to equilibrium in a standard laboratory atmosphere environment, or to equilibrium in a non-laboratory environment. Also included are procedures for determining the moisture loss during elevated temperature testing, as well as moisture loss resulting from thermal exposure after removal from the conditioning environment, such as during strain gauge bonding. While intended primarily for laminated polymer matrix composite materials, these procedures are also applicable to other materials that satisfy the assumptions of 1.2.

1.2 The calculation of the through-the-thickness moisture diffusivity constant in Procedure A assumes a single-phase Fickian material with constant moisture absorption properties through the thickness of the specimen. The validity of the equations used in Procedure A for evaluating the moisture diffusivity constant in a material of previously unknown moisture absorption behavior is uncertain prior to the test, as the test results themselves determine if the material follows the single-phase Fickian diffusion model. A reinforced polymer matrix composite material tested below its glass-transition temperature typically meets this requirement, although two-phase matrices such as toughened epoxies may require a multi-phase moisture absorption model. While the test procedures themselves may be used for multi-phase materials, the calculations used to determine the moisture diffusivity constant in Procedure A are applicable only to single-phase materials. Other examples of materials and test conditions that may not meet the requirements are discussed in Section 6.

1.3 The evaluation by Procedure A of the moisture equilibrium content material property does not assume, and is therefore not limited to, single-phase Fickian diffusion behavior.

1.4 The procedures used by this test method may be performed, and the resulting data reduced, by suitable automatic equipment.

1.5 This test method is consistent with the recommendations of CMH-17 Rev G (**1**),[2] which describes the desirable attributes of a conditioning and moisture property determination procedure.

1.6 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in

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

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.6.1 Within the text the inch-pound units are shown in brackets.

1.7 *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.*

1.8 *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:*[3]

D570 Test Method for Water Absorption of Plastics
D618 Practice for Conditioning Plastics for Testing
D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
D883 Terminology Relating to Plastics
D2584 Test Method for Ignition Loss of Cured Reinforced Resins
D2734 Test Methods for Void Content of Reinforced Plastics
D3171 Test Methods for Constituent Content of Composite Materials
D3878 Terminology for Composite Materials

2.2 *Military Standard:*[4]

MIL-B-131 Barrier Materials, Watervaporproof, Greaseproof, Flexible, Heat-Sealable

3. Terminology

3.1 *Definitions:*

3.1.1 Terminology D3878 defines terms relating to high-modulus fibers and their composites. Terminology D883 defines terms relating to plastics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the other terminology standards.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 If the term represents a physical quantity, its analytical dimensions are stated immediately following the term (or letter symbol) in fundamental dimension form, using the following ASTM standard symbology for fundamental dimensions, shown within square brackets: [*M*] for mass, [*L*] for length, [*T*] for time, [*Θ*] for thermodynamic temperature, and [*nd*] for non-dimensional quantities. Use of these symbols is restricted to analytical dimensions when used within square brackets, as the symbols may have other definitions when used without the brackets.

3.2.2 *accuracy criterion, n*—the maximum amount of change in average moisture content for a test coupon, over the span of the reference time period, which is allowable for the establishment of effective moisture equilibrium. (See also *average moisture content, moisture equilibrium,* and *reference time period.*)

3.2.3 *average moisture content, M (%), n*—the average amount of absorbed moisture in a material, taken as the ratio of the mass of the moisture in the material to the mass of the oven-dry material and expressed as a percentage, as follows:

$$M, \% = \frac{W_i - W_o}{W_o} \times 100 \quad (1)$$

where:

W_i = current specimen mass, g, and
W_o = oven-dry specimen mass, g.

(See also *oven-dry.*)

3.2.4 *Fickian diffusion, n*—a model of material moisture absorption and desorption that follows Fick's second law, as follows in one-dimension:

$$\frac{\partial c}{\partial t} = D_z \frac{\partial^2 c}{\partial z^2}$$

3.2.5 *glass transition temperature, T_g[Θ], n*—the approximate midpoint of the temperature range over which a reversible change takes place between a viscous or rubbery condition and a hard, relatively brittle condition, in an amorphous polymer, or in amorphous regions of a partially crystalline polymer.

3.2.5.1 *Discussion*—The glass transition temperature of many polymer matrix composites is lowered by the presence of absorbed moisture.

3.2.6 *moisture, n*—liquid (water, jet fuel, salt water, or any other liquid) that is either diffused in relatively small quantity and dispersed through a gas as a vapor, condensed on a surface as visible dew, or present in quantity sufficient for immersion of an object.

3.2.6.1 *Discussion*—The dictionary definition of moisture for this test method is extended to include not only the vapor of a liquid and its condensate, but the liquid itself in large quantities, as for immersion.

3.2.7 *moisture concentration, c [ML^{-3}], n*—the absolute amount of absorbed moisture in a material expressed as the mass of moisture per unit volume.

3.2.8 *moisture diffusivity constant, D_z [L^2T^{-1}], n*—the property of a material that describes the rate at which the material absorbs or desorbs moisture.

3.2.8.1 *Discussion*—In Fickian materials this property is relatively independent of the moisture exposure level (and thus the moisture equilibrium content material property). However, the moisture diffusivity constant is strongly influenced by temperature. Moisture diffusivity can be anisotropic; the subscript *z* indicates the value in the through-the-thickness direction for anisotropic diffusion behavior.

[3] 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.

[4] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

3.2.9 *moisture equilibrium, n*—the condition reached by a material when there is essentially no further change in its average moisture content with the surrounding environment.

3.2.9.1 *Discussion*—Moisture equilibrium can be either *absolute* or *effective*. Absolute moisture equilibrium requires no measurable change in moisture content, while effective moisture equilibrium allows a specified small change in the average moisture content of a material (the accuracy criterion) over a specified time span (the reference time period). (See also *accuracy criterion, average moisture content,* and *reference time period.*) Effective moisture equilibrium is a satisfactory definition for most engineering applications. Unless otherwise specified, references to moisture equilibrium in this test method mean effective moisture equilibrium, as quantified in 10.2. Moisture equilibrium can also be either *static,* when there is no moisture transport at all across the surfaces, or *dynamic,* when moisture transport exists, but the net sum for the material is zero. This test method is not capable of discerning between these two types of moisture equilibrium.

3.2.10 *moisture equilibrium content, M_m (%), n*—the maximum amount of absorbed moisture that a material can contain at moisture equilibrium for a given moisture exposure level, expressed as a percent of dry material mass. (See also *moisture saturation content.*)

3.2.10.1 *Discussion*—In polymer matrix composites, this property is relatively independent of temperature (and thus the moisture diffusivity constant material property), but it is a function of the moisture exposure level. For the purposes of this test method M_m is assumed to be equivalent to the average moisture content at effective moisture equilibrium, M_f.

3.2.11 *moisture exposure level, n*—a measure or description of the severity of a conditioning environment in terms of the amount of liquid or vapor present. (See also *moisture* and *relative vapor level.*)

3.2.12 *moisture saturation content, n*—the moisture equilibrium content at the maximum possible moisture exposure level, wherein the material contains the greatest possible amount of absorbed moisture. (See also *moisture equilibrium content.*)

3.2.13 *oven-dry, n*—the condition of a material that has been dried in accordance with Procedure D of this test method until moisture equilibrium is achieved.

3.2.14 *reference time period, n*—the time interval for mass measurement used to define effective moisture equilibrium in a material. (See also *accuracy criterion, average moisture content,* and *moisture equilibrium.*)

3.2.14.1 *Discussion*—A small change in the average moisture content (the accuracy criterion) for a material during the reference time period indicates effective moisture equilibrium.

3.2.15 *relative vapor level (%), n*—the ratio of the pressure of a vapor present to the pressure of the saturated vapor, at the same temperature, expressed as a percent.

3.2.15.1 *Discussion*—Applicable only to the gaseous form of a fluid. When the vapor is water vapor the term is called *relative humidity.* (See also *moisture exposure level.*)

3.2.16 *standard laboratory atmosphere, n*—an atmosphere (environment) having a temperature of 23 ± 2 °C [73.4 ± 3.6 °F] and a relative humidity of 50 ± 10 %.

3.2.17 *standard conditioned specimen, n*—the material condition of a test coupon that has reached effective moisture equilibrium at a nominal relative humidity of 50 % (considered to be a standard laboratory environment) in accordance with Procedure C of this test method.

3.2.18 *test temperature, n*—the environmental temperature used in Procedures A-E, Y, and Z.

3.2.18.1 *Discussion*—This is distinguished, for the purposes of this test method, from the environmental temperature used during any subsequent material evaluation testing.

3.2.19 *traveler coupon, n*—a surrogate coupon of the same material and thickness, and of appropriate size (but without tabs) that is used in a conditioning procedure to determine moisture content for specimen configurations (such as a tabbed mechanical coupon, or a coupon that does not meet the minimum mass requirement) that cannot otherwise be properly measured by this test method.

3.3 *Symbols:*

3.3.1 *c*—moisture concentration.

3.3.2 D_z—moisture diffusivity constant in the through-the-thickness direction.

3.3.3 *G(T,t)*—moisture absorption or desorption function for materials that follow Fickian diffusion.

3.3.4 *h*—thickness of a material panel or plate in the through-the-thickness direction for double-sided moisture exposure.

3.3.5 *M*—average moisture content of a material. The following subscripts denote the average moisture content for specific conditions:

M_b, the average moisture content at a baseline time;

M_e, the average moisture content at establishment of effective moisture equilibrium;

M_{ed}, the average moisture content at establishment of effective moisture equilibrium as a delta from the average moisture content at a baseline time;

M_{et}, the average moisture content at establishment of effective moisture equilibrium as a total value from the condition of zero moisture content;

M_f, the final moisture content at the end of conditioning for a fixed time;

M_i, the average moisture content at a given time;

M_{i-1}, the average moisture content at the previous time;

M_L, the moisture loss from the state of effective equilibrium due to subsequent heating; and

M_m, the moisture equilibrium content that is reached when a uniform through-the-thickness moisture profile occurs for a given temperature and moisture exposure level.

3.3.5.1 *Discussion*—Procedures A-H of this test method condition specimens to M_e. Except for the use of a thin specimen in Procedure A, conditioning specimens to M_m is generally not practical. See also the discussion in Appendix X2.

3.3.6 *t*—time.

3.3.7 t_m—the maximum time required for a material to reach moisture equilibrium under specified conditions of temperature and initial moisture content.