Designation: D7028 – 07 (Reapproved 2015)

Standard Test Method for Glass Transition Temperature (DMA Tg) of Polymer Matrix Composites by Dynamic Mechanical Analysis (DMA)[1]

This standard is issued under the fixed designation D7028; 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.

1. Scope

1.1 This test method covers the procedure for the determination of the dry or wet (moisture conditioned) glass transition temperature (T_g) of polymer matrix composites containing high-modulus, 20 GPa (> 3 × 10^6 psi), fibers using a dynamic mechanical analyzer (DMA) under flexural oscillation mode, which is a specific subset of the Dynamic Mechanical Analysis (DMA) method.

1.2 The glass transition temperature is dependent upon the physical property measured, the type of measuring apparatus and the experimental parameters used. The glass transition temperature determined by this test method (referred to as "DMA Tg") may not be the same as that reported by other measurement techniques on the same test specimen.

1.3 This test method is primarily intended for polymer matrix composites reinforced by continuous, oriented, high-modulus fibers. Other materials, such as neat resin, may require non-standard deviations from this test method to achieve meaningful results.

1.4 The values stated in SI units are standard. The values given in parentheses are non-standard mathematical conversions to common units that are provided for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3878 Terminology for Composite Materials

D4065 Practice for Plastics: Dynamic Mechanical Properties: Determination and Report of Procedures

D4092 Terminology for Plastics: Dynamic Mechanical Properties

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

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases (Withdrawn 2015)[3]

E1434 Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases (Withdrawn 2015)[3]

E1471 Guide for Identification of Fibers, Fillers, and Core Materials in Computerized Material Property Databases (Withdrawn 2015)[3]

E1640 Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis

E1867 Test Method for Temperature Calibration of Dynamic Mechanical Analyzers

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to polymer matrix composites. Terminology D4092 defines terms relating to dynamic mechanical property measurements on polymeric materials.

3.2 *Symbols:* E' = storage modulus

E'' = loss modulus

$\tan \delta = E''/E'$ = tangent delta

DMA Tg = glass transition temperature defined from dynamic mechanical analysis measurement

L = length of specimen

W = width of specimen

T = thickness of specimen

T_t = peak temperature from tangent delta curve

[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] 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. Summary of Test Method

4.1 A flat rectangular strip of laminate is placed in the DMA equipment and oscillated at a nominal frequency of 1 Hz. The specimen is heated at a rate of 5°C/min (9°F/min). The same loading frequency and heating rate is used for both dry and wet specimens (moisture conditioned) to allow for comparison. The temperature at which a significant drop in storage modulus (E') begins is assigned as the glass transition temperature (DMA Tg). The peak temperature of the tangent delta curve (T_t) is identified along with DMA Tg for comparison purposes.

5. Significance and Use

5.1 This test method is designed to determine the glass transition temperature of continuous fiber reinforced polymer composites using the DMA method. The DMA Tg value is frequently used to indicate the upper use temperature of composite materials, as well as for quality control of composite materials.

6. Interferences

6.1 The standard testing machine shall be of the Dynamic Mechanical Analysis (DMA) type of instrument that operates with forced oscillation and applies a flexural loading mode (either three-point bend or dual cantilever) to the test specimen. Refer to Practice D4065 for a summary of various other DMA practices. Other loading modes (such as tensile, torsion or shear) may produce different test results. If another equipment type or loading mode is used the non-standard approach shall be described in the report and the test result recorded as non-standard.

6.2 A fixed frequency of 1 Hz is standard in this test method. In general, for a given material, a higher testing frequency produces a higher DMA Tg value than this standard, while use of the resonance mode will yield a different DMA Tg that may be either higher or lower than the standard. If a non-standard frequency, or the resonance mode, is used, the non-standard approach shall be described in the report and the test result recorded as non-standard.

6.3 A heating rate of 5 ± 1°C/min (9 ± 2°F/min) is standard in this test method. A change in heating rate will affect the glass transition temperature result; the standard heating rate is the best available compromise for comparing DMA Tg results of dry and wet laminates. If a different heating rate is used it shall be described in the report and the result recorded as non-standard.

NOTE 1—Users should be advised that a heating rate of 5°C/min represents a compromise between various issues related to Tg measurement precision and bias. It is widely known that heat transfer limitations are more pronounced in DMA apparatus compared to other thermal analysis techniques, such as differential scanning calorimetry (DSC) and thermomechanical analysis (TMA). For greatest precision, it has been recommended that heating rates be 2°C/min or less. Test Method E1640 specifies a heating rate of 1°C/min. However, in many cases 5°C/min is recommended as a compromise between Tg measurement accuracy and test method convenience, especially for wet laminate measurements, since the slower heating rate will cause specimen drying that will itself bias the results.

6.4 Purge gas type and flow rate and the position of the thermocouple can affect the DMA Tg test result and shall be noted and reported. The same conditions shall be used for both calibration and testing runs. Instrumentation manufacturer recommendations should be followed.

6.5 It is standard in this test method that one of the major fiber directions shall be parallel to the length of the specimen. The span-to-depth ratio, ply orientation, and ply stacking sequence of a specimen with respect to the testing fixture have a profound effect on the DMA Tg result. A meaningful comparison of data requires that the specimen configuration be the same. A non-standard specimen configuration shall be described in the report and the result recorded as non-standard.

6.6 The standard definition in this test method for DMA Tg is based on intersecting two tangent lines from a semi-logarithmic plot of the storage modulus versus temperature. Other T_g definitions typically produce different test results. For example, a linear plot scale will result in a lower value of DMA Tg. A non-standard DMA Tg definition shall be described in the report and the result recorded as non-standard. For comparison purposes the peak temperature of the tangent delta curve (T_t) is identified along with DMA Tg.

7. Apparatus

7.1 *Micrometer,* suitable for reading to 0.025 mm (0.001 in.) accuracy for measuring the specimen thickness and width.

7.2 *Caliper,* suitable for reading to 0.025 mm (0.001 in.) accuracy for measuring the specimen length and instrument clamping distance.

7.3 *Dynamic Mechanical Analyzer (DMA),* with oven capable of heating to above the glass transition temperature and of controlling the heating rate to the specified value.

8. Sampling and Test Specimens

8.1 Two specimens shall be tested for each sample. If the testing is part of a designed experiment, other sampling techniques may be used if described in the test plan.

8.2 Consult the instrument manufacturer's manual for specimen size. A span-to-thickness ratio greater than ten is recommended. Specimen absolute size is not fixed by this method as various dynamic mechanical analyzers require different sizes. Depending on the analyzer, typical specimen size can range from 56 ± 4 × 12 ± 1 × 2.0 ± 0.5 mm (2.21 ± 0.16 × 0.47 ± 0.04 × 0.08 ± 0.02 in.) (L × W × T) to 22 ± 1 × 3 ± 1 × 1.0 ± 0.5 mm (0.9 ± 0.04 × 0.12 ± 0.04 × 0.04 ± 0.02 in.).

8.3 One of the major fiber directions in the specimen shall be oriented along the length axis of the specimen. It is standard that one of the major fiber directions shall be parallel to the length of the specimen, and specimens containing only off-axis plies shall not be used. Any deviations from the standard orientation shall be reported and the test results noted as non-standard.

8.4 The specimen surfaces shall be flat, clean, straight, and dry to prevent slippage in the grips and mitigate any effects due to moisture. Opposite surfaces must be essentially parallel and intersecting surfaces perpendicular. Tolerances in thickness and width must be better than ±2 %.

8.5 The selected sample shall be taken from a representative portion of the laminate. Laminate edges or other irregularities created in the laminate by mold or bagging techniques should be avoided.

9. Calibration

9.1 The DMA equipment shall be calibrated in accordance with Test Method E1867 for temperature signals and in accordance with the equipment manufacturer's recommendation for the storage modulus. The equipment must be calibrated in the same loading mode as will be used for testing, either dual cantilever or three-point bending. The temperature calibration points must span the DMA Tg result.

10. Conditioning

10.1 Moisture has significant effect on DMA Tg. Therefore, it is recommended that the test specimens should be weighed before and after DMA Tg testing to quantify the moisture change in the specimen resulting from the DMA Tg test.

10.2 *Dry Specimens*—To minimize the presence of moisture in the specimens, dry specimens must be conditioned prior to testing by using either of the following techniques:

10.2.1 Dry the specimens in an oven in accordance with Test Method D5229/D5229M, Procedure D, then stored until test in a desiccator or sealed MIL-PRF-131[4] (or equivalent) aluminized bag, or

10.2.2 Store the material in a desiccator or sealed aluminized bag immediately after material curing (lamination), where the material shall remain except for the minimum time required for removal during specimen preparation and testing. The maximum time between cure (lamination) and testing shall be 30 days, after which, prior to testing, specimens shall be oven-dried in accordance with 10.2.1.

10.3 *Wet Specimens*—Condition in accordance with Test Method D5229/D5229M, Procedure B. The conditioned specimens shall be tested within 30 minutes after removal from the conditioning chamber, or stored in sealed MIL-PRF-131 (or equivalent) aluminized bag until test.

11. Procedure

11.1 *Test Specimen*—Measure the specimen thickness and width to 0.025 mm (0.001 in.) and record. Measure the specimen length to 0.025 mm (0.001 in.) and record. Weigh the specimen to the nearest milligram (0.001 g) and record.

11.2 *Specimen Installation*—Install the specimen in the DMA test equipment oven based upon clamping method to be employed.

11.3 *Positioning of Specimen*—Follow the manufacturer's procedure for positioning the specimen in the clamps. Generally, the specimen should be centered between the clamp faces and be parallel to the base of the instrument. Mount the specimen in dual cantilever mode or three-point bending mode.

11.4 *Heating Rate*—The standard heating rate is 5 ± 1°C/min (9 ± 2°F/min). The same heating rate shall be used for all samples whose results are to be compared. Any deviations from this heating rate shall be noted in the report and the result shall be reported as non-standard.

11.5 *Frequency*—The standard frequency to be used in this standard is 1 Hz, and the instrument should be operated in constant strain mode.

11.6 *Strain Amplitude*—The maximum strain amplitude should be kept within the linear viscoelastic range of the material. Strains of less than 0.1 % are standard.

11.7 *Temperature Range*—Program the run to begin at room temperature or a temperature at least 50°C (90°F) below the estimated DMA Tg and to end at a temperature at least 50°C (90°F) above DMA Tg, but below decomposition temperature.

11.8 *Purge Gas Flow Rate*—Follow the manufacturer's manual or recommendations to set the purge gas flow rate. Five litres/minute (0.2 CFM) is a typical purge gas flow rate setting. For some types of dynamic mechanical analyzers, a purge gas flow setting is not required.

11.9 *Thermocouple Positioning*—Follow the manufacturer's manual or recommendations to position the thermocouple. Typically the thermocouple should be as close to the sample as possible.

11.10 *Test*—Conduct DMA Tg measurements using the instrument settings specified and record the load and displacement data as a function of temperature. Allow the oven to cool before removing the specimen. Weigh the specimen after the test to the nearest milligram (0.001 g) after the removal from the oven and record.

11.11 *Specimen Examination*—Examine the specimen after the test and inspect for any visual anomalies (that is, delamination, blisters, cracks, etc.). Record any visual anomalies observed.

12. Interpretation of Results

12.1 *Glass Transition Temperature (DMA Tg)*—Plot the logarithm of storage modulus (E') and linear tangent delta (tan δ) versus the linear temperature (Fig. 1). During the glass transition, the storage modulus of the composite material is significantly reduced. The DMA Tg is determined to be the intersection of two tangent lines from the storage modulus by this test method. The first tangent line (Line A, Fig. 1) is selected at a temperature before the transition. This temperature is designated as TA. The second tangent line (Line B, Fig. 1) is constructed at the inflection point to approximately the midpoint of the storage modulus drop. This temperature is designated as TB. The two tangent lines are intersected, and temperature corresponding to this intersection point is recorded as the DMA Tg. See Appendix X1 for additional guidelines to draw tangent lines.

12.2 *Tangent Delta (δ) peak (T_t)*—The peak temperature of the tangent delta curve (T_t) is identified and reported (Fig. 1).

13. Validation

13.1 Any specimen that has an obvious flaw or deviation from the requirements of this test method may be rejected. A

[4] MIL-PRF-131, Barrier Materials, Watervaporproof, Greaseproof, Flexible, Heat-Sealable. Available at http://assist.daps.dla.mil or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.