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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.

~~ε1 NOTE—Reference to a research report was added and figures corrected in August 2008.~~

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

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[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.

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

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.

[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.

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 new or spare specimen shall be prepared from the same material package and tested to replace any specimens rejected per this paragraph.

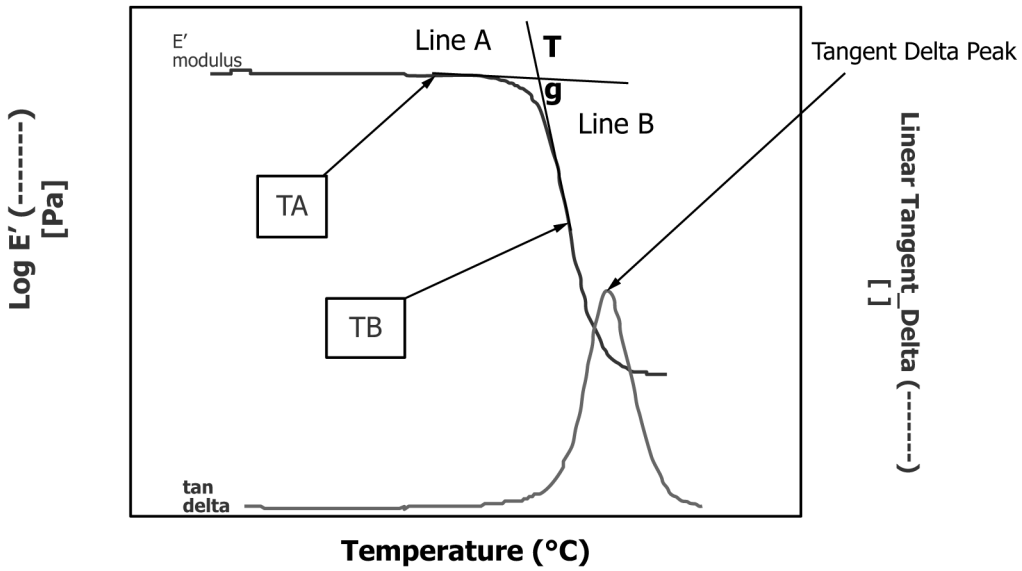

FIG. 1 Construction of Storage Modulus Glass Transition Temperature

13.2 Test results may be discarded for any conditions which compromise the integrity of the test. Should the results be retained, then these conditions shall be described in the test report. Specific examples include:

13.2.1 Cracks evident in the specimen after the test. This could indicate that the sample was taken from a defective portion of the laminate.

13.2.2 An irregularity of the plotted curve, such as change in slope, other than that due to the glass transition, or excessive noise. It is possible that more than one transition exists, but this should be confirmed by a separate run.

13.2.3 Slippage of the specimen in the grips.

14. Report

14.1 Report the following information, or references pointing to other documentation containing this information, to the maximum extent applicable (reporting of items beyond the control of a given testing laboratory, such as might occur with material details or panel fabrication parameters, shall be the responsibility of the requestor):

NOTE 2—Guides E1309, E1434, and E1471 contain data reporting recommendations for composite materials and composite materials mechanical testing.

14.1.1 The revision level or date of issue of this test method.

14.1.2 The name(s) of the test operator(s).

14.1.3 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.

14.1.4 Identification of all the materials constituent to the plate specimen tested, including for each: material specification, material type, manufacturer's material designation, manufacturer's batch or lot number, source (if not from manufacturer), date of certification, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, matrix type, matrix content, and volatiles content.

14.1.5 Description of the fabrication steps used to prepare the parent laminate including: fabrication start date, fabrication end date, process specification, cure cycle, consolidation method, and a description of the equipment used.

14.1.6 Ply orientation and stacking sequence of the laminate, relative to the longitudinal (long) dimension.

14.1.7 If requested, report density, volume percent reinforcement, and void content test methods, specimen sampling method and geometries, test parameters, and test results.

14.1.8 Method of preparing the test specimen, including specimen labeling scheme and method, specimen geometry, sampling method, and specimen cutting method.

14.1.9 Calibration dates and methods for all measurements and test equipment.

14.2 Report the following information:

14.2.1 Date of test.

14.2.2 Test span length and thickness.

14.2.3 Specimen conditioning history including weight gain or weight loss of specimen.

14.2.4 Instrument brand name, type, or model number.

14.2.5 Specimen loading condition and clamping details.

14.2.6 Heating rate and loading frequency.

14.2.7 Flow rate and type of the purge gas.

14.2.8 Any non-standard testing or data reduction parameters, including heating rate and loading frequency.

14.2.9 Deformation amplitude or strain.

14.2.10 Test results, including DMA Tg, peak tangent delta value (T_t), TA, TB, the method for DMA Tg determination, and comments on any irregularities or unexpected results.

14.2.11 Sample weight before and after DMA Tg testing and weight loss percentage.

15. Precision and Bias[5]

15.1 *Precision:*

[5] A research report is available from ASTM Headquarters. Request RR:D30-1004.

TABLE 1 Precision Statistics

Material	$\bar{X}$	S_r	S_R	r	R	$S_r/\bar{X}$,%	$S_R/\bar{X}$,%
DMA Tg (°C), Dry							
A	129	0.33	5.83	0.93	16.3	0.26	4.53
B	176	1.75	6.70	4.91	18.8	1.00	3.82
C	256	1.12	9.19	3.13	25.7	0.44	3.59
D	262	1.69	7.16	4.73	20.1	0.65	2.74
DMA Tg (°C), Wet							
A	79	1.03	6.55	2.88	18.3	1.30	8.31
B	120	1.41	7.03	3.96	19.7	1.18	5.85
C	190	0.41	7.70	1.14	21.6	0.22	4.06
D	190	2.27	9.23	6.35	25.9	1.19	4.85

15.1.1 The precision of the DMA Tg measurements depend on strict adherence to this test method and are influenced by mechanical and material factors, specimen preparation, and measurement errors.

15.1.2 Mechanical factors that can affect the test results include: the physical characteristics of the DMA testing equipment (stiffness, damping, and mass), accuracy of the loading and deflection measurements, loading frequency, alignment of the test specimen in the clamping device, clamping distance, thermocouple location.

15.1.3 Material factors that can affect test results include: material quality and representativeness, sampling scheme, and specimen preparation (surface quality, flatness, fiber alignment, aspect ratio, and so forth).

15.1.4 An interlaboratory test program was conducted where an average of two specimens each, of four different materials and layup configurations, were tested by seven different laboratories. The specimens were conditioned to both dry and wet environments per Test Method D5229/D5229M. Table 1presents the precision statistics generated from this study as defined in Practice E691 for DMA Tg dry and wet values. The materials listed in Table 1 are defined as:

A	Glass/Epoxy Fabric -$(90/0)_{10}$ layup
B	Carbon/Epoxy Tape -$(90/0)_{2s}$ layup
C	Carbon/Bismaleimide Tape -$(90/0)_{2s}$ layup
D	Carbon/Bismaleimide Fabric -$(90/0)_{12}$ layup

15.1.5 The averages of the coefficient of variation are shown in Table 2. The values of S_r/X and S_R/X represent the repeatability and the reproducibility coefficients of variation. These averages allow a relative comparison of the repeatability (within laboratory precision) and reproducibility (between laboratory precision) of the DMA Tg test parameters. These values indicate that the material factors did not have a significant impact on repeatability and reproducibility of the DMA Tg values measured. The DMA Tg dry values were found to exhibit higher repeatability and reproducibility than the DMA Tg wet values.

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

16. Keywords

16.1 composite; DMA; dynamic mechanical analysis; glass transition temperature; polymer matrix composite

APPENDIX

(Nonmandatory Information)

X1. EXAMPLES FOR INTERPRETATION OF RESULTS

X1.1 The DMA Tg is determined by this test method to be the intersection of two tangent lines from the storage modulus. Examples are shown in this appendix to provide graphical illustrations of how to select the two tangent lines.

X1.2 Fig. X1.1 shows an ideal DMA thermogram. It is ideal because the glass transition is clearly displayed. Before the transition the storage modulus is relatively constant, the sigmoidal change during transition is clear, and after the transition the storage modulus is relatively constant.[6] As described in 12.1, the first tangent line is selected at a temperature before the transition and the second tangent line is constructed at the inflection to mid-point of the modulus drop. Using this approach the intersection point is drawn as shown in Fig. X1.2. If the two tangent lines are constructed from temperatures too close to the transition, the intersection is depicted as shown in Fig. X1.3. On the other hand, if the two tangent lines are constructed at temperatures too far away from the transition, the intersection is depicted in Fig. X1.4. Figs. X1.3 and X1.4 illustrate that not following the approach of this test method can cause the intersection temperature of an ideal thermogram to vary by 3°C (5°F).

X1.3 Fig. X1.5 shows a non-ideal DMA thermogram. In this example the transition is less clear than the thermogram of Fig. X1.1. Before the transition the storage modulus continues to slope downward and after the transition the storage modulus continues to slope downward. Using the approach of this test method the intersection point is drawn as shown in Fig. X1.6. If the two tangent lines are constructed from temperatures too close to the transition, the intersection is depicted as shown in Fig. X1.7. On the other

[6] In Fig. X1.1 the loss modulus (E'') and tangent delta (tan delta) curves are also plotted. Alternative definitions of glass transition temperature such as the peak of the loss modulus or of the tan delta have been reported in literatures.

TABLE 2 Averages of the Coefficient of Variation

Parameter	Average of $S_r/\bar{X}$,%	Average of $S_R/\bar{X}$,%
DMA Tg Dry	0.59	3.7
DMA Tg Wet	0.97	5.8