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Test Procedures for Automotive Structural Composite Materials

RATIONALE

This document has been determined to contain basic and stable technology which is not dynamic in nature. This document was only referenced in one document at FCA US LLC but not in the references of ASTM test procedures or other standards that we could find.

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1. Scope—This SAE Standard is intended to serve as a guide for the collection of physical, mechanical, and thermal properties of fiber-reinforced polymer composite materials for automotive structural applications. This document attempts to utilize test methods applicable to the widest range of structural materials and processes without compromising the integrity of the data being sought. A summary of the material characterization is shown in Section 15.

2. References

2.1 Applicable Publications—The following publications form a part of the specification to the extent specified herein. Unless otherwise indicated the latest revision of SAE publications shall apply.

2.1.1 SAE PUBLICATION—Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

SAE EA-2253—SDRP Data Reporting Program

2.1.2 ASTM PUBLICATIONS—Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 618—Conditioning Plastics and Electrical Insulating Materials for Testing
ASTM D 638—Tensile Properties of Plastics
ASTM D 696—Coefficient of Linear Thermal Expansion of Plastics
ASTM D 792—Specific Gravity and Density of Plastics by Displacement
ASTM D 2584—Ignition Loss of Cured Reinforced Resins
ASTM D 3039—Tensile Properties of Fiber Composites
ASTM D 3410—Compression Properties of Fiber Composites
ASTM D 3417—Heats of Fusion and Crystallization of Polymers by Thermal Analysis
ASTM D 3593—Molecular Weight Averages and Distribution of Polymers by GPC Method
ASTM D 4065—Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics
ASTM D 5083—Tensile Properties of Reinforced Thermosetting Plastics Using Straight Sided Specimens
ASTM D 5296—Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size-Exclusion Chromatography
ASTM D 5379—Shear Properties of Composite Materials by the V-notched Beam Method

2.1.3 GENERAL MOTORS PUBLICATION—Available from Boise Cascade, 13301 Stephens Road, Warren, MI 48089.

GM Std 9077-P—Determination of Filler Content

2.1.4 CONFLICTS—In the event of a conflict between the text of this document and the references cited herein, the text of this document shall take precedence.

3. Definitions

3.1 Structural Polymer Composite—Automotive structural polymer composites are considered to be those having a tensile modulus greater than 7 GPa and a tensile strength greater than 100 MPa.

3.2 Planar Isotropic Composites—These materials are defined as being comprised of randomly oriented fibers that will result in properties that are nearly the same in all directions in-plane, but different in the thickness direction. Examples of such materials include liquid molded composites reinforced with continuous strand mats, chopped strand mats, or directed fiber preforms; random fiber sheet molded composites (SMC) and random fiber reinforced thermoplastic composites.

3.3 Orthotropic Composites—These materials do not have the same x, y, and z directional properties. Such materials include liquid molded composites reinforced with unidirectional and bidirectional stitched mats, woven fabrics, knitted fabrics, and directionally reinforced SMC and thermoplastic composites.

3.4 Special Reinforced Composites—Some reinforcements create different symmetry than planar isotropic or orthotropic reinforcements. Examples include some triaxial woven preforms, braided mats, or mixed lay-ups. Test specimen layout definition for these reinforcements is beyond the scope of this document. Sampling of these materials should be done with the prior approval of the customer.

3.5 Ambient Conditions—These are room temperature laboratory conditions, which are assumed to be 23 °C ± 2 °C and 50% ± 5% RH.

4. General Requirements

4.1 Calibrations—All measuring equipment for determining or controlling physical, mechanical, or thermal characteristics shall be calibrated and certified with traceability to the National Institute of Standards and Technology.

- 4.2 Equivalence**—Any substitution of supposed equivalent items, where “or equivalent” is specified herein, must first be approved by the customer.
- 4.3 Responsibility for Quality**—Unless otherwise specified, the supplier is responsible for all quality inspection and for meeting all of the test requirements specified herein. The supplier may use internal testing facilities or any other qualified test agencies.
- 4.4 Data Reporting**—All physical, thermal, and mechanical data and information on the constituent materials and panel molding shall be submitted to the customer using the Supplemental Data Reporting Package (SDRP). All data requested herein shall be manually recorded onto the SDRP forms or entered into the electronic SDRP program for output and transmittal to the customer. Graphs or other requested traces shall also be packaged and sent with the SDRP to the customer.

The SDRP Data Reporting Program (EA-2253) available from SAE as a 3-1/2 in or a 5-1/4 in diskette, is a menu-driven program, which facilitates data entry and reporting using an IBM-compatible personal computer. This program is the preferred method for reporting data.

The SDRP reporting forms are found in Appendix A. These include a “Constituent Materials/Process Data Sheet” (A3-A5) for the raw materials and processing data. This form may be modified as required to provide additional relevant information.

5. Panel Fabrication

5.1 General Panel Requirements

- 5.1.1 NUMBER OF PANELS**—Six panels shall be tested for any given material (a material is defined here as any particular combination of resin, reinforcement, and filler). Sufficient panels should be molded to assure that six panels are available after potential rejections cited in 5.1.4, 5.1.6, 5.3.2, or 5.4.2.
- 5.1.2 CONSTITUENT MATERIALS**—All of the panels shall be from a single production lot of composite using single batches of resin, fiber, filler, and additives. The resin type, all matrix constituents and fibers used in molding are to be clearly defined. RECORD IN SDRP.
- 5.1.3 PREFORM/MOLD CHARGE LAY-UP**—To allow for a direct comparison to be made among material systems, all plies shall be aligned in the same direction. Every ply in a given plaque shall also be the same reinforcement form and product code. Mixed lay-ups may be considered for submittal, but should be discussed with the customer prior to panel fabrication. If multiple plies are used, the plies must be aligned and directionally indicated. The number of plies must also be indicated. RECORD IN SDRP.
- 5.1.4 PANEL QUALITY**—Evidence of a flawed or an improperly prepared test panel shall be cause for discarding the panel prior to testing and fabrication of a replacement panel. Such causes include incorrect lay-up sequence, incorrect cure cycle, incorrect fiber content, fiber misalignment, nonfilling of mold cavity, blisters, excessive porosity, excessive void content, severe panel warpage, resin rich surfaces, thickness variation across the plaque, or compression of the preform towards the surface.
- 5.1.5 PANEL DIMENSIONS**—Nominal panel dimensions shall be 610 mm x 610 mm.
- 5.1.6 PANEL THICKNESS**—Panel thickness shall be 3.2 mm ± 0.2 mm with maximum thickness run out of 0.2 mm.
- 5.2 Panel Molding**—Molding conditions such as injection time, mold temperature, and postcure schedules must be reported. Reaction conditions for the material must be representative of conditions which would be utilized to mold an actual production part and should be reported in complete detail. Panels may be fixtured during postcure if warpage is likely to occur. RECORD IN SDRP.

- 5.2.1 **LIQUID COMPOSITE MOLDING**—Thermoplastic or thermoset binders in the preform shall be “set” prior to molding. All panels shall be molded in a center-gated mold. Following the cure, care should be taken to avoid unnecessary flexing of the plaque during demolding. Postcure is permissible, but should be noted in the SDRP.
- 5.2.2 **SMC MOLDING**—The structural SMC molding charge must be prepared in a manner consistent with production molding practice. If the material will be used with 80% mold coverage, a similar coverage should be used in the fabrication of test plaques. Using 80% as an example, the charge should consist of one or more plies cut to a square configuration and placed over the central 430 mm x 430 mm area of the mold. The charge should be from properly matured material and should be thermally equilibrated to ambient temperature prior to molding. Following cure, care should be taken to avoid unnecessary flexing of the plaque during demolding. Postcure is permissible but should be noted in the SDRP.
- 5.2.3 **THERMOPLASTIC COMPRESSION MOLDING**—Charge preparation and coverage should be similar to the procedure used for thermosetting SMC materials. Charge preheating should be done in a manner similar to actual production molding. Any process deviation used in charge preheating must be noted. Since the cooling rate and dwell time will influence the crystallinity level in some materials, care should be taken to reproduce the thermal cycle encountered in production. Any release agents used in component molding should also be used in plaque molding.

5.3 Orthotropic Composites

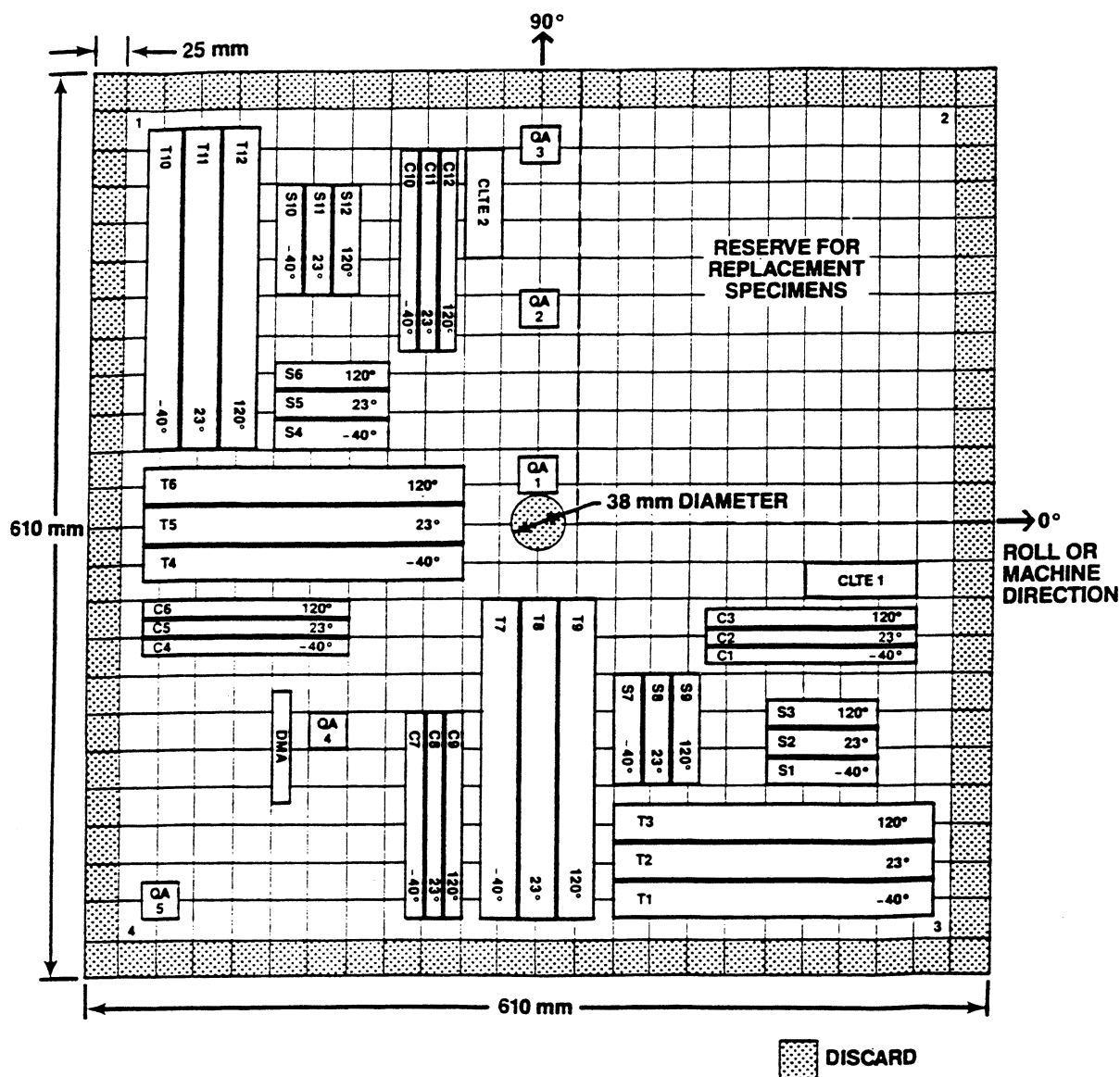
- 5.3.1 **FIBER ORIENTATION**—The primary fiber direction of the preform or mold charge is defined as 0° and is depicted in Figure 1.
- 5.3.2 **FIBER CONTENT**—The preferred fiber content for composites with orthotropic fiber orientation is 45% ± 2% by volume. See Section 14 for conversion between weight percent and volume percent. If this fiber content is not practical, alternative fiber contents of 35% ± 2% or 25% ± 2% by volume may be used. Consult with the customer if none of these fiber contents are acceptable.

5.4 Planar Isotropic Composites

- 5.4.1 **PANEL TEST ORIENTATION**—For materials with a nominal planar isotropic fiber orientation, a processing parameter such as roll direction for continuous strand mat products or machine direction for SMC will be defined as 0° as depicted in Figure 1 and designated in the SDRP tables.
- 5.4.2 **FIBER CONTENT**—The preferred fiber content for composites with planar isotropic fiber orientation is 25% ± 2% by volume. See Section 14 for conversion between weight percent and volume percent. If this fiber content is not practical, alternative fiber contents of 35% ± 2% or 45% ± 2% by volume may be used. Consult with the customer if none of these fiber contents are acceptable.

6. Material Sampling

- 6.1 **Sampling Procedure**—Specimens for physical, thermal, and mechanical tests shall be cut from the panels fabricated according to Section 5 using procedures described in Section 7.
- 6.2 **Specimen Layout**—Specific specimen locations for Planar Isotropic Fiber-Reinforced composites and Orthotropic Fiber-Reinforced composites are shown in Figure 1. Samples shall be cut and labeled per the codes indicated in Figure 1. This sample coding scheme is consistent with the SDRP. For liquid-molded panels, the center-gate area shall not be used and should be discarded. For all panels, the 25 mm trim areas shaded in Figure 1 shall not be used for specimen sampling and should be discarded.



SPECIMEN LOCATION TEMPLATE

SPECIMEN DIMENSIONS

TENSILE (T)	215 mm x 25 mm	COMPRESSION (C)	140 mm x 12.5 mm
SHEAR (S)	75 mm x 20 mm	CLTE	75 mm x 25 mm
DMA	65 mm x 10 mm	QA	25 mm x 25 mm

FIGURE 1—SPECIMEN LOCATION

7. Specimen Preparation

- 7.1 General**—The techniques described in this section are recommended requirements to ensure good quality test specimens produced in a consistent manner.
- 7.2 Rough Cutting**—Rough cutting of specimens can be accomplished by machining operations by cutting pieces from the panel to a size approximating the specimen configuration. The specimen layout plan shown in Figure 1 shall be used for specimen location.
- 7.3 Surface Edge Finishing**—Final surface edge preparation can be accomplished by surface grinding. The finish sanding should be done only in the direction of the length of the specimens and the sanding grit size should be 180 grit or finer. Sanding or grinding perpendicular to the length of the specimen may result in lower failure strengths because of high-stress concentrations at the scratch marks. No sanding is required, however, if specimens are cut with a diamond saw using a 180 grit or finer diamond blade and a minimum tip speed of 200 m/min. Water cooling of the saw blade is recommended for all materials, but is required for urethane materials.
- 7.4 Specimen Conditioning**—All specimens shall be conditioned prior to testing per ASTM D 618, Method A. For tests at any temperature other than ambient, specimens shall be further conditioned at the test temperature for 30 min immediately prior to testing.

8. Physical Test Methods

- 8.1 General**—It is desirable that the tests in 8.2 to 8.5 be performed on the same specimens. The specimen size for each test performed shall be 25 mm x 25 mm and shall be taken from the QA locations designated in Figure 1.
- 8.2 Specific Gravity**—Specific gravity shall be determined in accordance with ASTM D 792. RECORD IN SDRP.
- 8.3 Resin Content**—Resin content shall be determined in accordance with ASTM D 2584. See Section 14 for conversion between weight percent and volume percent. RECORD IN SDRP.
- 8.4 Filler Content**—Filler content shall be determined in conjunction with ASTM D 2584. Appropriate procedures (for example, GM Engineering Standard—Materials and Process Procedure GM 9077-P) utilizing acid wash for calcium carbonate filler shall be used to determine the amount of particulate fillers in the composite. See Section 14 for conversion between weight percent and volume percent. RECORD IN SDRP.
- 8.5 Fiber Content**—Fiber content shall be determined in accordance with ASTM D 2584. For reinforcements other than glass fiber, consult the customer. See Section 14 for conversion between weight percent and volume percent. RECORD IN SDRP.
- 8.6 Panel Thickness**—Panel thickness uniformity shall be determined as follows:
- Measure the thickness of each panel three separate times at each corner (25 mm from each edge) and at a region near the center of the panel (but away from the discarded gate region for liquid-molded panels).
 - Using all three measurements taken, report the average value for that region of the panel. RECORD IN SDRP.
- 8.7 Crystallinity**—For semi-crystalline thermoplastic composites, heat of fusion and heat of crystallization shall be determined in accordance with ASTM D 3417. An appropriate sample shall be taken from an area near the DMA specimen as designated in Figure 1. A Differential Scanning Calorimeter (DSC) shall be used to determine the areas under crystallization exotherm and fusion endotherm. RECORD IN SDRP.

8.8 Molecular Weight—For thermoplastic composites, molecular weight averages and molecular weight distribution shall be determined in accordance with ASTM D 5296. The “universal calibration” principles outlined in ASTM D 3593 or a suitable monodisperse polymeric standard shall be used for calibrating the test equipment. An appropriate sample shall be taken from an area near the DMA specimen as designated in Figure 1. High Performance Size-Exclusion Chromatography (HPSEC) shall be used to determine number and weight average molecular weights and molecular weight distribution. RECORD IN SDRP.

9. Thermal Test Methods

9.1 General—Dynamic Mechanical Analysis (DMA) and Coefficient of Linear Thermal Expansion (CLTE) tests shall be conducted on each of the specimens labeled DMA and CLTE, respectively, in Figure 1.

9.2 DMA Test Method—Dynamic mechanical analysis testing shall be conducted in accordance with ASTM D 4065 with the following provisions or exceptions cited herein.

9.2.1 **INSTRUMENTATION**—A Rheometrics RDS II or TA System's (formerly DuPont's) 983 Dynamic Mechanical Analyzer or instrumentation which provides equivalent results shall be utilized. The instrument utilized in the evaluation shall be calibrated in accordance with the manufacturer's recommendations.

9.2.2 **SAMPLE PREPARATION**—Test specimen preparation procedures described in Section 7 shall be followed to ensure good quality specimens.

9.2.3 **SAMPLE DIMENSIONS**—Sample dimensions for the previously named instruments shall be at least 65 mm x 10 mm x 3.2 mm. Distance between the instrument clamps shall be at least 40 mm. Sample dimensions for testing with other instrumentation may be selected using ASTM D 4065 guidelines.

9.2.4 **TEST TEMPERATURE RANGE**—A temperature sweep shall be conducted from -50 °C to an upper limit temperature which shall be at least 40 °C above the material's glass transition or 200 °C, whichever is higher.

9.2.5 **HEATING RATE**—The heating rate shall be no greater than 5 °C/min (5 °C steps with 1 min equilibration for the previously named Rheometrics instrumentation).

9.2.6 **FREQUENCY**—The frequency shall be maintained at 1 Hz (6.28 radians/s) for fixed frequency instrumentation.

9.2.7 **OTHER TEST PARAMETERS**—Strain shall be maintained at a minimum value which allows the measured stress to exceed the lower limit of sensitivity of the transducer. Typical strain levels are 0.02 to 0.2%. When possible, an amplitude of 0.6 mm should be utilized with other fixed frequency instrumentation. Test parameters for resonant frequency experiments (e.g., sample length) should be adjusted so that an initial frequency of 30 Hz at 0.2 mm amplitude is obtained at the onset of the experiment.

9.2.8 **GLASS TRANSITION TEMPERATURE**—The glass transition temperature (T_g) shall be determined from the storage modulus data. A line is drawn tangent to the storage modulus plateau at temperatures below the transition and a second line is drawn tangent to the storage modulus curve beginning at the transition inflection point, which is approximately midway through the sigmoidal curve associated with the transition. The temperature at which these two tangent lines intersect is reported as the glass transition temperature (see Figure 2).

9.2.9 **REPORT**—The test report shall include measured transition temperatures and thermograms for either shear moduli (G' , G'' , and $\tan \delta$) or flexural moduli (E' , E'' , and $\tan \delta$) versus temperature. One specimen per panel shall be run. RECORD IN SDRP AND PROVIDE THERMOGRAMS.

9.3 Coefficient of Linear Thermal Expansion (CLTE)

- 9.3.1 CLTE TESTING—Thermal expansion properties shall be measured in accordance with ASTM D 696 with the following provisions and exceptions cited herein.
- 9.3.2 INSTRUMENTATION—A fused-quartz dilatometer equipped with precise length measuring devices such as Linear Variable Differential Transformer (LVDT) shall be used to record change in length of the specimen as a function of temperature.
- 9.3.3 SAMPLE DIMENSIONS—The test specimen shall be of constant cross-section and the specimen size is 75 mm x 25 mm x 3.2 mm. Dimensional variation of specimen length shall be limited to 0.25 mm.
- 9.3.4 SAMPLE PREPARATION—Test specimen preparation procedures described in Section 7 shall be followed to ensure good quality specimens.
- 9.3.5 PROCEDURE—The CLTE shall be measured and/or reported over the following temperature ranges:
- a. -30 to 30 °C
 - b. 30 to 80 °C
 - c. 80 to 125 °C

If the instrumentation allows, the change in length should be monitored continuously or in increments of 20 °C over the temperature range of the test.

- 9.3.6 REPORT—The CLTE shall be reported over the ranges a, b, and c. If the data can be obtained from the instrumentation, a table showing $\Delta l/l$ values at 20 °C intervals should be provided. Use the specimen original length (l) at -30 °C as the “zero” point for $\Delta l/l$. RECORD IN SDRP AND PROVIDE TABULATED DATA.

10. Tensile Testing

- 10.1 General Description—Tensile testing shall be conducted in general accordance with ASTM D 3039 and ASTM D 5083 with the following provisions or exceptions cited herein. Mechanical properties determined shall include the following: tensile strength, modulus, strain to failure, Poisson's ratio, and energy to failure at -40 °C, 23 °C, and 120 °C. These tests shall be conducted on each of the appropriately labeled specimens in the numbers and directions indicated in Figure 1.

NOTE—If a material cannot be tested at 120 °C because it is too close to the resin T_g or exceeds the resin T_g, contact the customer to discuss an appropriate lower temperature.

- 10.2 Apparatus—Grips shall be self-aligning, so that the long axis of the specimen is aligned with the direction of the applied load through the centerline of the grip assembly. The specimen should not have any rotary motion which may induce slippage in the grips. Fine serrations on grip faces have been found to be effective for composite materials. A biaxial extensometer or strain gage accurate to 1% of the measured strain shall be used for measuring Poisson's ratio. Extensometer or strain gage attachment should not cause damage to the specimen surface.

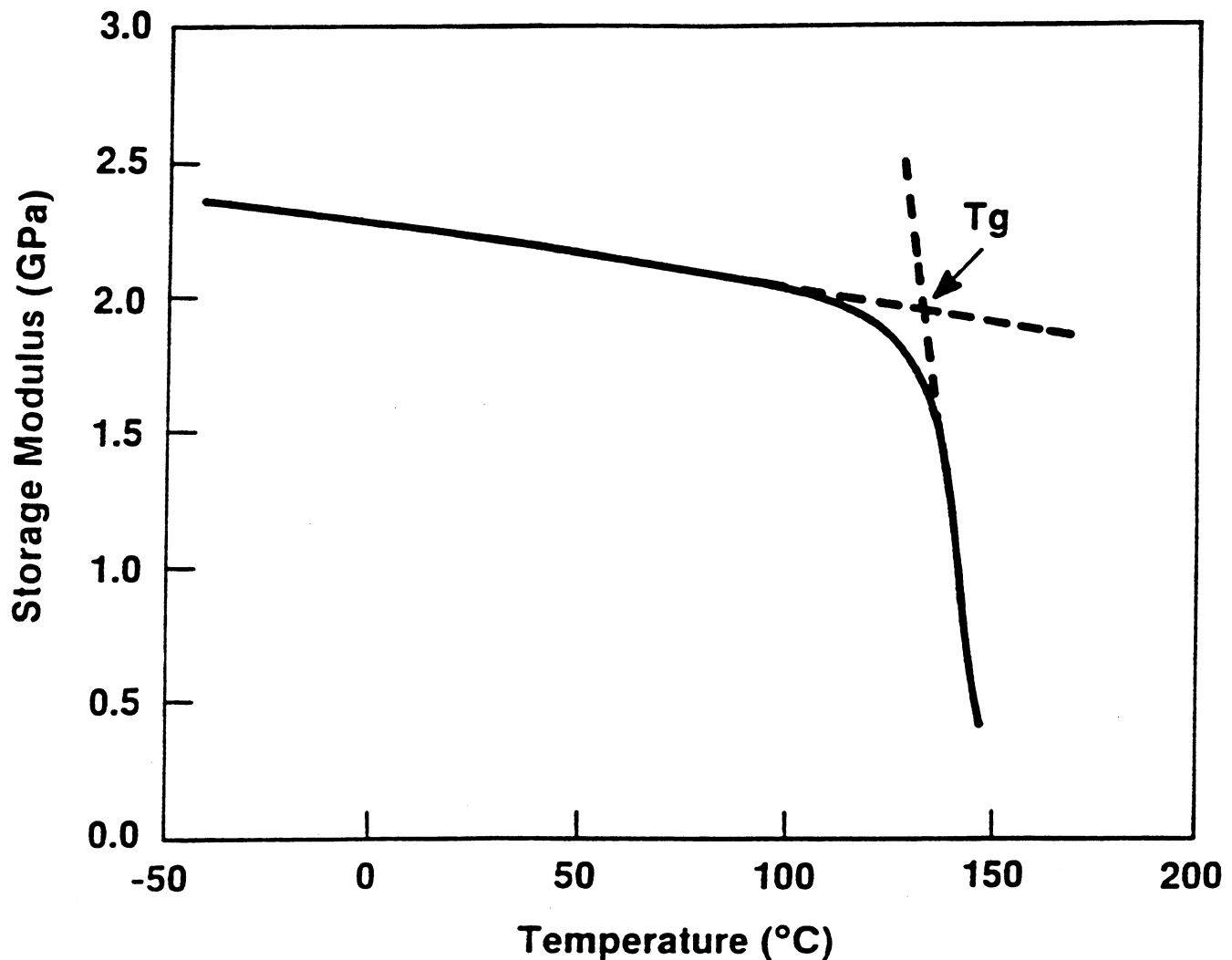


FIGURE 2—STORAGE MODULUS DIAGRAM

10.3 Test Specimen

- 10.3.1 SPECIMEN DIMENSIONS—The test specimen shall be of constant cross-section and the specimen size is 215 mm x 25 mm x 3.2 mm for random fiber composites and 215 mm x 12.5 mm x 3.2 mm for 0 degree continuous fiber composites. Dimensional variation of specimen width shall be limited to ± 0.15 mm. Measure samples to nearest 0.025 mm.
- 10.3.2 SPECIMEN PREPARATION—Test specimen preparation procedures described in Section 7 shall be followed to ensure good quality samples.
- 10.3.3 SPECIMEN TABBING—Tabbing of specimens is not required unless unacceptable failures, as defined in 10.4.3, are encountered.

10.4 Procedure

- 10.4.1 **SETUP**—Measure the width and thickness of the flat specimens with a suitable micrometer to the nearest 0.025 mm at a minimum of three points along its length. Calculate and record the cross-sectional area based on the average width and thickness measurements. Place the specimen in the grips with a 115 mm separation between the ends of the gripping surfaces. Attach the extensometer or connect the strain gage and record load-extension curve of the specimen. The speed of testing shall be the relative motion of the grips or test fixtures during the test. The standard speed of testing shall be 5 mm/min (0.2 in/min).
- 10.4.2 **REPLICATES**—Twelve specimens from each of six panels shall be tested in the directions, temperatures, and locations indicated in Figure 1.
- 10.4.3 **ACCEPTABLE FAILURES**—Specimens failing closer than 12.5 mm to the grip shall be discarded. If a significant number of at-the-grip failures is encountered, tabs as defined in ASTM D 3039 shall be applied. Most composites containing 0 degree continuous fibers will require tabbing.

10.5 Calculations—Determine the tensile strength, chord modulus, Poisson's ratio, and percent strain at failure using the standard procedures. The chord modulus shall be calculated using the 0.05% and 0.25% strain and corresponding stress values. Poisson's ratio shall be calculated using the 0.05% and 0.25% longitudinal strain and corresponding transverse strain values. The area under the stress-strain curve up to the point of failure (maximum stress) shall be determined and reported as the tensile energy to failure in units of MJ/m³. If the energy-to-failure is not reported for a material, the customer will approximate this number as one-half the product of the stress at failure multiplied by the strain at failure. This approximation may result in an underestimation of the energy-at-failure.

10.6 Report—The report shall include the following:

- a. Individual tensile strength values, average value, and standard deviation
- b. Tensile (chord) moduli, average value, and standard deviation
- c. Tensile strains to failure, average value, and standard deviation
- d. Poisson's ratio, average value, and standard deviation
- e. Tensile energies to failure, average value, and standard deviation
- f. A representative example of the stress/strain or load/extension curve

RECORD IN SDRP AND PROVIDE CURVE.

10.7 Resin Tensile Properties—Determine the tensile strength, modulus, and strain to failure for the neat, cured resin in accordance with ASTM D 638. RECORD ON SDRP.

11. Compression Testing

11.1 General Description—Compression testing shall be conducted according to ASTM D 3410, Procedure B (IITRI method), with the exceptions and/or provisions cited herein. Mechanical tests shall include compressive strength at -40 °C, 23 °C, and 120 °C. Modulus, strain to failure, and energy to failure at these temperatures are optional. These tests shall be conducted on each of the labeled specimens in the numbers and directions indicated in Figure 1.

NOTE—If a material cannot be tested at 120 °C because it is too close to the resin T_g or exceeds the resin T_g, contact the customer to discuss an appropriate lower temperature.

11.2 Compression Test Fixture—Test fixture dimensions shall correspond to those found in ASTM D 3410, Procedure B for specimens 12.5 mm wide.