Designation: D7291/D7291M – 15

Standard Test Method for Through-Thickness "Flatwise" Tensile Strength and Elastic Modulus of a Fiber-Reinforced Polymer Matrix Composite Material[1]

This standard is issued under the fixed designation D7291/D7291M; 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 determines the through-thickness "flatwise" tension strength and elastic modulus of fiber reinforced polymer matrix composite materials. A tensile force is applied normal to the plane of the composite laminate using adhesively bonded thick metal end-tabs. The composite material forms are limited to continuous-fiber or discontinuous fiber (tape or 2-dimensional fabric, or both) reinforced composites.

1.2 The through-thickness strength results using this test method will in general not be comparable to Test Method D6415 since this method subjects a relatively large volume of material to an almost uniform stress field while Test Method D6415 subjects a small volume of material to a non-uniform stress field.

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.4 *This standard does not purport to address all of the safety problems, 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]

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

D883 Terminology Relating to Plastics

D3171 Test Methods for Constituent Content of Composite Materials

D3878 Terminology for Composite Materials

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

D5687/D5687M Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation

D6415 Test Method for Measuring the Curved Beam Strength of a Fiber-Reinforced Polymer-Matrix Composite

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

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

E251 Test Methods for Performance Characteristics of Metallic Bonded Resistance Strain Gages

E456 Terminology Relating to Quality and Statistics

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

E1012 Practice for Verification of Testing Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application

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]

[1] This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.06 on Interlaminar Properties.

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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 high-modulus fibers and their composites. Terminology D883 defines terms relating to plastics. Terminology E6 defines terms relating to mechanical testing. Terminology E456 and Practice E177 define terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the other terminologies.

NOTE 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 with square brackets, as the symbols may have other definitions when used without the brackets.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *flatwise tensile ultimate strength, F^{tu} [M L^{-1} T^{-2}], n*—the ultimate strength of the composite material in the out-of-plane (through-thickness) direction.

3.2.2 *through-thickness tensile modulus, E^{chord} [M L^{-1} T^{-2}], n*—the chord modulus of elasticity of the composite material in the out-of-plane (through-thickness) direction.

3.3 *Symbols:*

3.3.1 *A*—cross-sectional area of specimen in the through-thickness direction,

3.3.2 *CV*—coefficient of variation statistic of a sample population for a given property (in percent),

3.3.3 E^{chord}— through-thickness tensile modulus.

3.3.4 F^{tu}— flatwise tensile ultimate strength.

3.3.5 *n*—number of specimens.

3.3.6 P_{max}— maximum force carried by test specimen before failure.

3.3.7 s_{n-1}—sample standard deviation.

3.3.8 x_i— measured or derived property for an individual specimen from the sample population.

3.3.9 $\bar{x}$—sample mean (average).

3.3.10 ε—indicated through-thickness tensile strain from strain transducer.

3.3.11 σ—through-thickness tensile stress.

4. Summary of Test Method

4.1 A composite specimen in the shape of either a straight-sided cylindrical disk or a reduced gage section cylindrical "spool" is adhesively bonded to cylindrical metal end tabs. The bonded assembly is loaded under "flatwise" tension loading by a force applied normal to the plane of the composite laminate until failure of the laminate occurs (Fig. 1). The test is considered valid only when failure occurs entirely within the composite laminate. The test is considered invalid if failure of the bond-line or partial failure of the bond-line and the surface layer of the composite occurs. The failure mode of this test is not controlled; therefore, the actual failure may be intralaminar or interlaminar in nature.

4.2 If force-strain data are required, the specimen may be instrumented with strain gages provided certain specimen thickness requirements are satisfied (see 8.2).

5. Significance and Use

5.1 This test method is designed to produce through-thickness failure data for structural design and analysis, quality assurance, and research and development. Factors that influence the through-thickness tensile strength, and should therefore be reported, include the following: material and fabric reinforcement, methods of material and fabric preparation, methods of processing and specimen fabrication, specimen stacking sequence, specimen conditioning, environment of testing, specimen alignment, speed of testing, time at temperature, void content, and volume reinforcement content.

6. Interferences

6.1 *Material and Specimen Preparation*—Poor material fabrication practices, lack of control of fiber alignment, voids, and

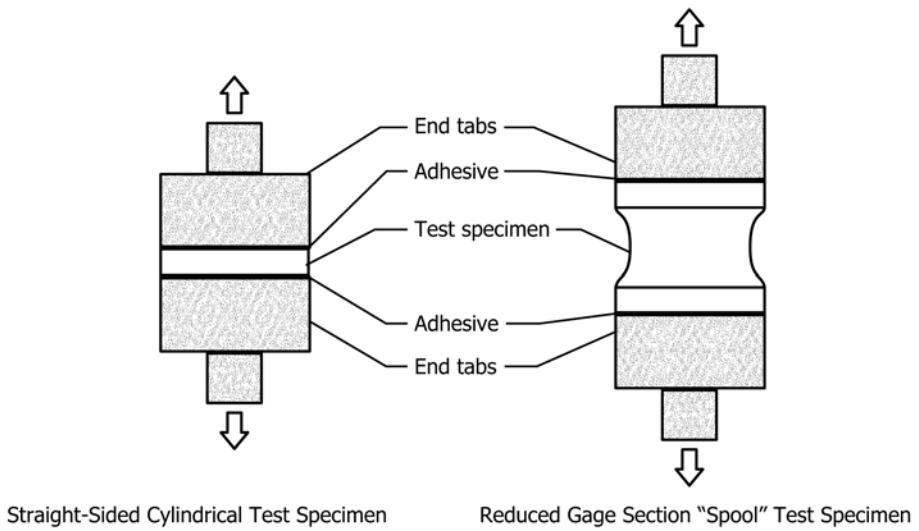

FIG. 1 Flatwise Tension Specimen and End Tab Assembly

damage induced by improper specimen machining are known causes of high material data scatter in composites in general. In addition, surface finish of the cylindrical machined surface and lack of control of parallelism of laminate surfaces can lead to erroneous through-thickness strength results. Laminate stacking sequences that are not balanced and symmetric could lead to adhesive bondline failures.

6.2 *Material with Coarse Structure*—This test method assumes that the material is relatively homogeneous with respect to the size of the test section. Certain fabric and braided composites with large repeating unit cell sizes (>12 mm [0.5 in.]) should not be tested with this specimen size. It may be possible to scale-up the specimen size and fixtures to accommodate such materials, but this is beyond the scope of this test method.

6.3 *Load Eccentricity*—Bending of the specimen during loading can occur, affecting strength results. Bending may occur due to poor specimen preparation, non-parallel laminate surfaces, improper bonding of the specimen to the end tabs, or machine/load train misalignment.

6.4 *Void content*—The through-thickness tension strength measured using this method is extremely sensitive to reinforcement volume and void content. Consequently, the test results may reflect manufacturing quality as much as material properties.

7. Apparatus

7.1 *Micrometers*—The micrometer(s) shall use a 4 to 6 mm [0.16 to 0.25 in.] diameter ball-interface on irregular surfaces such as the bag-side of a laminate, and a flat anvil interface on machined or very-smooth tooled surfaces. The accuracy of the instrument(s) shall be suitable for reading to within 1 % of the sample diameter and thickness. For typical specimen geometries an instrument with an accuracy of ±25 µm [±0.001 in.] is desirable for both diameter and thickness measurements.

7.2 *Fixtures*—The apparatus consists of three different fixtures.

7.2.1 The loading fixtures are used to load the specimen and end tab assembly. They can be either self-aligning or fixed grip and shall not apply eccentric loads.

7.2.2 The end tabs are bonded to the specimen (Figs. 2 and 3). The end tabs are attached to the loading fixture during the test. The threads on the end tabs provide a means to attach the specimen and end tab assembly to the loading fixture. They also provide a means to attach constant diameter bushings for the purpose of aligning the specimen and end tab assembly in the bonding fixture. The end tab thickness shall be a minimum of 12.7 mm [0.5 in.]. Section 8.3 provides further requirements for the end tabs.

7.2.3 The end tab bonding fixture (Figs. 4-6) is used to provide support and alignment to the specimen and end tab assembly during the entire bonding process. The threads on the end tabs are used to attach bushings to them during the bonding process. These bushings provide a fixed diameter reference surface for aligning the specimen and end tab assembly during bonding, thus allowing the resuse and re-machining of the end tabs.

7.3 *Testing Machine*—The testing machine shall conform with Practice E4, and shall satisfy these requirements:

7.3.1 *Testing Machine Heads*—The testing machine shall have two crossheads, with either a stationary head and a movable head or two movable heads.

7.3.2 *Platens/Adapter*—One of the testing machine heads shall be capable of being attached to the lower half of the specimen end tab by an adapter or platen interface as required.

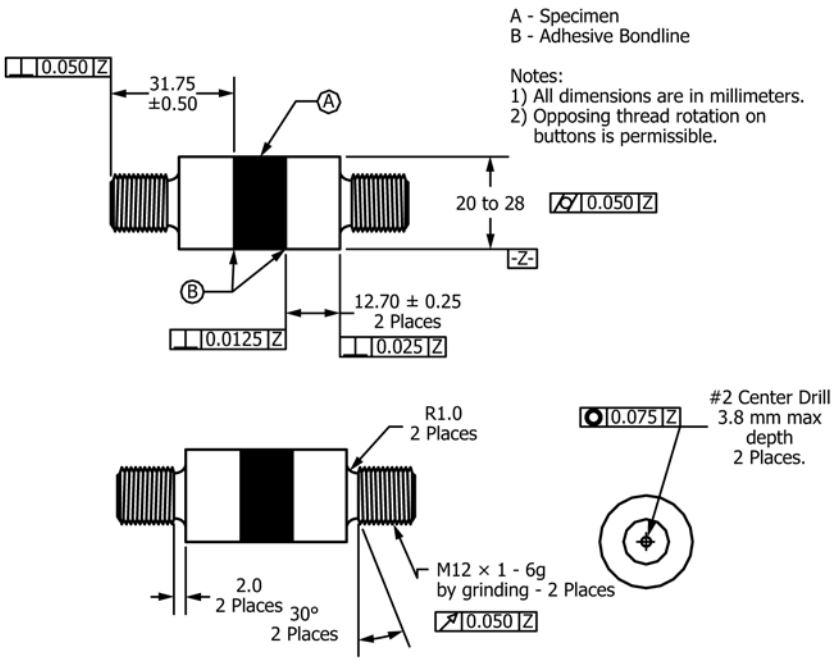

FIG. 2 Drawing of End Tabs and Cylindrical Specimen Assembly (SI units)

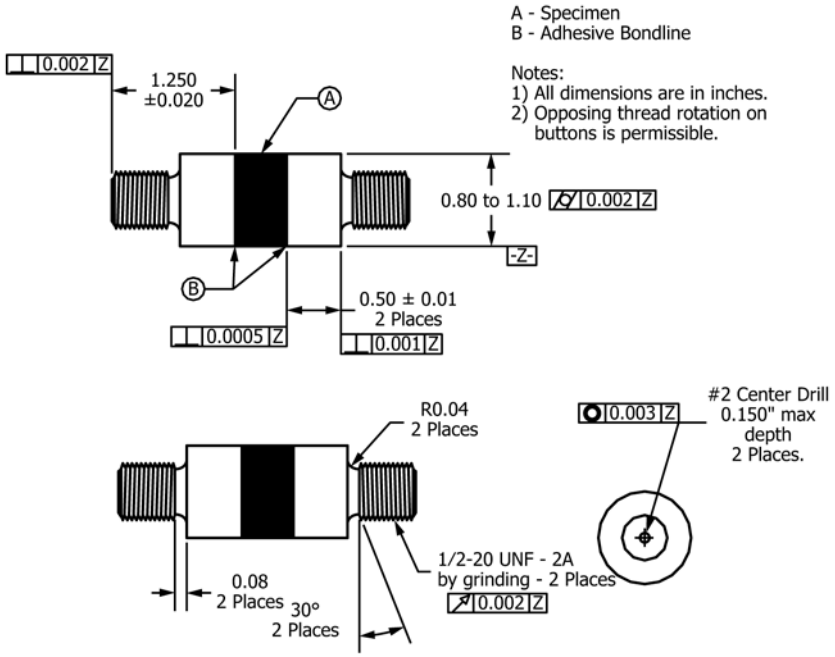

FIG. 3 Drawing of End Tabs and Cylindrical Specimen Assembly (inch-pound units)

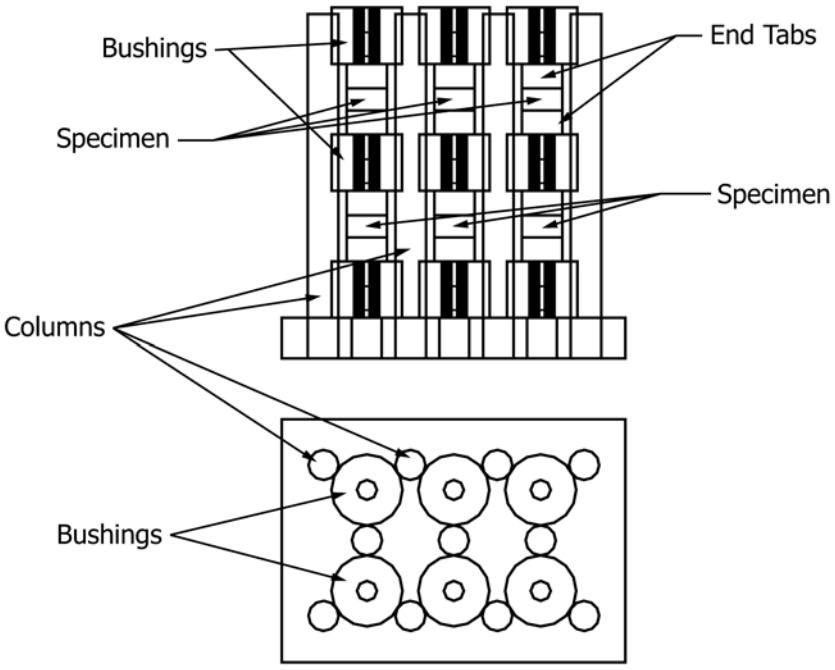

FIG. 4 Drawing of Alignment and Bonding Fixture (showing 12 specimens)

The other head shall be capable of being attached to the upper half of the specimen end tab.

7.3.3 *Drive Mechanism*—The testing machine drive mechanism shall be capable of imparting to the movable head a controlled velocity with respect to the stationary head. The velocity of the movable head shall be capable of regulation as specified in 11.3.

7.3.4 *Force Indicator*—The testing machine force-sensing device shall be capable of indicating the total force applied to the test specimen. This device shall be essentially free from response lag at the specified testing rate and shall indicate the force with an accuracy over the load range(s) of interest of within ±1 % of the indicated value, as specified by Practice

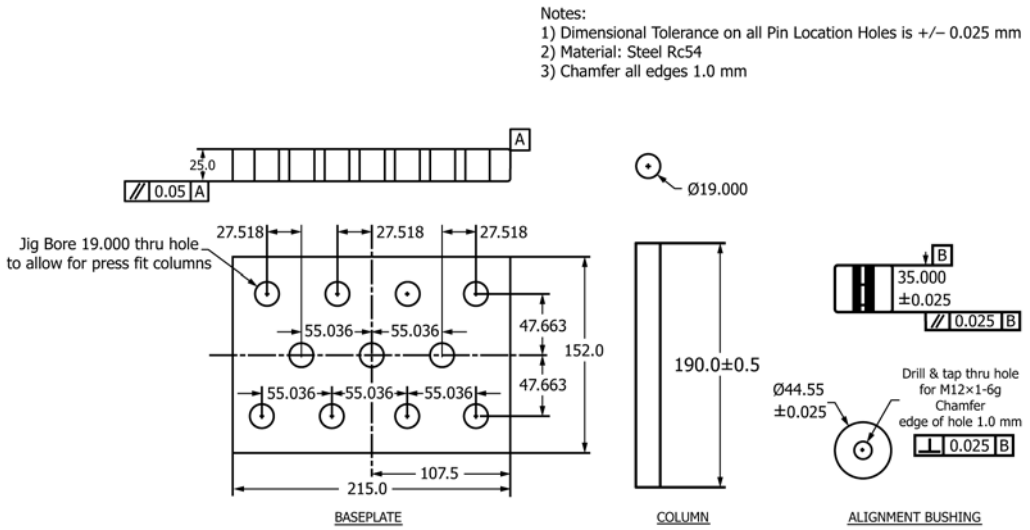

FIG. 5 Drawing of Alignment and Bonding Fixture (SI units)

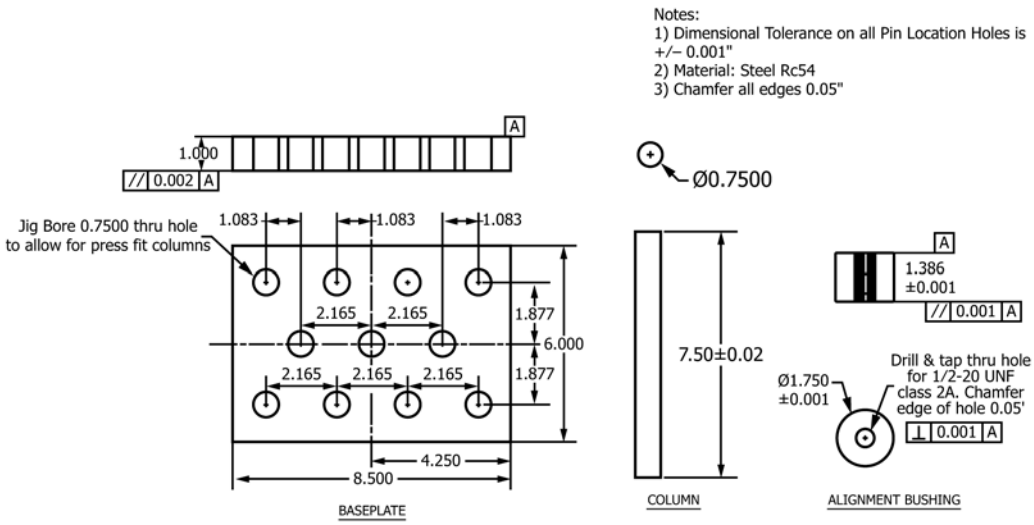

FIG. 6 Drawing of Alignment and Bonding Fixture (inch-pound units)

E4. The load range(s) of interest may be fairly low for modulus evaluation, much higher for strength evaluation, or both, as required.

7.4 *Force versus Displacement Record*—An X-Y plotter, or similar device, shall be used to make a permanent record of the force versus displacement during the test. Alternatively, the data may be stored digitally and post-processed.

7.5 *Strain-Indicating Device*—For the measurement of through-thickness modulus, bonded resistance strain gages shall be used to measure strain. Either two strain gages at locations that are 180 degrees apart or three strain gages at 120 degrees apart are required around the cylindrical surface of the specimen at the center of the gage section.

7.5.1 *Bonded Resistance Strain Gages*—Strain gage selection is a compromise based on the type of material. An active gage length of 1.5 mm [0.062 in.] is recommended for most materials although larger gages may be more suitable for some woven fabrics (with consolidated tow thicknesses larger than 1.5 mm [0.062 in.]), provided the specimen gage length can accommodate such gages (as specified in 8.2). Gage calibration certification shall comply with Test Method E251. For laminated composites, the strain gage should cover a minimum of three laminate plies.

7.6 *System Alignment*—Poor system alignment can be a major contributor to premature failure, to elastic property data scatter, or both. Practice E1012 describes bending evaluation guidelines and describes potential sources of misalignment during tensile testing. Alignment should be checked using a cylindrical metal specimen with a minimum of three strain gages equally spaced around the circumference per Practice E1012. While the maximum advisable amount of system misalignment is material and location dependent, good testing practice is generally able to limit percent bending to within 5 % at moderate strain levels (>1000 με). A system showing excessive bending for the given application should be re-adjusted or modified.

7.7 *Conditioning Chamber*—When conditioning materials in other than ambient laboratory environments, a temperature/

vapor-level controlled environmental conditioning chamber is required, that shall be capable of maintaining the required relative temperature to within ±3°C [±5°F] and the required relative vapor level to within ±3 %. Chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

7.8 *Environmental Test Chamber*—An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the gage section of the test specimen within ±3°C [±5°F] of the required test temperature during the mechanical test. In addition, the chamber may have to be capable of maintaining environmental conditions such as fluid exposure or relative humidity during the test (see 11.4).

8. Sampling and Test Specimens

8.1 *Sampling*—Test at least five specimens per test condition unless valid results can be gained through the use of fewer specimens, such as in the case of a designed experiment. Consult Practice E122 to determine statistically appropriate sample sizes. The method of sampling shall be reported.

8.2 *Geometry*—The coupons are cylindrical with either a constant cross-sectional area (Figs. 2 and 3) or a reduced gage section (Figs. 7 and 8). The nominal diameter where the specimen is bonded to the metal end tabs, for both specimen types, is 25 mm [1.0 in.]. However, this diameter can be in the range of 20 to 28 mm [0.8 to 1.1 in.] to allow the re-use and re-machining of the end tabs. For through-thickness failure stress measurement, the minimum specimen thickness shall be 2.5 mm [0.1 in.]. For the measurement of through-thickness strains and modulus, the minimum specimen thickness shall be 6 mm [0.25 in.]. The reduced gage section geometry is often used for materials that have a through-thickness strength that approaches the bond strength of the adhesive. This is also the preferred geometry for laminates that are at least 25 mm [1.00 in.] thick.

8.3 *Use of End-Tabs*—Tabs are required. The key factor in the selection of specimen tolerances and gripping methods is the successful introduction of load in the specimen and the prevention of premature failure due to misalignment. It is of primary importance that the bonding surfaces and threaded sections are perpendicular to minimize misalignment of the composite coupon and end-tab assembly. An additional consideration is the thermal residual stress caused by the significant difference between the laminate in-plane coefficient of thermal expansion (CTE) and the metal end tab CTE. This is especially important during end tab bonding, as well as during non-ambient testing. The end tab bonding surfaces shall be machined to the surface finish recommended by the adhesive manufacturer.

8.3.1 *End-Tab Geometry*—The end-tab geometry is shown in Figs. 2 and 3. For alignment purposes it is essential that the tab surfaces be parallel.

8.3.2 *End-Tab Material*—The most commonly used materials are Titanium (Ti-6Al-4V) and Steel. The end tab material should be selected appropriately for environmentally conditioned testing such that the conditioning does not chemically affect the end tabs. Aluminum is not recommended for most graphite composite materials since the low modulus of Aluminum leads to excessive deformations in the end tabs at the bond line leading to premature bond failures.

8.4 *Specimen Preparation*—Panels shall be fabricated and machined according to Guide D5687/D5687M. Panels with a balanced and symmetric cross-ply or quasi-isotropic stacking sequence are preferred. The dimensions of the specimen blanks shall be large enough to ensure that no damaged material

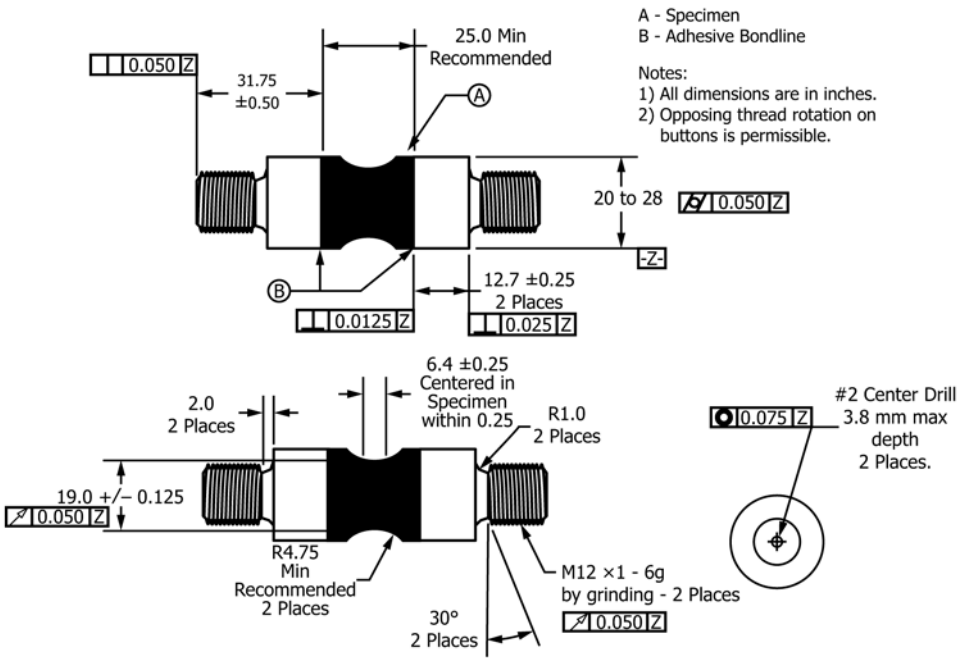

FIG. 7 Drawing of End Tabs and "Spool" Specimen Assembly (SI units)

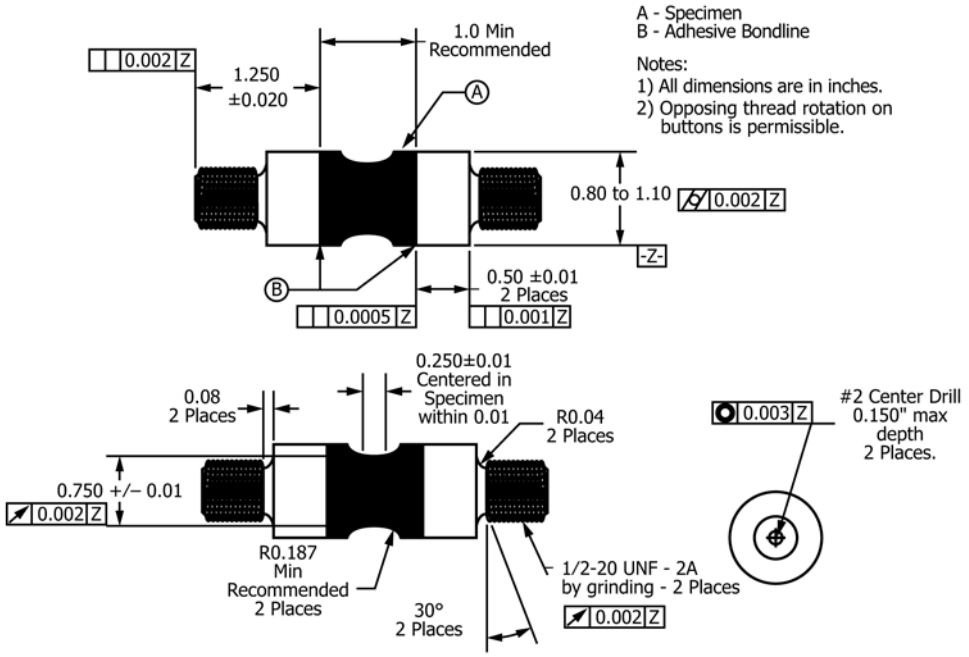

FIG. 8 Drawing of End Tabs and "Spool" Specimen Assembly (inch-pound units)

remains after final sample preparation. Prior to bonding, the parallelism and flatness of the sample surfaces shall be checked. Surface machining is not desirable, but grinding on one or both surfaces is permissible in cases where it is required to achieve the desired alignment.

8.5 *Adhesive*—Any high-elongation (tough) adhesive system that maintains a complete bond between the end tabs and the specimen up to failure of the composite may be used, provided it does not influence the specimen behavior by physically or chemically altering the composite. Strength of the adhesive and flow characteristics during the cure cycle are two of the important adhesive properties that must be considered when selecting an adhesive. Suitability of the adhesive to the anticipated test conditions and to any pre-conditioning of the specimens also needs to be considered. A uniform bond line of minimum thickness is desirable to reduce undesirable stresses in the assembly.

8.6 *Specimen Bonding*—Prior to bonding, the composite coupons shall be dried and the bonding surfaces wiped with a suitable cleaning solvent that will not chemically or physically affect the surfaces. Cleaned surfaces shall not be touched with the skin following cleaning. Apply the adhesive to the bonding surfaces of both end tabs and both sides of the composite specimen and place in a suitable bonding fixture. The bonding fixture shall be designed to provide support and alignment to the assembly during the entire bonding process. Figs. 4-6 provide drawings of a suggested fixture. Care must be taken to ensure that the composite coupon will not move during the bonding process. Cure the adhesive to the manufacturer's suggested cure cycle so long as this does not physically or chemically alter the composite. Label the coupon and end tabs assembly for traceability of the coupon back to the raw material.

8.7 *Machining*—After bonding, the composite specimen and end tab assembly must be machined to obtain the specified concentricity. Low stress grinding or turning techniques combined with the use of water as a coolant are the preferred method of machining. The machining process shall produce a smooth surface (better than 0.8 µm [32 µin.]), free of nicks and irregularities such as undercuts. The coolant used in the grinding or turning process, if water is not used, must not adversely affect the material. Specimens should be dried prior to testing to remove any moisture absorbed during the machining process. The end tabs can be re-used until their diameter after machining is 20 mm [0.8 in.].

8.8 *Re-use of End-Tabs*—The end-tabs may be reused and remachined as long as the geometry requirements for the end-tabs specified in Section 8.2 are met. Before reuse, the bonded specimens and adhesive need to be removed using an appropriate procedure. One such procedure is to heat the bonded specimen and end-tab assembly for an hour above the glass transition temperature of the adhesive. After specimen removal, the end-tab bonding surface can be grit-blast and cleaned using a suitable solvent.

8.9 *Void Content and Fiber Volume*—Through-thickness tension strength is very sensitive to void content and fiber volume fraction. The fiber volume fraction and void content may be determined using one of the procedures of Test Method D3171. It should be noted that the through-thickness tensile

strength is particularly sensitive to interply void content and that this can be different than void content throughout the laminate. The procedures noted measure the void content throughout the specimen, not focusing on the interply region.

9. Calibration

9.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment. Refer to Practice E4 for additional details on the calibration of the test frames.

10. Conditioning

10.1 The recommended pre-test specimen condition is effective moisture equilibrium at a specific relative humidity as established by Test Method D5229/D5229M; however, if the test requester does not explicitly specify a pre-test conditioning environment, conditioning is not required and the test specimens may be tested as prepared.

10.2 The pre-test specimen conditioning process, to include specified environmental exposure levels and resulting moisture content, shall be reported with the data.

NOTE 2—The term moisture, as used in Test Method D5229/D5229M, includes not only the vapor of a liquid and its condensate, but the liquid itself in large quantities, as for immersion.

10.3 If there is no explicit conditioning process the conditioning process shall be reported as "unconditioned" and the moisture content as "unknown."

11. Procedure

11.1 *Parameters to be Specified Prior to Test:*

11.1.1 The specimen sampling method, specimen geometry, and conditioning travelers (if required).

11.1.2 The properties and data reporting format desired.

NOTE 3—Determine specific material property, accuracy, and data reporting requirements prior to test for proper selection of instrumentation and data recording equipment. Estimate the operating stress and strain levels to aid in transducer selection, calibration of equipment, and determination of equipment settings.

11.1.3 The environmental conditioning test parameters.

11.1.4 If performed, the sampling method, specimen geometry, and test parameters used to determine density and constituent volumes.

11.2 *General Instructions:*

11.2.1 Report any deviations from this test method, whether intentional or inadvertent.

11.2.2 If specific gravity and density are to be reported, then obtain the samples from the same panels being tested. Specific gravity and density may be evaluated by means of Test Method D792.

11.2.3 Obtain samples from the same panels being tested for measurement of reinforcement volume and void content. Volume percent of the constituents may be evaluated by one of the procedures of Test Method D3171.

11.2.4 Bond the specimen to the end tabs using the recommended bonding fixture (Figs. 4-6 in accordance with the requirements of 8.6.

11.2.5 Machine the specimen and end tab assembly in accordance with the requirements of 8.7.

11.2.6 Condition the specimen and end tab assembly, either before or after strain gaging, as required. Store the specimens in the conditioned environment until test time, if the testing environment is different than the conditioning environment. Moisture absorption along the adhesive bond line may lead to degradation of the bond strength. Such moisture ingress may be prevented by the use of an appropriate masking agent along the adhesive bond line. Care should be exercised in minimizing the spill-over of the masking agent on to the surface of the specimen.

NOTE 4—Gaging before conditioning may impede moisture absorption locally underneath the strain gage, the conditioning environment may degrade the strain gage adhesive, or both. On the other hand, gaging after conditioning may not be possible for other reasons, or the gaging activity itself may cause loss of conditioning equilibrium. The timing on when to gage coupons is left to the individual application and shall be reported.

11.2.7 Apply strain gages to the specimen (see 7.5) if force-strain response or modulus of elasticity is to be measured.

11.2.8 Following final specimen machining and any conditioning, but before the testing, measure the specimen diameter at three locations around the circumference. Calculate the average and record it to the nearest 0.025 mm [0.001 in.]. Make note of any irregularities in the specimen that may influence the results.

11.3 *Speed of Testing*—The testing shall be performed at a constant crosshead displacement rate of 0.1 mm/min. [0.005 in./min.].

11.4 *Test Environment*—If possible, test the specimen under the same fluid exposure level used for conditioning. However, cases such as elevated temperature testing of a moist specimen place unrealistic requirements on the capabilities of common environmental chambers. In such cases the mechanical test environment may need to be modified, for example, by testing at elevated temperature with no fluid exposure control, but with a specified limit on time to failure after withdrawal of the specimen from the conditioning chamber. Record any modifications to the test environment.

11.5 *Specimen Insertion*—Place the specimen in the grips of the testing machine, taking care to align the long axis of the specimen and end tab assembly with the test direction.

11.6 *Transducer Installation*—Attach the strain recording instrumentation to the strain gages on the specimen (if applicable).

11.7 *Specimen Alignment*—It is recommended that at least one specimen per like sample be evaluated for alignment. Alignment of the test specimen should be determined as per Practice E1012. A minimum of three strain gages are required, at circumferential locations of 120 degrees apart, if bending deformation is to be detected. Specimens which exhibit strain response with greater than 5 % bending (at a strain level of 1000με or at 90 % of the expected ultimate strain) indicate unsuitable alignment for this test and may not represent accurate through-thickness tensile properties.

11.8 *Loading*—Apply a tensile force to the bonded specimen and end tab assembly in displacement control at a constant crosshead (or servo-hydraulic ram) displacement rate as specified in 11.3.

11.9 *Data Recording*—Record force versus crosshead displacement continuously or at frequent regular intervals. A plot of force versus crosshead displacement can assist in spotting test irregularities and other problems. If the specimen is strain gauged, strain data shall also be collected. If data is collected using an X-Y plotter, the x and y scales should be picked appropriately. If the data are collected digitally, the rate of sampling must be adequate to capture the response of the material and a minimum of 500 data points shall be recorded. For specimens that are to be tested to failure, record the maximum force, and the displacement (or strain) at, or as near as possible to, the moment of failure.

11.10 *Failure Mode*—Record the location and mode of failure. Acceptable failure modes are within the specimen at least one ply thickness away from the bond line. Four general failure modes can be noted, including: along a single plane within the gage section of the specimen (SG), along multiple planes within the gage section of the specimen (MG), partly through the specimen surface ply or plies and partly through the adhesive (SA), and adhesive failure along bond line (AB). The (SA) and (AB) failure modes are not acceptable failure modes and the strength data shall be noted as invalid. For the reduced gage section "spool" specimen, failures in the radius region (SGR) and outside the reduced gage section (OGR) are possible and are considered acceptable as long as OGR failures are at least one ply thickness away from the bond line.

12. Validation

12.1 Values for ultimate properties shall not be calculated for any specimen that breaks at some obvious flaw, unless such flaw constitutes a variable being studied. Retests shall be performed for any specimen on which values are not calculated.

12.2 A significant fraction of failures in a sample population occurring at the bond line(s) to the end tabs shall be cause to reexamine the means of force introduction into the material. Factors considered should include the fixture alignment, adhesive material, specimen and end tab surface characteristics, and lack of parallelism of specimen surfaces.

13. Calculations

13.1 *Flatwise Tensile Strength:*

13.1.1 Calculate the ultimate flatwise "through-thickness" tensile strength using Eq 1 and report the results to three significant digits. If the through-thickness tensile modulus is to be calculated, determine the through-thickness tensile stress at each required data point using Eq 2.

$$F^{tu} = P_{max}/A \quad (1)$$

$$\sigma_i = P_i/A \quad (2)$$

where:

F^{tu} = ultimate flatwise tensile strength, Pa [psi],
P_{max} = maximum force prior to failure, N [lbf],
P_i = force at i-th data point, N [lbf],
A = cross-sectional area at test section in through-thickness direction, m^2 [$in.^2$]. and
σ_i = tensile stress at the i-th data point, Pa [psi].

13.2 *Through-Thickness Modulus of Elasticity:*

13.2.1 If strain data are available, calculate the through-thickness modulus of elasticity using Eq 3. If data are not available at the exact strain range end points, use the closest available data point. Report the through-thickness modulus of elasticity to three significant digits. Also report the strain range used in the calculation.

$$E^{chord} = \Delta\sigma/\Delta\varepsilon \quad (3)$$

where:

E^{chord} = through-thickness modulus of elasticity, Pa [psi],
$\Delta\sigma$ = difference in applied normal stress between the two strain points, Pa [psi], and
$\Delta\varepsilon$ = difference in the average normal strain between the closest available two strain points at 500 με and 1500 με. For materials that fail at strains below 1500 με, a strain range of 25 to 50 % of the ultimate strain shall be used.

13.3 *Statistics*—For each series of tests calculate the average value, standard deviation and coefficient of variation (in percent) for each property determined:

$$\bar{x} = \left(\sum_{i=1}^{n} x_i\right)/n \quad (4)$$

$$S_{n-1} = \sqrt{\left(\sum_{i=1}^{n} x_i^2 - n\bar{x}^2\right)/(n-1)} \quad (5)$$

$$CV = 100 \times S_{n-1}/\bar{x} \quad (6)$$

where:

$\bar{x}$ = sample mean (average),
S_{n-1} = sample standard deviation,
CV = sample coefficient of variation, in percent,
n = number of specimens, and
x_i = measured or derived property.

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 of panel fabrication parameters, shall be the responsibility of the requester):

NOTE 5—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 date(s) and location(s) of the test.
14.1.3 The name(s) of the test operator(s).
14.1.4 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.
14.1.5 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer's lot or batch 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, prepreg matrix content, and prepreg volatiles content.

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

14.1.7 Ply orientation stacking sequence of the laminate.

14.1.8 If requested, report density, reinforcement volume fraction, and void content test methods, specimen sampling method and geometries, test parameters, and test data.

14.1.9 Average ply thickness of the material.

14.1.10 Results of any non-destructive evaluation tests.

14.1.11 Method of preparing the test specimens, including specimen labeling scheme and method, specimen geometry, sampling method, coupon cutting method, identification of tab geometry, tab material, and tab adhesive used.

14.1.12 Calibration dates and methods for all measurement and test equipment.

14.1.13 Type of test machine, alignment data, and data acquisition sampling rate and equipment type.

14.1.14 Measured dimensions for each test specimen.

14.1.15 Conditioning parameters and results, use of travelers and traveler geometry, and the procedure used if other than that specified in the test method.

14.1.16 Relative humidity and temperature of the testing laboratory.

14.1.17 Environment of the test machine environmental chamber (if used) and soak time at environment.

14.1.18 Number of specimens tested.

14.1.19 Speed of testing.

14.1.20 Transducer placement on the specimen, transducer type, and calibration data for each transducer used.

14.1.21 The strain gage type, resistance, size, gage factor, temperature compensation method, transverse sensitivity, lead-wire resistance, and any correction factors employed.

14.1.22 Tabulated data of force versus displacement and force-displacement curves for each specimen.

14.1.23 Tabulated data of stress versus strain and stress-strain curves for each modulus specimen.

14.1.24 Percent bending results for each specimen so evaluated.

14.1.25 Individual strengths and average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.26 Individual values of modulus of elasticity and the average value, standard deviation, and coefficient of variation (in percent) for the population if modulus measurement is required.

14.1.27 Strain range used for chord modulus determination.

14.1.28 If another definition of modulus of elasticity is used in addition to chord modulus, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

14.1.29 Failure mode and location of failure for each specimen using the notation in 11.10.

15. Precision and Bias

15.1 *Precision*—The precision of this test method is based on an interlaboratory study (ILS) of ASTM D7291, Standard Test Method for Through Thickness Flatwise Tensile Strength of a Fiber Reinforced Polymer Matrix Composite Material, conducted in 2009. Two different laminate types were tested. One was a graphite/epoxy (IM7/977-3) tape laminate with a stacking sequence of [45/-45/0/45/-45/90/45/-45]s and the other was a glass/epoxy (E7781/8552) fabric laminate with a total thickness of 3.3 mm (0.13 in.). Seven laboratories measured the flatwise tensile strength of the two different materials. All the specimens were prepared and bonded at one location to reduce variability in specimen preparation and bonding procedures. Each of the seven laboratories received randomized samples for testing. All tests were performed at ambient laboratory conditions. Every "test result" represents an individual determination. Each laboratory was asked to submit six replicate test results, from a single operator, for each material type. The average results for each laboratory and each material are listed in Table 1 and Table 2. Except for the limited number of materials tested, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D30-1008[4].

15.1.1 The results in Table 1 and Table 2 indicate that the glass/epoxy material exhibited a higher percentage of failures in the SA and AB "invalid" failure modes compared to the graphite/epoxy material. The specific nature of the glass/epoxy material with its lower fiber-matrix interface strength and higher thermal expansion coefficient might be responsible for

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D30-1008. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Flatwise Tensile Strength (MPa) Results for Graphite/Epoxy

Lab	Average Tension Strength (MPa) — Valid Failures	CV	Specimens Tested	Valid Failures	Specimens Failing in SA Failure Mode	Specimens Failing in AB Failure Mode
1	42.9	7.8%	5	4	1	0
2	38.7	20.4%	6	6	0	0
3	43.1	6.4%	6	5	0	1
4	45.7	4.4%	6	3	2	1
5	45.8	2.7%	6	5	1	0
6	43.7	2.9%	6	5	1	0
7	42.1	9.4%	6	6	0	0
		Total:	41	34	5	2
			Percent Failures:	82.9%	12.2%	4.9%

TABLE 2 Flatwise Tensile Strength (MPa) Results Summary for Glass/Epoxy

Lab	Average Tension Strength (MPa) — Valid Failures	CV	Specimens Tested	Valid Failures	Specimens Failing in SA Failure Mode	Specimens Failing in AB Failure Mode
1	36.7	...	6	1	5	0
2	39.2	...	6	1	1	4
3	43.6	10.8%	6	3	2	1
4	44.8	7.0%	6	6	0	0
5	47.6	...	6	1	5	0
6	...	...	5	0	0	5
7	44.0	4.7%	6	6	0	0
		Total:	41	18	13	10
			Percent Failures:	43.9%	31.7%	24.4%

this difference. Performance of statistical F-test and t-tests to compare the "valid" glass/epoxy data and the data with the SA failure mode indicated equivalency of mean and variance for the two data sets. The AB failure mode data set was found to be statistically not equivalent to the valid data set. Based upon these findings, a second precision and bias analysis was performed, combining the valid failure mode and SA failure mode data sets. The results of this analysis are listed in Table 3.

15.1.2 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material; *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

15.1.2.1 Repeatability limits for the valid failures for the graphite/epoxy and the glass/epoxy materials are listed in Table 3. Also included in Table 3 are the repeatability limits for the glass/epoxy material by including specimens that failed in (i) the valid failure mode and (ii) the SA failure mode.

15.1.3 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the *R* value for that material; *R* is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

15.1.3.1 Reproducibility limits for the valid failures for the graphite/epoxy and the glass/epoxy materials are listed in Table 3. Also included in Table 3 are the reproducibility limits for the glass/epoxy material by including specimens that failed in (i) the valid failure mode and (ii) the SA failure mode.

15.1.4 The above terms ("repeatability limit" and "reproducibility limit") are used as specified in Practice E177.

15.1.5 Any judgment in accordance with statements 15.1.2 and 15.1.3 would normally have an approximate 95% probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95% probability limit would imply. Consider the repeatability limit as a general guide, and the associated probability of 95% as only a rough indicator of what can be expected.

15.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

15.3 The precision statement was determined through the statistical examination of 52 and 65 analytical results (out of a total of 82), from seven laboratories, on two materials. The materials were identified as:
Material A: Graphite/epoxy, 2.54 cm × 2.54 cm
Material B: Glass/epoxy, 2.54 cm × 2.54 cm

16. Keywords

16.1 composite materials; flatwise tension strength; interlaminar tensile strength; out-of-plane tension; through-thickness modulus of elasticity.; through-thickness strength

TABLE 3 Through-Thickness Flatwise Tensile Strength (MPa)

Material	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Graphite/Epoxy Valid Failures	43.1484	4.0935	4.4340	11.4618	12.4151
Glass/Epoxy Valid Failures	42.6739	3.4702	5.0908	9.7167	14.2542
Glass/Epoxy Valid and SA Failures	44.4783	3.6349	3.6718	10.1777	10.2811

[A]The average of the laboratories' calculated averages.