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Standard Test Methods for Properties of Continuous Filament Carbon and Graphite Fiber Tows¹

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This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the preparation and tensile testing of resin-impregnated and consolidated test specimens made from continuous filament carbon and graphite yarns, rovings, and tows to determine their tensile properties.

1.2 These test methods also cover the determination of the density and mass per unit length of the yarn, roving, or tow to provide supplementary data for tensile property calculation.

1.3 These test methods include a procedure for sizing removal to provide the preferred desized fiber samples for density measurement. This procedure may also be used to determine the weight percent sizing.

1.4 These test methods include a procedure for determining the weight percent moisture adsorption of carbon or graphite fiber.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ These test methods are under the jurisdiction of ASTM Committee D30 on Composite Materials and are the direct responsibility of Subcommittee D30.03 on Constituent/Precursor Properties.

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2. Referenced Documents

2.1 ASTM Standards:²

D70 Test Method for Specific Gravity and Density of Semi-Solid Asphalt Binder (Pycnometer Method)

D1193 Specification for Reagent Water

D3800 Test Method for Density of High-Modulus Fibers

D5550 Test Method for Specific Gravity of Soil Solids by Gas Pycnometer

E4 Practices for Force Calibration and Verification of Testing Machines

E83 Practice for Verification and Classification of Extensometer Systems

2.2 Other Document:

CRC Handbook of Chemistry and Physics³

3. Terminology

3.1 Definitions:

3.1.1 *sizing, n*—a generic term for compounds which, when applied to yarn or fabric, form a more or less continuous solid film around the yarn and individual fibers.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *desized fiber, n*—fiber which has had a sizing removed from it.

3.2.2 *fiber, n*—continuous filament carbon or graphite yarn, roving, or tow.

3.2.3 *sized fiber, n*—a fiber with a sizing applied to it.

3.2.4 *unsized fiber, n*—fiber which has never had a sizing applied to it.

3.3 Symbols:

3.3.1 *A*—Unit conversion factor for tensile strength

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 CRC Press Inc., Boca Raton, Ann Arbor, London, Tokyo, 73rd Edition, 1992–1993.

- 3.3.2 B —Unit conversion factor for tensile modulus
- 3.3.3 E —fiber chord modulus
- 3.3.4 ϵ_l —Lower strain limit
- 3.3.5 ϵ_u —Upper strain limit
- 3.3.6 k_c —correction factor for density of sizing
- 3.3.7 L —specimen length
- 3.3.8 MUL —the mass per unit length of the sized fiber
- 3.3.9 MUL_I —the mass per unit length of the impregnated and consolidated fiber
- 3.3.10 P —maximum load
- 3.3.11 P_l —tensile load at lower strain limit
- 3.3.12 P_u —tensile load at upper strain limit
- 3.3.13 ρ_f —density of the fiber
- 3.3.14 ρ_{sf} —density of the fiber with sizing
- 3.3.15 RC —weight percent of resin (resin content)
- 3.3.16 W_I —specimen mass

4. Summary of Test Methods

4.1 These test methods include procedures for determining the tensile strength and modulus of a resin-impregnated and consolidated carbon fiber tow. Also included are procedures to measure the mass per unit length and density of the carbon fiber and the resin content of the resin-impregnated and consolidated specimens.

4.2 MUL —The MUL of the fiber is determined by dividing the mass of a sample of sized fiber by its length.

4.3 $Density$ —The density of the fiber is determined using Archimedes' method or a pycnometer method. The recommended specimen is desized or unsized fiber. The ideal immersion fluid is one that completely wets the specimen and provides minimum toxicity or environmental hazard.

4.4 $Resin Content$ —The resin content (weight percent) of the resin-impregnated and consolidated fiber is determined by comparing the mass per unit length of the impregnated and consolidated specimen to the fiber mass per unit length.

4.5 $Tensile Properties$ —The tensile strength and tensile chord modulus of the fiber are determined by the tensile loading to failure of the resin-impregnated and consolidated fiber. The chord modulus is determined between defined strain limits. The purpose of the impregnating resin is to provide the fiber, when consolidated, with sufficient mechanical strength to produce an easily handled test specimen capable of sustaining uniform loading of the individual filaments in the specimen. The resin shall be compatible with the fiber and any sizing applied to it. The strain capability of the consolidated resin shall be at least twice the strain capability of the fiber.

5. Significance and Use

5.1 The properties determined by these test methods are of value in material specifications, qualifications, data base generation, certification, research, and development.

5.2 These test methods are intended for the testing of fibers that have been specifically developed for use as reinforcing

agents in advanced composite structures. The test results of an impregnated and consolidated fiber should be representative of the strength and modulus that are available in the material when used as intended. The performance of fibers in different resin systems can vary significantly so that correlations between results using these test methods and composite testing may not always be obtained.

5.3 The reproducibility of test results is dependent upon precise control over all test conditions. Resin type, content and distribution, curing process, filament alignment, gripping in the testing machine, and alignment in the testing machine are of special importance.

5.4 The measured strengths of fibers are not unique quantities and test results are strongly dependent on the test methods used. Therefore the test method described here will not necessarily give the same mean strengths or standard deviations as those obtained from single filaments, dry fibers, composite laminas, or composite laminates.

6. Apparatus

6.1 Three sets of apparatus are required. One set is for resin impregnation of the fiber. A second set is for curing the resin-impregnated specimens. A third set is for tensile testing the resin-impregnated and consolidated specimens. Optional apparatus may be used for applying end tabs to the specimens and removing sizing.

6.1.1 $Resin Impregnation$ —The goal of the resin-impregnation apparatus is to apply and uniformly impregnate resin into the fiber. This is normally achieved by dipping the fiber into the resin and then working the wet fiber over rolls or through a die, or both. While automated apparatuses are preferred for consistency, any apparatus which achieves uniform impregnation of 35 % to 60 % resin by weight and does not damage the fiber is acceptable.

6.1.2 $Consolidation$ —An apparatus to hold the impregnated specimens under tension during consolidation is required.

6.1.3 $Optional End Tabs$ —An apparatus to cast resin end tabs on specimens may be used. Apparatuses to apply and align other forms of end tabs such as bonded on cardboard or metal tabs may also be used.

6.1.4 $Testing$ —A tensile testing machine and recorder meeting the requirements of Practices **E4** at the maximum expected test load are required. The load recording device shall be coordinated with the extensometer and strain recorder to assure that the corresponding load and elongation of the specimen are recorded at essentially the same time. The testing machine shall also have the following features:

6.1.4.1 $Grips$ —Grips suitable for loading the tabbed or untabbed specimen without damaging it are required. For resin tabbed specimens a custom grip is generally required. For untabbed or cardboard tabbed specimens pneumatic or hydraulically powered grips are typically used. Different grip pressure settings may be required for different fibers.

6.1.4.2 $Jaws$ —Jaws compatible with the grips and capable of holding a specimen without damaging it are required. For untabbed specimens, flat jaws with rubber or other compliant materials bonded to the face are generally used. Sandpaper may also be placed on the grips to reduce slippage. For

cardboard tabbed specimens serrated jaws are generally used. Jaws should be inspected regularly and cleaned or repaired as required.

6.1.4.3 *Extensometer and Recorder*—An extensometer and recording device in accordance with the requirements of Practice E83 Class B-2 are required. The extensometer and recorder shall be coordinated with the tensile testing machine so that the corresponding load and elongation of the specimen are recorded at essentially the same time.

6.1.4.4 *Balance, Analytical*—Accuracy of ± 0.0002 g for mass per unit length, density, and resin or sizing content determinations.

6.1.4.5 *Balance, Laboratory*—Accuracy of ± 0.1 g for measuring components of resin.

6.1.4.6 *Length Measuring Devices*—Devices to measure the length of impregnated and consolidated specimens and dry fiber to ± 2 mm accuracy are required.

6.1.4.7 *Density Measuring Device*—See Test Methods D70, D3800, or D5550.

6.1.4.8 *Forced Air Oven*—A forced air oven of sufficient sizing and temperature capabilities to cure the impregnated fiber on the curing device. The temperature shall be controlled to ± 10 °C.

7. Reagents and Materials

7.1 *Reagent Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D1193.

7.2 *Resin*.

7.3 *Commercial Grade Solvent*, (optional) for resin dilution.

7.4 *Hardener or Catalyst*.

7.5 *Surfactant* (optional).

8. Test Specimens

8.1 *Mass per Unit Length Specimen*—The specimen to measure mass per unit length is an 1 m minimum length of fiber in the form in which it is intended to be used. Test one specimen per sample. Coil the specimen into a usable form for testing. Care is required to not damage and lose filaments from the specimen.

8.2 *Density Specimen*—Density specimens may be sized, unsized, or desized material. Unsized or desized samples are preferred. Test one specimen per sample. An 1 m minimum length is required for Test Method D3800. For methods in Test Methods D70 and D5550, a suitable volume to fill the container is recommended. Coil the specimen into a usable form for testing. Care is required to not damage and lose filaments from the specimen.

8.3 *Tensile Test Specimen*—The tensile specimen shall be a tabbed or untabbed resin-impregnated and consolidated fiber. Tabbed specimens shall have a $150 \text{ mm} \pm 5 \text{ mm}$ gauge length between the tabs. Untabbed specimens shall be of sufficient length to allow a $150 \text{ mm} \pm 5 \text{ mm}$ gauge length between the grips when they are tested. Samples with 3000 filaments or less may be tested using specimens of more than one fiber bundle to facilitate handling. Combine the bundles before resin im-

pregnation and count the test results as one specimen. Test a minimum of four test specimens per sample. Separate test specimens for modulus and strength determination are permitted; however, four specimens for each test are required. Specimens that are fuzzy, curved, not uniform in cross section, have broken filaments, resin lumps, or other observable defects should be rejected unless they are representative of the material being tested.

9. Conditioning

9.1 Fibers that adsorb less than 0.5 % moisture by weight at $23^\circ\text{C} \pm 2^\circ\text{C}$ and 90 % or greater relative humidity require no conditioning before testing. Conduct mass per unit length, density, and tensile testing for these materials at $23^\circ\text{C} \pm 7^\circ\text{C}$ and $50\% \pm 20\%$ relative humidity unless other conditions are the variables of interest.

9.2 Fibers that adsorb more than 0.5 % moisture by weight at $23^\circ\text{C} \pm 2^\circ\text{C}$ and 90 % or greater relative humidity require conditioning before testing. Condition fibers at $23^\circ\text{C} \pm 2^\circ\text{C}$ and $50\% \pm 10\%$ relative humidity for a minimum of 24 h. Conduct mass per unit length and density testing at $23^\circ\text{C} \pm 2^\circ\text{C}$ and $50\% \pm 10\%$ relative humidity unless other conditions are the variables of interest. Conduct tensile testing at $23^\circ\text{C} \pm 7^\circ\text{C}$ and $50\% \pm 10\%$ relative humidity unless other conditions are the variables of interest.

9.3 To determine moisture adsorption, dry a minimum of five mass per unit-type specimens at $120^\circ\text{C} \pm 5^\circ\text{C}$ for 24 h in a circulating air oven. Cool the specimens in a desiccator. Remove the specimens from desiccator one at a time and immediately weigh them. Place the specimens in a humidity chamber and maintain at $23^\circ\text{C} \pm 2^\circ\text{C}$ and 90 % or greater relative humidity for 24 h. Remove the specimens from the humidity chamber one at a time and weigh immediately.

9.4 If all specimens are conditioned for 24 h and mass per unit length and density testing are performed at $23^\circ\text{C} \pm 2^\circ\text{C}$ and $50\% \pm 10\%$ relative humidity, then no testing for moisture adsorption is required.

9.5 Moisture adsorption testing is only required once annually for a standard product.

10. Specimen Preparation

10.1 *Mass per Unit Length*—No preparation is required. The sample is taken from a package of material as it is intended to be used.

10.2 *Density*—The preferred density specimen is a desized or unsized fiber. This eliminates any need to correct for the density of the sizing. The sizing may be removed using solvent extraction, pyrolysis, or other means. An example method is described in Appendix X1. An unsized sample of a sized fiber would have to be collected during the production of the fiber and is therefore not recommended.

10.3 *Tensile Test*—The tensile test specimen must be impregnated with resin and consolidated before testing.

10.3.1 *Resin Preparation*—Any resin that meets the requirements of 4.5 may be used. The resin when combined with the fiber shall produce a composite (reinforcement/matrix) failure.

A resin generally found satisfactory is a combination of bisphenol A (or bisphenol F) epoxy and diethyltoluene diamine in the weight ratio of 3.9:1. A solvent that lowers the viscosity of a resin mixture or softens the sizing, or both, may be selected for use with the resin. The amount and type of solvent used will vary with the product and apparatus used for impregnation. Prepare and store the resin in accordance with the manufacturer's instructions.

10.3.2 *Fiber Impregnation*—The resin shall be maintained within a suitable viscosity or temperature range throughout the impregnation process that assures reproducible specimen constituency and quality. An automated impregnation device is recommended but not required. The objective of impregnation is a uniform, well-collimated strand with adequate resin content, typically 35 % to 60 % by weight.

10.3.2.1 Wind or attach the impregnated fiber to a rack or other device to hold it under tension to avoid fiber waviness but not to cause fiber breakage during consolidation.

10.3.2.2 Check the resin content of impregnated and consolidated fiber to ensure adequate resin pickup. Determine the mass of a known length of impregnated and consolidated fiber. Compute the resin content using the mass per unit length of the fiber as a reference.

10.3.2.3 *Tabbing*—End tabs may be applied to the impregnated and consolidated specimens but are not required. End tab material must adequately grip the specimen without slipping. Heavy paper or poster board have been used successfully as tabs. The distance between tabs shall be 150 mm ± 5 mm.

11. Procedure

11.1 *Mass per Unit Length*—Take the mass per unit length specimen from the fiber in the same form as it is intended to be used. Test fiber with sizing on the fiber. Test one specimen per sample.

11.1.1 Using a calibrated measuring device measure a minimum of an 1 m (±2 mm) length of fiber. Hold the fiber under light hand tension while measuring and cutting it. Coil the fiber onto a usable form taking care not to damage or lose filaments.

11.1.2 Determine the mass of the specimen using an analytical balance.

11.1.3 *Retests*—Reject any specimen which has an obvious flaw or deviation from the requirements of these test methods. Prepare a new or spare specimen from the same sample and test to replace any specimens eliminated in accordance with this paragraph.

11.1.3.1 Discard test results for any condition that compromises the integrity of the test. These conditions must be documented.

11.1.3.2 Retest any suspect single specimen or sample. Retesting of specimens requires twice the number of specimens that were suspect. Retesting of a sample requires twice the number of specimens that were tested in the suspect sample. The average result of the retested specimens shall replace the original value. However, all values must be recorded.

11.2 *Density*—Test density in accordance with Method A of Test Methods **D3800** (for Archimedes' method) or **D70/D5550** (for a pycnometer method) with modifications. The preferred

density sample is unsized or desized fiber. Test one specimen per sample. Coil the fiber into a suitable form taking care not to damage or lose filaments.

11.2.1 *Retests*—Any specimen that has an obvious flaw or deviation from the requirements of these test methods shall be rejected. Prepare a new or spare specimen from the same sample and test to replace any specimens eliminated in accordance with this paragraph.

11.2.1.1 Discard test results for any condition that compromises the integrity of the test. These conditions must be documented.

11.2.1.2 Retest any suspect single specimen or sample. Retesting of specimens requires twice the number of specimens that were suspect. Retesting of a sample requires twice the number of specimens that were tested in the suspect sample. The average result of the retested specimens shall replace the original value. However, all values must be recorded.

11.3 *Tensile Test*—Test the impregnated and consolidated fiber in a calibrated tensile testing machine using a calibrated extensometer. Test a minimum of four specimens for strength and modulus. These may be the same four specimens or two sets of four specimens. (If the extensometer causes premature tensile failure it may be necessary to test four additional specimens without the extensometer for strength only.)

11.3.1 Install the appropriate grips and jaws into the tensile testing machine. For untabbed specimens set the distance between the grips to 150 mm ± 5 mm.

11.3.2 Set the speed of the testing machine. The maximum acceptable speed is 250 mm/min. The maximum practical speed may be limited by the speed of data sampling or recording equipment.

11.3.3 Select the appropriate load range of the tensile testing machine.

11.3.4 Set the speed of any recording or sampling equipment as required.

11.3.5 Place the specimen in the jaws and carefully align it so that it is centered in the jaws and aligned with the loading axis. Close the grips assuring that the specimen remains aligned.

11.3.6 Preload the specimen to the appropriate load in accordance with **Table 1**. Determine the load using the expected modulus and cross-sectional area of the fiber.

11.3.7 Attach the extensometer if modulus is to be measured. Take particular care not to damage or cut filaments.

11.3.8 Start the recording or sampling equipment and the tensile testing machine.

11.3.9 When the maximum strain required for modulus calculation has been exceeded, the extensometer may be removed. This may be done while the test is in progress or after stopping the testing machine. Take particular care not to damage or cut filaments.

TABLE 1 Maximum Preloads

| Typical Product | Maximum Preload |
|---------------------------------|-----------------|
| Strain at failure (microstrain) | (microstrain) |
| $\epsilon \geq 12\ 000$ | 200 |
| $6000 \leq \epsilon < 12\ 000$ | 100 |
| $\epsilon < 6000$ | 100 |

11.3.10 Test the specimen to failure. Observe the failure of specimen.

11.3.11 Record the failure load and the load at the strains in accordance with Table 2 for use in calculating the chord modulus.

11.3.12 Retests—Any specimen that has an obvious flaw or deviation from the requirements of these test methods shall be rejected. Prepare a new or spare specimen from the same sample and test to replace any specimens eliminated in accordance with this paragraph.

11.3.12.1 Discard test results for any condition that compromises the integrity of the test. These conditions must be documented. Examples of these conditions include slippage of the specimen or extensometer, breaks in the tabs or grips, breaks at the extensometer attachment points, and abnormal breaks which may be due to poor resin impregnation (see 11.4).

11.3.12.2 Retest any suspect single specimen or sample. Retesting of specimens requires twice the number of specimens that were suspect. Retesting of a sample requires twice the number of specimens that were tested in the suspect sample. The average result of the retested specimens shall replace the original value. However, all values must be recorded.

11.4 Failure Mode—Record typical mode and location of failure of individual specimens. Typical failure mode and location may be grouped for the fiber. Choose, if possible, a standard description using the three-part failure mode code that is shown in Fig. 1.

11.5 Grip/Tab Failures—Reexamine the means of load introduction into the material if a significant fraction of failures in a sample population occur within one specimen width of the tab or grip. Factors considered should include the tab alignment, tab material, tab adhesive, grip type, grip pressure, and grip alignment.

12. Calculations

12.1 Mass per Unit Length, MUL—Calculate the mass per unit length of the fiber using the following equation.

$$MUL = W_1/L \tag{1}$$

where:

- MUL = mass per unit length, g/m;
- W₁ = mass of the specimen, g; and
- L = length of the specimen, m.

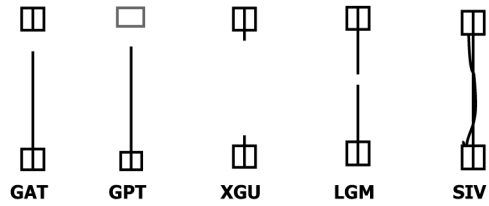
12.2 Density—Calculate the density of the fiber using following sets of equations.

12.2.1 Desized or Unsized Fiber—Desized or unsized fiber is preferred over sized fiber for fiber density measurement. Use equations found in Test Methods D3800, D70, or D5550.

12.2.2 Sized Fiber—Determine the fiber density as follows:

TABLE 2 Chord Modulus Strain Limits

| Typical Product | Chord Modulus | |
|---------------------------------|-----------------------|-------|
| | lower | upper |
| Strain at failure (microstrain) | strains (microstrain) | |
| ε ≥ 12 000 | 1000 | 6000 |
| 6000 ≤ ε < 12 000 | 1000 | 3000 |
| ε < 6000 | 500 | 1500 |



| First Character | | Second Character | | Third Character | |
|--------------------------------|------|--------------------|------|------------------|------|
| Failure Type | Code | Failure Area | Code | Failure Location | Code |
| Grip/Tab | G | Inside Grip/Tab | I | Bottom | B |
| Lateral | L | At Grip/Tab | A | Top | T |
| Long Splitting (fiber pullout) | S | <1 W from grip/tab | W | Middle | M |
| Explosive | X | Gage | G | Various | V |
| Other | O | Tab Pullout | P | Unknown | U |
| | | Various | V | | |
| | | Unknown | U | | |

FIG. 1 Three-Part Failure Mode Code

$$\rho_f = \rho_{sf} - k_c \tag{2}$$

where:

- ρ_f = fiber density, g/cm³;
- ρ_{sf} = density of fiber + sizing, g; and
- k_c = correction factor (if required). (k_c is determined by means of 10 repeated tests on sized and desized or unsized samples to correlate sized fiber results. k_c may be a function of sizing level and sizing type. A correction factor is not required if the test differences are less than 1.0 % of the preferred technique result.)

12.3 Tensile Test:

12.3.1 Resin Content—Calculate the resin content of the impregnated and consolidated fiber using the following equation:

$$RC = 100(1 - (MUL/MUL_i)) \tag{3}$$

where:

- RC = resin content, weight %;
- MUL = mass per unit length of fiber, g/m; and
- MUL_i = mass per unit length of the impregnated and consolidated fiber, g/m.

12.3.2 Tensile Strength—Calculate the tensile strength of the fiber using the following equation:

$$\text{Tensile Strength (MPa)} = A \times P \times \rho_f / MUL \tag{4}$$

where:

- P = maximum load measured in tensile test, N (lb);
- ρ_f = fiber density, g/cm³;
- MUL = fiber mass per unit length, g/m; and
- A = unit conversion factor (1 if load in N).

12.3.3 Tensile Modulus—Calculate the tensile chord modulus of the fiber between the strain values in accordance with 11.3.11 using the following equation:

$$E = B \times (P_u - P_l) \times \rho_f / ((\epsilon_u - \epsilon_l) \times MUL) \tag{5}$$

where:

- E = fiber chord modulus, GPa;
- P_u = tensile load at upper strain limit, N (lb);
- P_l = tensile load at lower strain limit, N (lb);