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Standard Test Methods for Properties of Continuous Filament Carbon and Graphite Fiber Tows[1]

This standard is issued under the fixed designation D4018; 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.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D70 Test Method for Density of Semi-Solid Bituminous Materials (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 Verification of Testing Machines

E83 Practice for Verification and Classification of Extensometer Systems

2.2 *Other Document:*

CRC Handbook of Chemistry and Physics[3]

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

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 size

3.3.7 *L*—specimen length

3.3.8 *MUL*—the mass per unit length of the sized fiber

3.3.9 MUL_f—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

[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] 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.

3.3.12 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_1—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 size 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 size 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 size 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 a 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. A 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 ± 5 mm gage length between the tabs. Untabbed specimens shall be of sufficient length to allow a 150 ± 5 mm gage 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 impregnation 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 ± 2°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 ± 7°C and 50 ± 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 ± 2°C and 90 % or greater relative humidity require conditioning before testing. Condition fibers at 23 ± 2°C and 50 ± 10 % relative humidity for a minimum of 24 h. Conduct mass per unit length and density testing at 23 ± 2°C and 50 ± 10 % relative humidity unless other conditions are the variables of interest. Conduct tensile testing at 23 ± 7°C and 50 ± 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 ± 5°C for 24 h in a circulating air oven. Cool samples in a desiccator. Remove samples from desiccator one at a time and immediately weigh them. Place samples in a humidity chamber and maintain at 23 ± 2°C and 90 % or greater relative humidity for 24 h. Remove samples 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 ± 2°C and 50 ± 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