Designation: D 4018 – 99

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

This standard is issued under the fixed designation D 4018; 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 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 the standard. The values in parentheses are for information only.

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:*

D 70 Test Method for Specific Gravity/Density of Semi-solid Bituminous Materials/Asphalt Cement/Soft Tar Pitches by Pycnometer Test[2]

D 123 Terminology Relating to Textiles[3]

D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals[4]

D 1193 Specification for Reagent Water[5]

D 3800 Test Method for Density of High-Modulus Fibers[6]

D 3878 Terminology of High-Modulus Reinforcing Fibers and Their Composites[5]

D 5550 Test Methods for Soil Solids—Specific Gravity by Gas Pycnometer Test[7]

E 4 Practices for Force Verification of Testing Machines[8]

E 6 Terminology Relating to Methods of Mechanical Testing[6]

E 83 Practice for Verification and Classification of Extensometers[6]

E 100 Specification for ASTM Hydrometers[9]

2.2 *Other Document:*

CRC Handbook of Chemistry and Physics[10]

3. Terminology

3.1 *Definition:*

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

3.3.12 P_u—tensile load at upper strain limit

3.3.13 ρ_f—density of the fiber

[1] These test methods are under the jurisdiction of ASTM Committee D-30 on Composite Materials and are the direct responsibility of Subcommittee D30.03 on Constitutuent/Precursor Properties.

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[2] *Annual Book of ASTM Standards*, Vol 04.03

[3] *Annual Book of ASTM Standards*, Vol 07.01.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

[6] *Annual Book of ASTM Standards*, Vol 15.03.

[7] *Annual Book of ASTM Standards*, Vol 04.09.

[8] *Annual Book of ASTM Standards*, Vol 03.01.

[9] *Annual Book of ASTM Standards*, Vol 14.03.

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

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 E 4 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 E 83 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 D 70, D 3800, or D 5550.

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 D 1193.

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 D 3800. For methods in Test Methods D 70 and D 5550, 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-mm gage length between the tabs. Untabbed specimens shall be of sufficient length to allow a 150-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 Condition fibers that adsorb more than 0.5 % moisture by weight (at 23 ± 2°C and 90 % or greater relative humidity) for a minimum of 24 h at 23 ± 2°C and 50 ± 10 % relative humidity. 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 ± 20 % 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 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 during consolidation.

10.3.2.2 Check the consolidated impregnated fiber resin content at least daily 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.

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 size with the size on the fiber. Test one specimen per sample.

11.1.1 Using a calibrated measuring device measure a minimum of a 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 D 3800 (for Archimedes' method) or D 70/D 5550 (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.

11.3.2 Set the speed of the testing machine. The maximum acceptable speed is 250 mm/min (10 in./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.

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 atypical 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

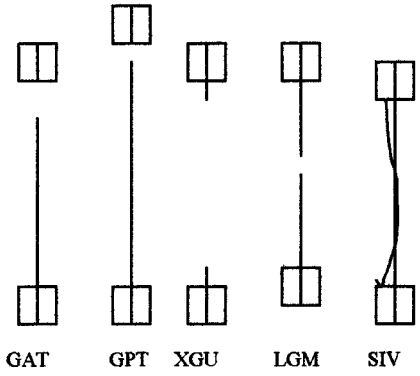
11.5 *Grip/Tab Failures*—Reexamine the means of load introduction into the material if a significant fraction of failures

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

TABLE 2 Chord Modulus Strain Limits

Typical Product	Chord Modulus	
Strain at failure (microstrain)	strains (microstrain)	
	lower	upper
$\epsilon \geq 12\,000$	1000	6000
$6000 \leq \epsilon < 12\,000$	1000	3000
$\epsilon < 6000$	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 pulloout)	S	<1W from grip/tab	W	Middle	M
		Tab Pullout	P	Various	V
EXplosive	X	Various	V	Unknown	U
Other	O	Unknown	U		

FIG. 1 Three-Part Failure Mode Code

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;
W_1 = 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 (Preferred)*—Use equations found in Test Methods D 3800, D 70, or D 5550.

12.2.2 *Sized Fiber*—Determine the fiber density as follows:

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

where:
ρ_f = fiber density, g/cm^3;
ρ_{sf} = density of fiber + sizing, g; and
k_c = correction factor (if required). (k_c is determined by means of repeated tests on sized and desized or unsized samples to correlate sized fiber results with the preferred technique. k_c may be a function of size level and size 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/cm^3;
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);
ρ_f = fiber density, g/cm^3;
ϵ_u = upper strain limit, microstrain;
ϵ_l = lower strain limit, microstrain;
MUL = fiber mass unit length, g/m; and
B = unit conversion factor (10^{-3} if load is in N).

13. Report

13.1 Report the following information:

13.1.1 *MUL Report*:

13.1.1.1 Complete identification of the samples tested to assure traceability of test results,

13.1.1.2 Date of test,

13.1.1.3 Test facility,

13.1.1.4 Identification of the individual performing test,

13.1.1.5 *MUL* of the specimens tested, and

13.1.1.6 Any deviations from these test methods.

13.1.2 *Density Report*:

13.1.2.1 Complete identification of the samples tested to assure traceability of test results,

13.1.2.2 Date of test,

13.1.2.3 Test facility,

13.1.2.4 Identification of the individual performing test,

13.1.2.5 Immersion fluid and degassing technique if appropriate,

13.1.2.6 Density of the specimens tested, and

13.1.2.7 Any deviations from these test methods.

13.1.3 *Tensile Test Report*:

13.1.3.1 Complete identification of the samples tested to assure traceability of test results,

13.1.3.2 Date of test,

13.1.3.3 Test facility,

13.1.3.4 Identification of the individual performing test,

13.1.3.5 Impregnation apparatus, resin, and consolidation or cure cycle used,

13.1.3.6 Tensile testing apparatus,

13.1.3.7 Individual and average tensile strength and tensile chord modulus of the specimens tested, and

13.1.3.8 Reason for specimen rejection and atypical specimen failure modes, as well as a typical failure mode.

13.1.3.9 Any deviations from these test methods.

14. Precision and Bias

14.1 *Precision*—The precision of the procedures for measuring *MUL*, density, tensile strength and tensile chord modulus, and weight percent size is being determined.

14.2 *Bias*—Since there is no accepted reference material suitable for the procedure for measuring tensile strength and tensile chord modulus, bias has not been determined. The procedures for measuring *MUL* and weight percent size have no bias because the values of *MUL* and weight percent size are defined only in terms of these test methods.

APPENDIX

(Nonmandatory Information)

X1. REMOVING THE SIZE FROM SIZED FIBER SPECIMENS AND DETERMINING THE PERCENT SIZING CONTENT

X1.1 Scope

X1.1.1 This test method describes a procedure for removing the size from sized fiber specimens and determining the percent sizing content.

X1.2 Summary of Test Method

X1.2.1 Sizing is completely pyrolyzed from the fiber by heating the specimen in a furnace under inert atmosphere. A typical temperature and time to accomplish this are 450°C for 15 min. However, a particular set of time and temperature requirements must be determined for each size and fiber under consideration.

X1.3 Significance and Use

X1.3.1 The result of this test method, a desized fiber specimen, is the preferred specimen for density measurement.

X1.3.2 This test method may be used to determine the weight percent sizing of the fiber specimen.

X1.4 Apparatus

X1.4.1 *Electric Furnace*—An electric furnace with adequate exhaust ventilation and capable of the temperature required to pyrolyze the sizing under consideration. The furnace is supplied with nitrogen to maintain an inert atmosphere.

X1.4.2 *Flow Meter*—A flow meter for nitrogen gas with a recommended range from 0 to 50 L/min for use in the furnace apparatus.

X1.4.3 *Flow Meter*—A flow meter for nitrogen gas with a recommended range from 0 to 10 L/min for use in the cool-down sagger apparatus.

X1.4.4 *Desiccator*—A desiccator of sufficient size to hold the specimens.

X1.4.5 *Specimen Tray*—A specimen tray to hold the specimens during pyrolysis and cool down. A stainless steel tray with permanently marked or numbered bins is recommended.

X1.4.6 *Cool-Down Sagger*—A cool-down sagger of sufficient size to enclose the specimen tray. Stainless steel construction is recommended to avoid heat damage from the hot specimen tray. The cool-down sagger is supplied with nitrogen to maintain an inert atmosphere.

X1.5 Test Specimen

X1.5.1 Test one test specimen per sample.

X1.6 Conditioning

X1.6.1 See Section 9.

X1.7 Procedure

X1.7.1 Cut a minimum 1-m specimen of fiber. Coil the fiber into a usable form taking care not to damage or lose filaments. It is recommended that the specimen mass not exceed 2 g.

X1.7.2 Determine the mass of the specimen using an analytical balance with ± 0.0002-g accuracy.

X1.7.3 Increase the nitrogen flow in the preheated furnace to reduce the influx of air. Remove a preheated tray from the furnace and place the specimens in the tray.

X1.7.4 Return the tray to the preheated furnace for the required time at temperature. Maintain the furnace nitrogen flow at a sufficient rate to maintain an inert atmosphere but not so high as to remove loose filaments from the samples.

X1.7.5 After the pyrolysis is complete, remove the specimen tray from the furnace and place in the cool-down sagger under nitrogen purge for approximately 15 min. Maintain the sagger nitrogen flow at a sufficient rate to maintain an inert atmosphere but not so high as to remove loose filaments from the samples.

X1.7.6 At the end of the cool-down period, remove the tray from the sagger and determine the mass of the desized fiber immediately. If this weighing will be delayed, place the specimens in a desiccator to prevent moisture adsorption on the fiber.

NOTE X1.1—It is necessary to place the desized fiber in a desiccator for this procedure for two reasons. First, the mass differences between the sized and desized fiber are small because of the small mass of size applied to the fiber (typically 1 %). Second, the desized fiber has a higher water adsorption than the sized or unsized fiber as a result of a small amount of oxidation occurring during this test. It is not necessary to keep the specimen desiccated for subsequent use as a density specimen because the density test has a 50 to 60 % mass difference making it much less sensitive to moisture adsorption affects.

X1.7.7 Run a standard of unsized fiber periodically to assure that no significant fiber weight loss is occurring.

X1.7.8 Perform a test pyrolysis condition study with any new sizing or fiber to determine the conditions under which the size is pyrolyzed and the fiber is not affected. Correlate the results of this study with pyrolysis results from unsized and sized standards or with acid digestion or solvent extraction results on unsized and sized fiber. Correlation constants determined from this study may be applied to compensate for any differences.

X1.7.9 *Retests*—Reject any specimen that has an obvious flaw or deviation from the requirements of this test method. Prepare and test a new or spare specimen from the same sample to replace any specimens eliminated in accordance with this paragraph.

X1.7.9.1 Discard test results for any condition which compromises the integrity of the test. These conditions must be documented.

X1.7.9.2 Any suspect single specimen or sample may be retested using double the number of specimens from the same sample unit. The average result of the retested specimens shall replace the original value. However, all values must be recorded.

X1.8 Calculations

X1.8.1 Calculate the percent size content as follows:

$$\% \text{ size content} = 100(W_1 - W_2)/W_1 \quad \text{(X1.1)}$$

where:

W_1 = mass of specimen before pyrolysis, g and

W_2 = mass of specimen after pyrolysis, g.

X1.8.2 Corrections to calculations may be used by application of correlation constants determined in X1.7.8.

X1.9 Percent Size Report

X1.9.1 Report the following information:

X1.9.1.1 Complete identification of the samples tested to assure traceability of test results,

X1.9.1.2 Date of test,

X1.9.1.3 Test facility,

X1.9.1.4 Pyrolysis conditions,

X1.9.1.5 Identification of the individual performing test,

X1.9.1.6 Size of the specimens tested, %, and

X1.9.1.7 Any deviations from this test method.