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MIL-Y-87125A(USAF)
27 July 1979
SUPERSEDING
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7 APRIL 1978

MILITARY SPECIFICATION

YARN, GRAPHITE, 1000/3000 FILAMENTS

This specification is approved for use by Air Force Materials Laboratory, Department of the Air Force, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification establishes the requirements and quality assurance provisions for continuous filament high modulus graphite yarn derived from pyrolyzed polyacrylonitrile filaments.

1.2 Classification. The yarn consists of 1000 filament or 3000 filament material exhibiting and minimum single fiber tensile strength of 360×10^3 psi (2480 MPa) and minimum single fiber tensile modulus of 52×10^6 psi (359 GPa).

Type I	1000 filaments
Type II	3000 filaments

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on the date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

MILITARY

MIL-C-45662 - Calibration System Requirements

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: AFML/MXA, WPAFB, O. 45433 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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STANDARDS

Military

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes

MIL-STD-129 - Marking for Shipment and Storage

MIL-STD-130 - Identification Marking of U.S. Military Property

MIL-STD-794 - Parts and Equipment, Procedures for Packaging and Packing of

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the Contracting Officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on the date of invitation for bids or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D891 - Specific Gravity of Industrial Aromatic Hydrocarbon and Related Materials

ASTM D1423 - Twist in Yarns by the Direct-Counting Method

ASTM D3379 - Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials

(Application for copies should be addressed to American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103).

SOCIETY OF AUTOMOTIVE ENGINEERS (AMS)

AMS 3892 - Fibers, Graphite Tow and Yarn - for Structural Composites

(Applications for Copies should be addressed to Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, Pennsylvania 15906).

(Technical Society and Technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal Agencies.)

When the second parameter is one, the discrete element is

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3. REQUIREMENTS

3.1 Preproduction. Three one pound spools of yarn which are processed and wound in accordance with the proposed production methods shall be subjected to preproduction tests prior to the beginning of production. The yarn shall meet the requirements of 3.2, 3.3, 3.4 and 3.5.

3.2 Product characteristics. The yarn shall conform to the following requirements.

3.2.1 Physical properties. The physical properties of the yarn shall conform to the requirements of Table I.

TABLE I. Physical characteristics.

	Type I	Type II
Length per unit weight lot average m/Kg Denier	12,860 to 15,000 600 to 700	4240 to 4690 1920 to 2120
Density, gm/cm^3, minimum	1.82	1.82
lbs/in^3, minimum	.066	.066
Carbon content, unsized percent, minimum	99.6	99.6

3.2.2 Construction. Yarn shall be formed from parallel lays of multifilament graphite fibers lightly stranded or twisted together with either approximately one-half turn per inch or 25 mm, or not more than one turn per foot or 300 mm, into one or more plies. If two or more plies, the final yarn shall be manufactured by twisting the plies together in a direction opposite to the twist direction of the individual plies. The number of twists used to make the final yarn shall be approximately the same as the number of twists used in making the individual plies. There shall be no residual rotation.

3.2.3 Splicing. When aplicing is necessary to achieve required package size, the yarn shall be spliced by an overlap joint made with a compatible resin. The minimum distance between splices shall be 500 feet or 150 m and the average distance between splices shall be not less than 2500 feet or 750 m. There shall be no unbonded overlap lengths (tails). The splice strength shall be capable of withstanding a 50 gram load as measured on the dry fiber bundle. A splice shall be not more than 3 inches or 75 mm long and shall be capable of being drawn through a slot two times as wide as the nominal fiber bundle diameter.

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3.2.4 Sizing. Polyvinyl alcohol sizing content shall be 0.4% to 1.5% for 1000 filament yarn & 0.4% to 1.2% for 3000 filament yarn. There shall be no accumulation of sizing in any local areas so as to form bubbles or drops on the yarn surface or to cause matting of the fiber bundle.

3.2.5 Minimum length. The yarn shall be manufactured or spliced into continuous lengths corresponding to standard size packages, such as 1, 2 and 4 pounds (0.45, 0.91, and 1.81 kg) as ordered by the procuring agency.

3.2.6 Surface condition. As unwound from its holder, the yarn shall not contain loose plies, cuts, tears, chafes or slips, excessive accumulations of broken fibers or fuzz balls. The yarn shall be tightly wound and its surface straight and centered on the holder core. It shall not break or tear during unwinding.

3.3 Mechanical properties. The mechanical properties of the yarn shall meet the requirements of Table II.

TABLE II. Tensile properties of 1000 and 3000 yarn filaments.

	Single filament	Yarn
TENSILE STRENGTH		
Lot Avg., Min		
PSI X 10^3	360	320
MPa	2480	2210
TENSILE MODULUS		
Lot Avg., Min		
PSI X 10^6	52	50
GPa	359	345

3.3.1 Tensile properties. Tensile strength and modulus testing may be conducted on either single filaments or on yarn bundles. No more than 10 per cent of the lot average value shall be below the minimum lot average specified in Table II.

3.4 Identification and marking. Identification and marking shall be in accordance with MIL-STD-130. Each spool of yarn shall be identified using a label affixed to the inside of one end of the yarn holder. The information on the label shall be in permanent ink. The label shall contain the following information:

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a. Manufacturer's type, grade, and finish
b. Lot number of yarn and date of manufacture
c. Twist
d. Net weight of holder
e. Purchase order number.

3.5 Workmanship. The graphite yarn shall be sized and wound on its holder in a careful and workmanlike manner. Particular attention shall be given to assuring that the yarn contains the proper number of filaments, the sizing is uniform and contains no bubbles, the splices are properly bonded and cured, and the yarn surface does not contain nicks, cuts, or fuzz balls.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Classification of inspections. The inspection requirements specified herein are classified as follows:

a. Preproduction testing (see 4.3).

b. Quality conformance inspection (See 4.4).

4.3 Preproduction tests. Subject each of the three one pound spools of preproduction yarn to the following tests. Verify compliance with 3.

a. Length/unit weight (4.6.1).
b. Density (4.6.2).
c. Carbon content (4.6.3).
d. Construction (4.6.4).
e. Splicing (4.6.5).
f. Sizing (4.6.6).
g. Surface condition (4.6.7).
h. Tensile strength & Modulus (4.6.8).
i. Identification & marking (4.6.9).
j. Workmanship (4.6.10).
k. Preparation for delivery (4.6.11).
l. Length (4.6.12).

4.4 Quality conformance inspection:

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4.4.1 Acceptance tests. Subject each lot of yarn to the following tests.

a. Length/unit weight (4.6.1).
b. Density (4.6.2).
c. Carbon content (4.6.3).
d. Construction (4.6.4).
e. Splicing (4.6.5).
f. Sizing (4.6.6).
g. Surface condition (4.6.7).
h. Tensile strength & modulus (4.6.8).
i. Ident & marking (4.6.9).
j. Workmanship (4.6.10).
k. Preparation for delivery (4.6.11).
l. Length (4.6.12).

4.4.1.1 Lot. A lot shall consist of all graphite fibers manufactured and gathered into a yarn in a continuous operation from the same batches of raw materials.

4.4.1.2 Sampling. Sampling for acceptance inspection shall be in accordance with special inspection level S-1, sample size in Table I and normal inspection Table IIA with an acceptable Quality Level of 10 as specified in MIL-STD-105. Test specimens shall be taken at random throughout each lot. If number of inspection units to be sampled equals or exceeds the production lot size, inspect each unit.

4.4.1.2.1 Inspection unit. Unless otherwise specified, an inspection unit is defined as each holder (see5.1.1) of continuous length yarn, but shall not exceed 5.0 lb. or 2.25 kg of material.

4.5 Test conditions. Unless otherwise specified tests shall be conducted at an atmospheric pressure of 28-32 inches of mercury, a temperature of 72 to 80° F (22 to 26° C), and a relative humidity of 40 to 60 percent.

4.5.1 Measurements. All measurements shall be taken with instruments whose accuracy has been verified in accordance with MIL-C-45662.

4.6 Test methods.

4.6.1 Length per unit weight. The following procedure shall be used to determine length per unit weight of yarn.

a. Measure a 3 meter length of yarn to the nearest millimeter.

b. Weigh the yarn to the nearest 10 milligrams.

c. Calculate the length per unit weight as follows:

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$$\text{Length per unit weight, m/kg} = \frac{L}{W} \times 10^6$$

where, L = yarn length, m
W = yarn weight mg

Verify compliance with 3.2.

4.6.2 Density. Density shall be determined in accordance with ASTM D891, Items C and D or an equivalent method. Verify compliance with 3.2.

4.6.3 Carbon content.

4.6.3.1 Sample preparation. Weigh approximately 1 gram of yarn into a tared 50 ml porcelain crucible. Heat 16 hours minimum in a muffle furnace at 400 ± 20°C. Cool in a desiccator. Determine the carbon content by gas chromatography. Verify compliance with 3.2. An acceptable apparatus is the F&M Model 185 CHN Analyzer in association with Cahn ratio - recording electrobalance and appropriate accessories and standards as follows.

4.6.3.1.1 Apparatus. A recommended apparatus is the F&M Model 185 CHN Analyzer which includes a Chan Ratio - Recording Electrobalance for weighing samples and a Honeywell Electronik 18 0-1 millivolt strip chart recorder to trace the nitrogen carbon dioxide and water peaks. Peak heights are measured with an accurate millimeter rule such as a Lufkin 30 millimeter rule graduated in one-half millimeter divisions. Sample boats, catalyst, sample rods and O-rings are supplied with the instrument.

4.6.3.1.2 Reagents. Standard materials of known elemental composition such as acetanilide, cyclohexanone-2, 4-dinitrophenylhydrazone (2,4-D) and melamine are supplied with the instrument. Additional standards, such as benzoic acid and cystine, may be purchased from the National Bureau of Standards.

4.6.3.2 Operation.

4.6.3.2.1 Instrument operating conditions.

a.	Oxidation Furance Temperature:	1050°C
b.	Reduction Furance Temperature:	400-550°C
c.	Column Oven Temperature:	90 ± 10°C
d.	Oven Shell Temperature:	75 ± 10°C
e.	Bridge Current:	155 milliamperes

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4.6.3.2.2 Instrument conditioning. At the beginning of each day's work, the instrument must be conditioned. This is accomplished by repeated combustion of cyclohexanone-2,4-dinitrophenylhydrazone (2,4-D) until the N_2CO_2 and H_2O peaks are well defined. The amount of 2,4-D need only be estimated (rather than weighed) for the conditioning step. Usually, no more than three combustions are necessary.

4.6.3.3 Weighing procedure.

4.6.3.3.1 Calibration of balance.

a. Move the range switch to the extreme clockwise position.

b. Rotate the range switch counterclockwise two click stops (x 10 range).

c. Using tweezers, place a sample boat on each stirrup.

d. Move the main switch to the ZERO position.

e. Turn the BALANCE control to the full counterclockwise position.

f. Move the coarse level until the beam is just slightly below the reference lines (viewed through the illuminated lens).

g. Close the door and precisely align the beam and the reference lines using the BALANCE control.

h. Depress the meter switch and hold it down while rotating the ZERO control to precisely align the null meter needle with the center mark. Use the mirror to be certain that parallax does not produce a false reading. Release the meter switch when properly nulled. Depress momentarily to note any slight deflection. Null again if there is.

i. Turn the BALANCE control clockwise to the sixth click position. Using tweezers, place the 10 mg calibrating weight on the left sample boat.

j. Close the sliding glass door.

k. Turn the main switch to the CAL position.

l. Align the beam with the reference lines by slowly rotating the BALANCE Control counterclockwise.

m. Hold the meter switch depressed while nulling the meter with the CALIBRATE Control. Release the meter switch when properly nulled.

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n. Move the main switch to the ZERO position.

o. Open the sliding door and remove the calibrating weight.

p. Rotate the range switch to the X1 position (fully clockwise).

4.6.3.3.2 Obtaining sample weight.

a. Rotate the BALANCE control to the extreme counterclockwise position (past all six click stops).

b. Rotate the BALANCE control slowly clockwise until the beam aligns with the reference line. Do not rotate the BALANCE control to a point where it passes the first click stop. If the beam cannot be aligned without going past the first clock stop, return the BALANCE control to the extreme counter-clockwise position and adjust the coarse lever until the beam approaches the reference lines. At this point, return to the BALANCE control for the precise final alignment.

c. Depress the meter switch and hold while nulling the meter with the ZERO control.

d. Rotate the BALANCE to the fourth click position.

e. Open the door and carefully tap sample from the spatula into the left sample boat until the beam approaches the reference lines. High boiling, (nonvolatile) liquid samples can be introduced into the sample boat by means of a micro pipette or dropper. Volatile liquids must be first loaded into capillary tubes as described in the F&M Instruction Manual supplied with the instrument.

f. Close the door and move the main switch to the READ position.

g. Slowly turn the BALANCE control counterclockwise (fine range) to precisely align the beam with the reference lines.

h. Hold meter switch depressed while nulling meter using the weight dial. When perfectly nulled, release meter switch.

i. The weight of sample can be read directly from the dial in decimals of mg., such as, 6198 = 0.6198 mg. NOTE: If reading is above 0.8000 mg, remove same sample; if below 0.6000 mg, add sample.

4.6.3.4 Preparation of sample for combustion.

a. Position one of the combustion rod assemblies on the rod holder and slide the retaining nuts to within 2 inches of the rod end. The small retaining nut must not be tightened into the large nut.

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b. Using tweezers, remove the weighed sample from the balance and place it in the boat cavity of the rod.

c. Pack the boat with oxidizing catalysts until it is even with the top of the boat. Tap the catalyst down firmly with the spatula. Samples that are difficult to burn should be mixed with a small amount of catalyst prior to filling boat.

d. Remove the hot combustion rod from the furance by unscrewing the large retainer nut and pulling the entire assembly outward. Place the hot rod in the cooling position.

e. Position the loaded rod in the SAMPLE PORT and immediately secure in position by tightening the large retaining nut. Use only hand pressure for this step. Never use a wrench or pliers.

f. Hand tighten the small retaining nut. Be careful to keep the handle grip in a perfectly vertical position while tightening the nut, or the sample will fall out into the neck of the combustion tube. When the hot rod was removed, the carrier system was partially opened to atmosphere; and when the loaded rod was installed, the system was resealed. As a result, the atmosphere slug that was admitted in the interim will now be carried onto the chromatographic column by the restored carrier gas flow. The detector and recorder will show this chain of events. The next sequence of steps is also recorded on the scan.

g. After the atmosphere peak is recorded and the baseline is returned to the approximate normal position, loosen the small retaining knob slightly while holding the rod handle to prevent it from turning. Depress the START TIMED CYCLE pushbutton, then push the rod into the combustion tube as far as it will go. Do not completely loosen the small nut to perform the above step. It is only necessary to loosen it to the point when the rod can be pushed in about one fourth turn.

NOTE: The instrument will now combust the sample for 40 seconds. The gases will then be swept into the reduction furance and chromatographic column. If automatic attenuation mode is used, the instrument will automatically attenuate the recorder signal to keep all the peaks on scale. If the manual mode of attenuation is used, adjust attenuation for each peak as follows:

Peak 1, Nitrogen, x 8
Peak 2, Carbon Dioxide, x 32
Peak 3, Water, X 8.

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4.6.3.4.1 Calibration.

a. Set the weight dial to read 700 and set the SENSITIVITY SELECTOR to MANUAL. Move the ATTUNUATION control to the 8 position. Place an empty sample boat in the rod cavity.

b. Fill the boat with catalyst and pack the catalyst lightly with a spatula.

c. Load the catalyst-only rod into the instrument and run a chromatogram.

d. The resulting chromatogram will show how much C-H-N is contained in the catalysts. Under normal conditions the first peak (N) will not exceed 1 scale division high; the second peak (C) will not exceed 4 scale divisions; and the third peak (H) will not exceed 5 divisions. If the peaks fall within the tolerances shown in Table V (at a manual X 8 attenuation), skip the next step and go to step f).

TABLE V. Chromatograph tolerances.

	Normal Height (Scale Divisions)	Upper Limit (Scale Divisions)
1st Peak (N)	1	1
2nd Peak (C)	4	8
3rd Peak (H)	5	10

e. If the peaks exceeded the upper limit shown in column 3 of Table V, recondition the catalysts according to the manufacturer's instructions.

f. If the blank peak heights are within tolerance, continue running blanks until the peaks are at a minimum level and are reproducible. Contaminants used in the manufacturing of the combustion rods will also be eliminated as the blanks are run.

g. Record the peak height of the C,H, & N peaks to be used later in calibrating the instrument. Remember to divide the C peak height by a factor of 4, as we will now switch back to automatic operating, and the C peak will be attenuated (x 32).

h. Move the SENSITIVITY SELECTOR switch to the AUTOMATIC position. If desired, however, analysis can be made with this switch in the MANUAL position. In this case, peaks must be attenuated by the operator.

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4.6.3.4.2 Standard sample. A standard sample whose carbon content most closely matches that of the unknown and therefore gives a CO_2 peak similar in height to that obtained on the unknown is used to obtain the correction contents. From the measured peak height values, the constants are computed as follows; using 2,4-D as an example:

$$\text{Carbon } K_C = \frac{51.79 \text{ percent}}{(\text{Measured C Peak Height from sample run}) - (\text{Measured C Peak Height from blank run})}$$

4.6.3.4.3 Unknown sample. The unknown sample is treated in exactly the same manner, and using the above calculated constants of proportionality, the contents are computed, such as percent C equals peak height unknown x K_C.

4.6.4 Construction. The yarn shall be tested in accordance with ASTM D1423. Verify compliance with 3.2.2.

4.6.5 Splice breaking strength. Splices in the yarn conforming to the requirements specified in 3.2.3 and 3.5 shall withstand a 50 gram load applied on the dry fiber bundle with the splice in the test length of the yarn.

4.6.6 Sizing content. Determine the percent PVA content of the finished yarn by heating an accurately weighed sample in air at 600 F (315 C). Verify compliance with 3.2.4.

4.6.6.1 Apparatus.

Porcelain Crucibles - 50 to 100 CC capacity such as Coors No. 2 or 3.

Analytical Balance - Any standard balance having an accuracy of 0.0001 gram (0.1 mg) such as a Mettler B-5.

Desiccator - Standard type.

Oven - Freas Model 14 or equivalent which can be maintained at 600 ± 5°F (315 ± 3°C) for long periods of time.

4.6.6.2 Procedure.

4.6.6.2.1 Crucible preparation. Heat the crucibles at 600°F or higher for 6 hours or more. Place the heated crucibles in a desiccator and cool to room temperature. Weigh the crucibles to the nearest tenth of a milligram (0.1 mg).

4.6.6.2.2 Sample preparation. Place a 1 to 2 gram sample of the finished yarn in a previously conditioned, weighed crucible and weigh to the nearest milligram. Note: An approximate 2 gram sample should be used when the PVA content is expected to be lower than 1 percent.

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4.6.6.2.3 PVA determination. Place the crucible with sample in an oven and heat at 600 ± 5°F (315 ± 3°C) for 15 to 17 hours. Place the heated crucible with sample in a desiccator, cool to room temperature, then weigh to the nearest milligram.

4.6.6.2.3.1 Calculation. The difference between the weight of the initial sample and the weight of the sample after 15-17 hours heating is the weight of PVA. The percent PVA content on the yarn is calculated by:

$$\% \text{ PVA} = \frac{\text{Wt. of initial sample} - \text{Wt. of sample after heating}}{\text{Wt. of initial sample}} \times 100$$

4.6.7 Surface condition. The yarn, as unwound from its holder, shall be visually inspected for conformance to 3.2.6.

4.6.8 Tensile properties.

4.6.8.1 Tensile properties of yarn. Tensile properties of the yarn shall be determined in accordance with the procedures given in AMS 3892 or an equivalent method. Verify compliance with 3.3.

4.6.8.2 Tensile properties of single filaments. Tensile properties of single filaments shall be determined in accordance with ASTM 3379, using nine specimens for each determination. Verify compliance with 3.3.

4.6.9 Identification and marking. Visually inspect the yarn holder to verify compliance with 3.4.

4.6.10 Workmanship. Visually examine the yarn to verify compliance with 3.5.

4.6.11 Preparation for delivery. Visually inspect the individual yarn/holder packages, packing of the individual packages, and markings of interior packages and the shipping container. Verify compliance with Section 5.

4.6.12 Length. Weigh the yarn/holder package. Verify compliance with 3.2.5.

5. PACKAGING

5.1 Preservation-packaging. The yarn shall be wound on holders as specified by the purchase order. The average weight per holder shall be not less than that specified in the purchase order.

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5.1.1 Individual holders shall be separately packaged in accordance with MIL-STD-794, Level A, or Level C, as specified in the purchase order. Commercial packaging by the manufacturer to assure transportability and ease of handling while maintaining the material shipped in its most usable form is acceptable unless Level A or Level C is specified.

5.2 Packing. Individual packages shall be packed in an exterior shipping container in accordance with MIL-STD-794, Level A or Level C. Only one type class, and grade shall be in one package. Commercial packing by the manufacturer to assure transportability and ease of handling while maintaining the material in its most usable form is acceptable unless Level A or Level C is specified.

5.3 Marking. In addition to any special marking required by the contract or order, interior packages and shipping containers shall be marked in accordance with MIL-STD-129.

5.3.1 Interior marking. Each interior package (holder) shall carry a sticker exhibiting the following information:

a. Manufacturer's type, grade, and finish

b. Lot number of yarn and date of manufacture

c. Twist

d. Net weight of holder

e. Purchase order number.

5.3.2 Shipping carton. Each shipping carton containing one to twenty-four holders of yarn shall be marked as follows:

a. Manufacturer's grade

b. Manufacturer's production order number

c. Net weight in carton

d. Customer's purchase order number

e. Number and date of this specification

6. NOTES

6.1 Intended use. The graphite yarns covered by this specification are intended for reinforcement in fiber reinforced composites.

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6.2 Ordering data. Purchasers should select the preferred options permitted herein and include the following information in their procurement documents.

a. Title, number and date of this specification.

b. Type, class and grade of fiber required (see 1.2).

c. Level of twist required.

d. Package (holder) size (see 5.1.1).

e. Quantity of fiber desired.

f. Level of packaging and packing desired (see 5.1.2 and 5.2).

6.3 Preproduction approval information. The request for preproduction approval should be accompanied by the results of test on the material submitted and three packages of material, approximately 1 pound or 0.45 kg net each of each type of yarn for which approval is desired.

Custodian:
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