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7 April 1978

MILITARY SPECIFICATION

# RODS, PULTRUDED, GRAPHITE FIBER REINFORCED, PROCESSING OF

This specification is approved for use by the 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 processing requirements and quality assurance provisions for pultruded rods.

## 2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on 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 Systems Requirements |
| MIL-Y-87125 | Yarn, Graphite, 1000/3000 Filaments |

(Copies of specifications, 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 date of invitation for bids or request for proposal shall apply.

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

FSC 9330

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

| | |
|---|---|
| ASTM D445 | Kinematic Viscosity of Transparent and Opaque Liquids |
| ASTM D1553 | Water in Insulating Liquids (Karl Fischer Method) |
| ASTM D1652 | Epoxy Content of Epoxy Resins, Test for |

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

3. REQUIREMENTS

3.1 Equipment. The equipment shall consist of the following:

3.1.1 Creel. A textile payoff device capable of handling a minimum of two one-pound standard cardboard spools of yarn.

3.1.2 Resin bath. A metal container capable of containing one pint of resin with metal or plastic rollers ½ to 2 inches in diameter.

3.1.3 1st B-Stage oven. Capable of maintaining a temperature of 107 ± 14°C (225 ± 25°F). A typical oven would be 6 to 7 feet long.

3.1.4 2nd B-Stage oven. Capable of maintaining a temperature of 204 ± 27°C (400 ± 50°F). A typical oven would be 6 to 7 feet long.

3.1.5 Curing oven. Capable of maintaining a temperature of 260 ± 27°C (500 ± 50°F). A typical oven would be 2 to 3 feet long.

3.1.6 Take-up rollers. Adjustable, capable of pultruding the rod at 15 to 25 feet per minute. The rolls shall be fabricated of metal or plastic with a rubber coating.

3.1.7 Analytical balance. (One-pan reading model such as Sartruious or Mettler Types.)

3.1.8 25 ml graduated cylinder. Pyrex No. 3022 or equivalent.

3.1.9 50 ml buret.

3.1.10 Magnetic stirrer and glass enclosed stirring element.

3.1.11 200 ml beaker.

3.1.12 100 ml graduated cylinder.

3.1.13 250 ml flask.

3.1.14 Dies. Standard wire drawing dies:

Die 1 - 0.030 to 0.035 inch diameter.
Die 2 - 0.025 to 0.027 inch diameter.

3.1 Materials.

3.2.1 Yarn. Conforming to Specification MIL-Y-87125, type II.

3.2.2 Epoxy resin. Conforming to the following requirements.

3.2.2.1 Moisture content. The moisture content of the resin shall be 0.4 percent by weight maximum.

3.2.2.2 Specific gravity. The specific gravity of the resin shall be 1.17 to 1.27 at 20°C ± 1°C (68° ± 1.8°F).

3.2.2.3 Epoxide equivalent. The epoxide equivalent of the resin shall be 150 to 170 grams of resin, containing one gram equivalent.

3.2.2.4 Viscosity. The viscosity of the resin shall be 1.0 to 3.5 poises at 0 to 10°C (32 to 50°F).

3.2.2.5 Storage stability. The resin shall meet all the requirements of this specification after storage in the original unopened container at 16 to 35°C (60 to 95°F) for a period of one year from the date of receipt.

3.2.3 Modified polyamine hardener. Conforming to the following requirements.

3.2.3.1 Specific gravity of hardener. The specific gravity of the hardener shall be 1.13 to 1.14 at 23 ± 1°C (73.5 ± 2°F).

3.2.3.2 Viscosity of hardner. The viscosity of the hardener at 23 ± 1°C (73.5 ± 2°F) shall be 3600 to 5900 centiposes.

3.2.3.3 Amine value. The amine value of the hardener shall be 75 to 85 grams of hardener per gram equivalent.

3.2.3.4 Storage stability. The hardener shall meet all the requirements of this specification after storage in a sealed container at ambient temperature of 16 to 35°C (60 to 95°F) and for a period of one year from the date of receipt.

3.2.4 Nitrobenzene. (Eastman Kodak Reagent No. 387, or equivalent.)

3.2.5 Glacial acetic acid. (Reagent grade).

3.2.6 Perchloric acid. 70-72 percent (Reagent grade).

3.2.7 Potassium acid phthalate. Primary standard grade.

3.2.8 Crystal violet indicator solution.

3.3 Procedure.

3.3.1 Setup.

a. Remove spools of yarn from container.
b. Remove plastic film from spools of yarn.
c. Install spools of yarn on creel.
d. Install excess resin removal die (Die #1, paragraph 3.1.14.)
e. Install compaction die (Die #2, paragraph 3.1.14.)
f. Turn on first B-stage oven and set temperature at 107 ± 14°C (225 ± 25°F).
g. Turn on second B-stage oven and set temperature at 204 ± 27°C (400 ± 50°F).
h. Turn on curing oven and set temperature at 260 ± 27°C (500 ± 50°F).

i. Draw the yarn from the 2 spools on the creel simultaneously and with a smooth motion pass through the resin bath container, excess resin removal die, first B-stage oven compaction die, second B-stage oven, curing oven and drive rollers. A schematic of a typical rod pultruding facility is shown in Figure 1.
j. Stabilize ovens.
k. Mix resin of paragraph 3.2.2, and hardener of paragraph 3.2.3 by hand, stirring 1 to 2 minutes, formulation shall be as follows:

| Material | Parts by weight |
|---|---|
| Epoxy resin per 3.2.2 | 100 ± 1 |
| Hardener per 3.2.3 | 28 ± 1 |

l. Pour resin into resin bath and maintain at room temperature.

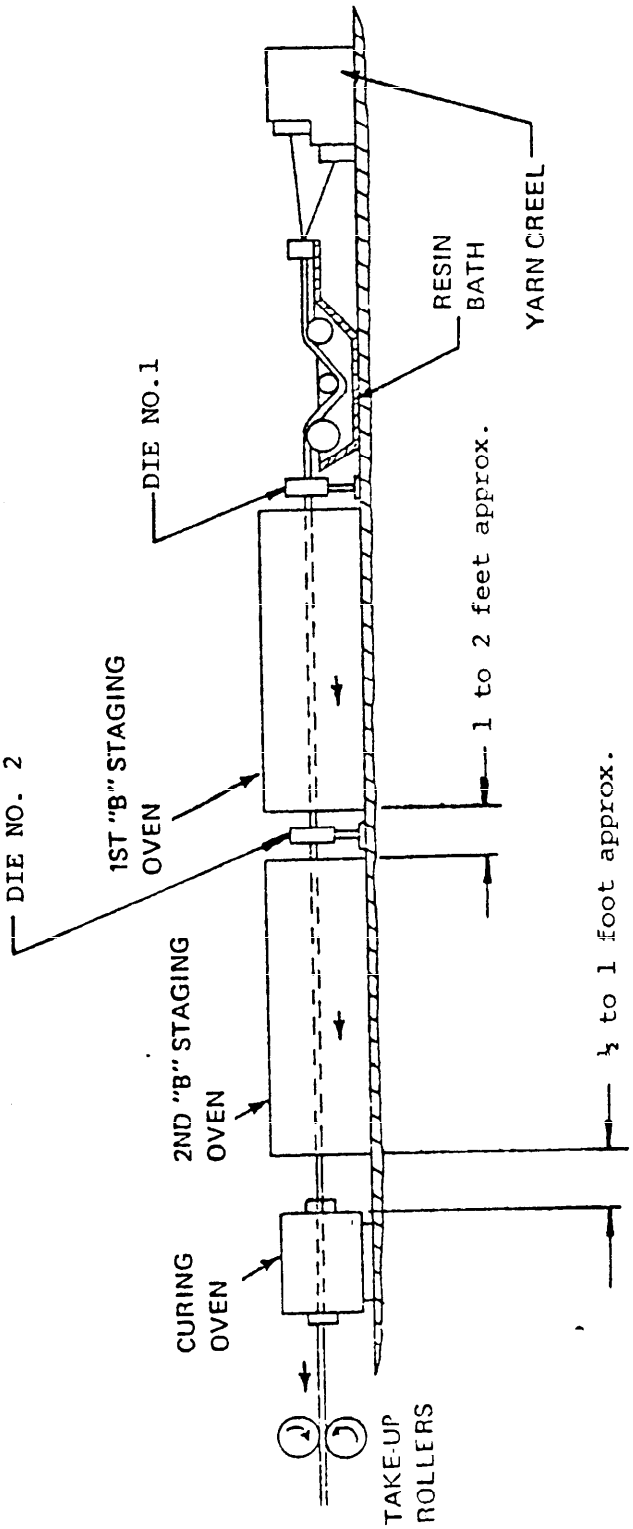


FIGURE 1. Schematic of rod pultruding facility.

3.3.2 Operation.

a. Turn on drive roller and set speed to achieve a rate of 20 ± 5 feet per minute.
b. Allow dry yarn to run through facility and discard.
c. Pultrude 20 ± 10 feet of rod.
d. The resin bath shall be changed and the dies cleaned at least once every one and a half hours. The old resin shall be discarded and new resin prepared per paragraph 3.3.1.

3.3.3 Rod inspection. After completion of the pultruding process, the rod shall meet the following requirements.

3.3.3.1 Separation. There shall be no separation of fibers.

3.3.3.2 Size. The sample rod shall be between 0.024 and 0.031 inches in diameter.

3.4 Certification. Ten rods, fabricated in accordance with this process shall be subjected to certification tests of 4.4. Each rod shall meet the requirements of 3.3.3.

4. QUALITY ASSURANCE PROVISIONS

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

4.2 Calibration. All measurements shall be made using instruments whose accuracy has been verified in accordance with MIL-C-45662. All instruments shall be calibrated every six months. The curing and staging ovens shall be calibrated for temperature and stability to ± 10°C daily while operating and every six months against a master standard.

4.3 Inspection conditions. Unless otherwise specified, tests shall be made at an atmospheric pressure of 28 to 32 inches of mercury and at a temperature of 22 to 26°C (75 ± 4°F) and a relative humidity of 40 to 60 percent.

4.4 Special tests and examinations.

4.4.1 Process certification. Randomly select and remove a 12 ± 1 inch test section from each of the first ten rods produced and subject the test sections to the following tests:

| | | |
|---|---|---|
| a. | Separation | (4.6.8) |
| b. | Size | (4.6.9) |

4.4.2 Raw material testing.

4.4.2.1 Yarn. Each lot of yarn received shall be verified for conformance to 3.2.1.

4.4.2.2 Resin. A one quart sample of resin, from each lot of received, shall be subjected to the following tests:

| | | |
|---|---|---|
| a. | Moisture | (4.6.1) |
| b. | Specific gravity | (4.6.2) |
| c. | Expoxide equivalent | (4.6.3) |
| d. | Viscosity | (4.6.4) |

4.4.2.3 Hardener. A one quart sample of hardener, from each lot received, shall be subjected to the following tests:

| | | |
|---|---|---|
| a. | Specific gravity | (4.6.5) |
| b. | Viscosity | (4.6.6) |
| c. | Amine value | (4.6.7) |

4.5 Quality conformance inspection.

4.5.1 Acceptance testing. Acceptance testing shall consist of in-process inspection and individual tests of 4.5.1.1 and 4.5.1.2.

4.5.1.1 In-process inspection. During the processing of each lot of rods, verify the following conditions:

| | | |
|---|---|---|
| a. | First B-stage oven temperature | (3.3.1f) |
| b. | Second B-stage oven temperature | (3.3.1g) |
| c. | Curing oven temperature | (3.3.1h) |
| d. | Resin coating formulation | (3.3.1k) |

4.5.1.2 Acceptance tests. Take a 6 to 12 inch section from between each 20 feet of rod produced and subject it to the following tests:

| | | |
|---|---|---|
| a. | Separation | (4.6.8) |
| b. | Size | (4.6.9) |

4.6 Test methods.

4.6.1 Moisture. Determine the moisture content in accordance with ASTM D1553. Check for conformance to 3.2.2.1.

4.6.2 Specific gravity. Determine the specific gravity in accordance with the following procedure. Check for conformance to 3.2.2.2.

4.6.2.1 Preparation of graduated cylinder. The graduated cylinder is cut off as close as possible to the 25 ml mark using a silicon carbide glass cut off wheel or other suitable tool. The cut is made as rectininear as possible and the edges may be smoothed by grinding or fire polishing.

4.6.2.2 Rapid determination of density or specific gravity. The graduate is standardized as indicated below. The true volume, given to the nearest 0.01 ml is engraved on the base with a vibra-tool or indicated on a label affixed to the base and protected with a chemically inert coating of Krylon or a similar clear lacquer. If several of these graduates are prepared, a unique identification number or letter for each one shall be affixed.

4.6.2.3 Calibration of graduated cylinder.

a. Weigh the clean, dry and empty graduate to the nearest milligram and record this weight as $W_1$ grams.

b. Fill the graduate with room temperature distilled water so that the meniscus rises above the top of the graduate. Carefully slide a glass plate or other flat surface, larger than the cross-section of the graduate, across the top to "level off" the liquid. Wipe the outside of the graduated with a lint-free absorbent tissue. Weigh the filled graduate to the nearest milligram and record the weight as $W_s$ grams. Immediately after removal from the balance, insert a thermometer in the water and record the temperature to the nearest degree as $t_s$. Duplicate determinations should agree within $\pm$ 0.03 grams.

4.6.2.4 Density measurement of unknown material.

a. Weigh the clean, dry and empty graduate to the nearest milligram and record this wieght as $W_1$ grams.

b. Fill the graduate with the material to be measured which is in equilibrium with room temperature. Fill the graduate so that the top of the meniscus is level with the top of the graduate. This may be accomplished, by leveling off the top of viscous liquids by using a wooden "tongue depressor" or stirring rod. Remove excess materials from the outside of the graduate, wipe lint-free absorbent tissue and weigh the filled graduate to the nearest milligram. Record this weight as $W_2$ grams and the temperature of the material to the nearest degree centigrade as $t_c$. Duplicate determinations should agree within $\pm$ 0.05 grams.

4.6.2.5 Calculations.

a. Standardization.

From Table I, obtain the apparent density of water in air, $d^t$ at the temperature, $t_s$ of the standardization. The true volume of the $H_2O$ graduate at 20°C, $V_s$, is then given by:

$$V_s = \frac{W_s - W_1}{d^t}$$

b. Density of unknown material.

The density ($d^t$) of an unknown material at temperature t°C, is calculated by:

$$d^t = \frac{W_2 - W_1}{V_s} \quad g/ml$$

c. Specific gravity of unknown material.

The specific gravity,

$$\text{sp. gr.} \frac{t_c}{t_s} = \frac{W_2 - W_1}{W_s - W_1} \quad g/ml$$

Where $t_c$ is the temperature at which the density of the unknown material is measured and $t_s$ is the temperature of the standard material.

TABLE I. Apparent density of water weighed in air; calibrated table for determining true volume of glassware at 20°C.

| Temperature °C | Apparent Density ($d^t$) g./ml. | Temperature °C | Apparent Density ($d^t$) g./ml. |
|---|---|---|---|
| 15 | 0.99793 | 26 | 0.99594 |
| 16 | 0.99780 | 27 | 0.99570 |
| 17 | 0.99765 | 28 | 0.99545 |
| 18 | 0.99751 | 29 | 0.99519 |
| 19 | 0.99734 | 30 | 0.99492 |
| 20 | 0.99718 | 31 | 0.99464 |
| 21 | 0.99700 | 32 | 0.99435 |
| 22 | 0.99680 | 33 | 0.99406 |
| 23 | 0.99661 | 34 | 0.99375 |
| 24 | 0.99639 | 35 | 0.99345 |
| 25 | 0.99618 | | |

4.6.3 Epoxide equivalent. Determine the epoxide equivalent in accordance with ASTM D1652. Check for conformance to 3.2.2.3.

4.6.4 Viscosity. Determine the viscosity of the epoxy resin using the Ubbelohde method in accordance with ASTM D445 at a temperature of 22 to 24°C (75 to 79°F). Check for conformance to 3.2.2.4.

4.6.5 Specific gravity. Determine the specific gravity in accordance with 4.6.2. Check for conformance to 3.2.3.1.

4.6.6 Viscosity. The hardener shall be placed in a 600 ml Griffin low form beaker for equivalent) with spout and the beaker placed in a constant temperature bath at 24 to 26°C. When the sample temperature has stabilized at the bath temperature, the viscosity shall be determined with a Brookfield Viscosimeter Model No. RVF using spindle number 2 and a speed of 5 revolutions per minute. Check for conformance to 3.2.3.2.

4.6.7 Amine value. Determine the amine value as follows. Check for conformance to 3.2.3.3.

4.6.7.1 Standardization of 0.1 N perchloric acid. Into a clean wide mouth 250 ml Erlenmeyer flask, accurately weigh 0.6 to 0.8 grams of dried potassium acid phthalate, recording this weight as $W_S$ grams. Dissolve this sample in 50 ml of glacial acetic acid, add four drops of 0.1 percent crystal violet indicator solution and titrate with perchloric acid-acetic acid solution to a greenish-blue color which persists for 30 seconds. Record the volume consumed in this titration as $V_S$ ml.

4.6.7.2 Sample $V_A$. Into a clean dry 200 ml beaker, accurately weigh a 0.2 to 0.5g sample. Record this weight as $W_A$ grams. Add 90 ml of nitrobenzene, stir with the magnetic stirrer and warm if necessary to dissolve the sample. Add 20 ml of glacial, acetic acid, stir and cool if necessary. Add six drops of the crystal violet indicator solution and titrate with the perchloric acid-acetic standard solution to a green end-point; record the volume as $V_A$ ml.

4.6.7.3 Sample $V_B$. Prepare a blank solution exactly as above, omitting only the addition of the sample and record the volume of standard perchloric acid consumed in this blank titration as $V_B$ ml.

4.6.7.4 Calculations.

Standardization of 0.1 N perchloric acid.

$$N = \frac{W_S}{0.20422\ V_S}$$

Amine value (mg of KOH equivalent to 1g of sample).

$$\text{Amine Value} = \frac{(V_A - V_B)\ (N)\ (56.1)}{W_A}$$

4.6.8 Separation. Visually inspect the rod. Verify compliance with 3.3.3.1.

4.6.9 Size. Pass the entire length of the rod through a 0.031 inch diameter gage. Determine if either end of the rod will enter a 0.024 inch diameter gage. Verify compliance with 3.3.3.2.

4.7 Storage stability. The resin and hardener shall pass the tests of 4.4.2.2 and 4.4.2.3 after storage in the original unopened container for a period of one year from the date of receipt. The storage temperature for the hardener shall be 16 to 35°C (60 to 95°F) and for the resin, 0 to 10°C (32 to 50°F). Unused resin and hardener in stock shall be tested in accordance with 4.4.2.2 and 4.4.2.3 after one year and at six month intervals thereafter.

5. PACKAGING

This section is not applicable to this specification.

6. NOTES

6.1 Intended use. The pultruded rods are intended for use in the fabrication of reinforced composites.

6.2 Definitions.

6.2.1 Lot. A lot shall be defined as the quantity of material that is produced during a single continuous operation within one processing container.

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(Project No. 9330-F859)

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