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AEROSPACE MATERIAL SPECIFICATION

SAE

AMS 3616B

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Superseding AMS 3616A

Submitted for recognition as an American National Standard

RESIN, POLYIMIDE, LAMINATING AND MOLDING
High Temperature Resistant, 370 °C (700 °F)
Unfilled, Addition Polymer

This specification has been declared "NONCURRENT" by the Aerospace Materials Division, SAE, as of July, 1992. It is recommended, therefore, that this specification not be specified for new designs.

This cover sheet should be attached to revision "A" of the subject specification.

"NONCURRENT" refers to those materials which have previously been widely used and which may be required on some existing designs in the future. The Aerospace Materials Division, however, does not recommend these as standard materials for future use in new designs. Each of these "NONCURRENT" specifications is available from SAE upon request.

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SAE AMS 3616A

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RESIN, POLYIMIDE, LAMINATING AND MOLDING
High Temperature Resistant, 370°C (700°F)
Unfilled, Addition Polymer

1. SCOPE:

- 1.1 Form: This specification covers a single-component, unfilled, heat-reactive, thermosetting, acetylene-substituted, aromatic-polyimide resin in the form of dry solid powder or particles. The resin thermally cures to a polyimide polymer.
- 1.2 Application: Primarily as a laminating and molding resin useful in closed-die or press-cured fabrication of composite structures requiring long-term exposure (500 - 1000 hr) up to 315°C (600°F), moderate-term exposure (25 - 50 hr) up to 370°C (700°F), or short-term exposure (1 - 2 hr) up to 425°C (800°F).

2. APPLICABLE DOCUMENTS: The following publications form a part of this specification to the extent specified herein. The latest issue of Aerospace Material Specifications shall apply. The applicable issue of other documents shall be as specified in AMS 2350.

- 2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

2.1.1 Aerospace Material Specifications:

AMS 2350 - Standards and Test Methods
AMS 3892/1 - Tow or Yarn, Carbon (Graphite) Fibers, For Structural Composites, GF 400 (2760) Tensile Strength, 33 (228) Tensile Modulus

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2.2 ASTM Publications: Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

- ASTM D150 - A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials
- ASTM D638 - Tensile Properties of Plastics
- ASTM D695 - Compressive Properties of Rigid Plastics
- ASTM D790 - Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials
- ASTM D792 - Specific Gravity and Density of Plastics by Displacement
- ASTM D2583 - Indentation Hardness of Plastics by Means of a Barcol Impressor

2.3 U.S. Government Publications: Available from Commanding Officer, Naval Publications and Form Center, 5801 Tabor Avenue, Philadelphia, PA 19120.

2.4 Military Standards:

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

3. TECHNICAL REQUIREMENTS:

3.1 Material: The uncured resin shall be a dry, solid, single-component, fusible, thermosetting, aromatic-polyimide powder, soluble in a solvent system of N-methylpyrrolidinone (N-methylpyrrolidone) or dimethylformamide, which cures to form a polyimide molecular structure.

3.2 Storage Life: The dry, solid resin, stored in vendor-packaged, air-tight containers at not higher than 30°C (85°F), shall meet the requirements specified herein when tested at any time up to one year from date of manufacture; it shall also meet these requirements when tested at any time up to 60 days from date of receipt by purchaser when stored at not higher than 50°C (120°F).

3.3 Properties of Uncured Resin: The as-received, dry resin shall conform to the following requirements; tests shall be performed on the product supplied and in accordance with specified test methods:

3.3.1 Softening Temperature: Shall be in the range 175° - 200°C (347° - 392°F), determined in accordance with 4.5.1.1.

3.3.2 Ratio of Aromatic C-H to Acetylenic C-H: The ratio of the area under the aromatic C-H peaks to the area under the acetylenic C-H peaks shall be within the range 18:1 to 24:1, determined in accordance with 4.5.1.2.

3.3.3 Solubility: The resin shall be soluble in N-methylpyrrolidinone (N-methylpyrrolidone) to the extent of not less than 13%, determined in accordance with 4.5.1.3.

3.3.4 Gel Time: Shall be 100 - 230 sec, determined in accordance with 4.5.1.4.

3.3.5 Infrared Spectrum: The infrared transmission spectrogram of uncured production resin shall not deviate from that of the preproduction test sample of 4.4.1 by more than agreed upon by purchaser and vendor, determined in accordance with 4.5.1.5.

3.4 Properties of Cured Resin: The resin, molded and cured in accordance with 4.5.2 and machined into suitable test specimens, shall conform to the following requirements; tests shall be performed at 20° - 30°C (70° - 85°F), unless otherwise specified herein, and in accordance with specified test methods:

3.4.1 Specific Gravity	1.30 - 1.40	ASTM D792
3.4.2 Hardness, Barcol	50 $\pm$ 5	ASTM D2583
3.4.3 Tensile Strength, min	10,000 psi (69 MPa)	ASTM D638, Specimen Type IV, Speed A or Strain rate: 0.04 in. (1.0 mm) per min.
3.4.4 Elongation, min	0.8%	Same as 3.4.3
3.4.5 Secant Modulus, min	500,000 psi (3450 MPa)	Same as 3.4.3
3.4.6 Flexural Strength, min	13,000 psi (90 MPa)	ASTM D790, Method I
3.4.7 Flexural Modulus, min	500,000 psi (3450 MPa)	ASTM D790, Method I
3.4.8 Compressive Strength, min	25,000 psi (170 MPa)	ASTM D695
3.4.9 Dielectric Constant at 9.4 $\pm$ 0.1 GHz	3.3 $\pm$ 0.2	ASTM D150
3.4.10 Dissipation Factor at 9.4 $\pm$ 0.1 GHz	0.01 max	ASTM D150

3.5 High Temperature Composite Properties: The resin, reinforced with randomly oriented chopped graphite fibers and molded as specified in 4.5.3, shall conform to the following requirements, determined in accordance with ASTM D790:

3.5.1 <u>Flexural Properties:</u>	Flexural Strength	Flexural Modulus
3.5.1.1 Flexural Properties at Room Temperature, min	50,000 psi (345 MPa)	4,000,000 psi (27,580 MPa)

3.5.1.2 Flexural Property Change, max, at room temperature after 500 hr at 315°C (600°F) -10% -10%

3.5.1.3 Flexural Property Change, max, at room temperature after 25 hr at 370°C (700°F) -10% -15%

3.6 Quality: The resin, as received by purchaser, shall be uniform in quality and condition, clean, and free from foreign materials insoluble in N-methylpyrrolidinone (N-methylpyrrolidone), and free from contaminants detrimental to usage of the resin.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection: The vendor of resin shall supply all samples for vendor's tests and shall be responsible for performing all required tests. Results of such tests shall be reported to the purchaser as required by 4.6. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the resin conforms to the requirements of this specification.

4.2 Classification of Tests:

4.2.1 Acceptance Tests: Tests of uncured resin to determine conformance to requirements for softening temperature (3.3.1), aromatic C-H/acetylenic C-H ratio (3.3.2), solubility (3.3.3), and infrared spectrum (3.3.5) and of cured resin to determine conformance to specific gravity (3.4.1) requirements are classified as acceptance tests and shall be performed on each lot.

4.2.2 Periodic Tests: Tests of uncured resin to determine conformance to requirements for gel time (3.3.4) and of cured resin to determine conformance to requirements for hardness (3.4.2), tensile strength (3.4.3), elongation (3.4.4), secant modulus (3.4.5), and dielectric constant (3.4.9) are classified as periodic tests and shall be performed at a frequency selected by the vendor unless frequency of testing is specified by purchaser.

4.2.3 Preproduction Tests: Tests to determine conformance to all technical requirements of this specification are classified as preproduction tests and shall be performed prior to or on the first shipment of resin to a purchaser, when a change in material, processing, or both requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

4.2.3.1 For direct U.S. Military procurement, substantiating test data and, when specified, preproduction test material shall be submitted to the cognizant agency as directed by the procuring activity, the contracting officer, or the request for procurement.

4.3 Sampling: Shall be as follows:

- 4.3.1 For Acceptance Tests: Sufficient resin shall be taken at random from each lot to perform all required tests. The number of determinations for each requirement shall be as specified in the applicable test procedure or, if not specified therein, not less than three.
- 4.3.1.1 A lot shall be all resin produced in a single production run from the same batches of raw materials under the same fixed conditions and presented for vendor's inspection at one time. An inspection lot shall not exceed 500 lb (225 kg). A lot may be packaged in small quantities under the basic lot approval provided lot identification is maintained.
- 4.3.1.2 A batch shall be the quantity of material run in a reactor or mixer at one time.
- 4.3.2 For Periodic Tests and Preproduction Tests: As agreed upon by purchaser and vendor.
- 4.4 Approval:
- 4.4.1 Sample resin shall be approved by purchaser before resin for production use is supplied, unless such approval be waived by purchaser. Results of tests on production resin shall be essentially equivalent to those on the approved sample.
- 4.4.2 Vendor shall use ingredients, manufacturing procedures, processes, and methods of inspection on production resin which are essentially the same as those used on the approved sample resin. If necessary to make any change in ingredients, in type of equipment for processing, or in manufacturing procedures, vendor shall submit for reapproval a statement of the proposed changes in material, processing, or both and, when requested, sample resin. Production resin made by the revised procedure shall not be shipped prior to receipt of reapproval.
- 4.5 Test Methods:
- 4.5.1 Uncured Resin:
- 4.5.1.1 Softening Temperature: Shall be determined by heating approximately 0.5 g of powdered resin between glass plates (microslides) under slight pressure on a suitable hot-plate melting-point apparatus. Heat sample quickly to 150°C (300°F), then continue heating at a rate of 2°C (4°F) per minute. The temperature at the first visible evidence of melting shall be recorded as the softening temperature.
- 4.5.1.2 Aromatic C-H to Acetylenic C-H Ratio: Shall be determined by using a suitable nuclear magnetic resonance spectrometer (See 8.2). Tests shall be conducted at 20° - 30°C (70° - 85°F) using 60 MHz in accordance with the spectrometer manufacturer's instructions. The resin solution shall be prepared by dissolving approximately 0.15 g of dry powdered resin in approximately 0.5 mL of deuterated dimethylsulfoxide at 125°C + 5 (255°F + 9). After cooling to room temperature, one drop of tetramethylsilane shall be added to the solution.

- 4.5.1.3 Solubility in N-methylpyrrolidinone (N-methylpyrrolidone): Shall be determined by stirring together at $25^{\circ}\text{C} + 5$ ($77^{\circ}\text{F} + 9$) a mixture of $3.0\text{ g} + 0.2$ of powdered resin and $20\text{ mL} \pm 1$ of N-methylpyrrolidinone (N-methylpyrrolidone). Complete solution should be achieved.
- 4.5.1.4 Gel Time: Shall be determined by placing $0.15 - 0.20\text{ g}$ of the powdered resin on a hot plate preheated to, and maintained at, $250^{\circ}\text{C} + 5$ ($482^{\circ}\text{F} + 9$). When a dark, glazed, liquid melt is observed, timing shall be started using a timer. The melt shall be touched continuously with a spatula until the melt has changed to a product having a crepe rubber consistency. When this is observed, the timing shall be stopped and the lapsed time shall be designated as the gel time.
- 4.5.1.5 Infrared Spectrogram:
- 4.5.1.5.1 An uncured resin pellet shall be prepared by thoroughly mixing dry, powdered, uncured resin (10% by weight) in dry, powdered, anhydrous potassium bromide and pressing the mixture into a small pellet to withstand handling and mounting on the spectrophotometer.
- 4.5.1.5.2 An infrared transmission spectrogram of the pellet shall be prepared covering wave lengths from 2.5 to 16 microns or 4000 to 625 reciprocal centimetres on uncured resin pellets of sufficient thickness to produce major spectral bands with transmissions approximating 40%.
- 4.5.1.5.3 The infrared spectrogram of production resin shall be compared with that of the sample approved as in 4.4.1.
- 4.5.2 Cured, Unfilled Resin Specimen Panel Preparation: Test panels of suitable thickness (dielectric constant specimens shall be not less than 0.06 in. (1.5 mm) thick) and size to perform the specified tests shall be prepared by molding in a hydraulic press at a pressure of 1000 - 2000 psig or 6,900 - 14,000 kPag and a temperature of $250^{\circ} - 260^{\circ}\text{C}$ ($480^{\circ} - 500^{\circ}\text{F}$) for $2\text{ hr} + 0.1$. Subsequently, they shall be post-cured for $16\text{ hr} + 0.1$ at $315^{\circ}\text{C} \pm 5$ ($600^{\circ}\text{F} \pm 9$) and $6\text{ hr} \pm 0.1$ at $370^{\circ}\text{C} \pm 5$ ($700^{\circ}\text{F} \pm 9$).
- 4.5.3 Graphite-Fiber-Reinforced, Cured Resin Test Specimen Preparation: Test panels approximately 0.10 in. (2.5 mm) thick and not less than 4 in. (100 mm) square, or any reasonable size consistent with obtaining a good distribution of fiber direction, shall be prepared from a quantity of AMS 3892/1 graphite fibers impregnated with a 40 - 50% solution of uncured resin dissolved in hot N-methylpyrrolidinone (N-methylpyrrolidone) at $175^{\circ}\text{C} + 3$ ($345^{\circ}\text{F} + 5$), dried in a forced air circulating oven for $45\text{ min.} + 1$ at $175^{\circ}\text{C} + 3$ ($345^{\circ}\text{F} + 5$), and chopped into approximately 1 in. (25 mm) lengths. The chopped, impregnated fibers shall be placed in a steel mold coated with a suitable release agent and preheated to $220^{\circ}\text{C} + 3$ ($430^{\circ}\text{F} + 5$) and cured at a pressure of 1000 psig ± 10 (6900 kPag ± 69). The mold temperature shall be increased to $260^{\circ}\text{C} \pm 3$ ($500^{\circ}\text{F} + 5$) over a period of not less than 25 min. and allowed to remain at $260^{\circ}\text{C} + 3$ ($500^{\circ}\text{F} + 5$) for $2\text{ hr} + 0.1$. The fiber content of the test panel shall be $60\% \pm 3$, by weight. Flexural test specimens $0.10 \times 0.75 \times 3.0\text{ in.}$ ($2.5 \times 20 \times 75\text{ mm}$) shall be machined from the cooled, cured panels and progressively postcured by heating to $230^{\circ}\text{C} \pm 3$ ($465^{\circ}\text{F} \pm 5$), raising the