



AEROSPACE MATERIAL

Society of Automotive Engineers, Inc.
400 COMMONWEALTH DRIVE, WARRENDALE, PA. 15096

SPECIFICATION

AMS 3616

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Revised

RESIN, POLYIMIDE, LAMINATING AND MOLDING
High Temperature Resistant, 370°C or 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) at temperatures up to 315° C (600°F), moderate-term exposure (25 - 50 hr) at temperatures up to 370°C (700°F), or short-term exposure (1 - 2 hr) at temperatures 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 (AMS) shall apply. The applicable issue of other documents shall be as specified in AMS 2350.

2.1 SAE Publications: Available from Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096.

2.1.1 Aerospace Material Specifications:

AMS 2350 - Standards and Test Methods

AMS 3892/1 - Tow, Graphite Fibers, For Structural Composites, GT 350 (2413) Tensile Strength, 30 000 000 (207) Tensile Modulus

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 Dielectric Constant (Permittivity) of Solid Electrical Insulating Materials

ASTM D638 - Tensile Properties of Plastics

ASTM D695 - Compressive Properties of Rigid Plastics

ASTM D790 - Flexural Properties of Plastics

ASTM D792 - Specific Gravity and Density of Plastics by Displacement

ASTM D2583 - Indentation Hardness of Plastics by Means of a Barcol Impressor

2.3 Government Publications: Available from Commanding Officer, Naval Publications and Forms 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.

SAE Technical Board rules provide that: "All technical reports, including standards approved by the Board, are advisory only. Their use by anyone engaged in industry or trade is entirely voluntary. There is no agreement to adhere to any SAE standard or recommended practice, and no commitment to conform to or be guided by any technical report. In formulating and approving technical reports, the Board and its committees will not investigate or consider patents which may apply to the subject matter. Prospective users of the report are responsible for protecting themselves against liability for infringement of patents."

- 3.2 **Storage Life:** The dry, solid resin, stored in vendor-packaged, air-tight containers at a temperature 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 a temperature 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 qualified test sample 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, and in accordance with specified test methods:
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|---|----------------------------|---|
| 3.4.1 Specific Gravity | 1.30 - 1.40 | ASTM D792 |
| 3.4.2 Hardness, Barcol | 50 ± 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
(3 448 MPa) | Same as 3.4.3 |
| 3.4.6 Flexural Strength, min | 13 000 psi
(90 MPa) | ASTM D790 |
| 3.4.7 Flexural Modulus, min | 500 000 psi
(3 448 MPa) | ASTM D790 |
| 3.4.8 Compressive Strength, min | 25 000 psi
(172 MPa) | ASTM D695 |
| 3.4.9 Dielectric Constant at
9.4 ± 0.1 GHz | 3.3 ± 0.2 | ASTM D150 |
| 3.4.10 Dissipation Factor at
9.4 ± 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 Flexural Properties:

	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 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 fabrication or to performance of parts.

4. QUALITY ASSURANCE PROVISIONS:

- 4.1 Responsibility for Inspection: The vendor of resin shall supply all samples 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 perform such confirmatory testing as he deems 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 softening temperature (3.3.1), aromatic C-H/acetylenic C-H ratio (3.3.2), solubility (3.3.3), and infrared spectrum when specified (3.3.5) requirements and of cured resin to determine conformance to specific gravity (3.4.1) requirements are classified as acceptance tests.
- 4.2.2 Periodic Tests: Tests of uncured resin to determine conformance to gel time (3.3.4) requirements and of cured resin to determine conformance to hardness (3.4.2), tensile strength (3.4.3), elongation (3.4.4), secant modulus (3.4.5), and dielectric constant (3.4.9) requirements are classified as periodic tests.
- 4.2.3 Qualification Tests: Tests to determine conformance to all technical requirements of this specification are classified as qualification tests and may be the basis for approval of the resin (See 4.4.1).

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

4.3 Sampling:

- 4.3.1 Acceptance Tests: Sufficient resin shall be taken at random from each lot to perform each test in triplicate.

- 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 submitted for vendor's inspection at one time.

- 4.3.1.2 A batch shall be the quantity of material run in a reactor or mixer at one time.

- 4.3.2 Periodic Tests: Frequency and extent of sampling shall be as agreed upon by purchaser and vendor.

4.3.3 Qualification 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. 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 any change is necessary 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 and processing 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 cm³ of deuterated dimethylsulfoxide at 125°C ± 5 (257°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°C ± 5 (77°F ± 9) mixture of 3.0 g ± 0.2 of powdered resin and 20 cm³ ± 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 g of the powdered resin on a hot plate preheated to, and maintained at, 250°C ± 5 (482°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 qualified test sample.