

# **AEROSPACE** MATERIAL Society of Automotive Engineers, Inc. SPECIFICATION

**AMS** 3686

9-15-75 Issued Revised

ADHESIVE, POLYIMIDE RESIN, FILM AND PASTE High Temperature Resistant, 315°C or 600°F

#### SCOPE:

400 COMMONWEALTH DRIVE, WARRENDALE, PA. 15096

- 1.1 Form: This specification covers a high-temperature, electrical-grade, polyimide resin adhesive in the form of film or paste.
- 1.2 Application: Primarily as an adhesive for bonding polyimide-laminate-faced Sandwich structures for use as radar-transparent assemblies. The adhesive is useful over the temperature range  $-55^{\circ}$  to  $+315^{\circ}$ C or  $-67^{\circ}$  to  $+600^{\circ}$ F.
- 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.
- 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

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

ASTM C297 - Tension Test of Flat Sandwich Constructions in Flatwise Plane ASTM D618 - Conditioning Plastics and Electrical Insulating Materials for Testing ASTM D1002 - Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal)

- Government Publications: Available from Commanding Officer, Naval Publications and Forms 2.3 Center, 5801 Tabor Avenue, Philadelphia, PA 19120.
- 2.3.1 Military Standards:

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

- 3. TECHNICAL REQUIREMENTS:
- Material: The adhesive shall be a polyimide resin system supplied in sheets, in rolls of film, or in paste form. The adhesive formulation shall be suitable for use in electrical applications and shall contain no metal fillers or other inorganic additives, except for anti-oxidants and thixotropic agents of not more than 35% by weight, total, based on cured resin solids.
- 3.1.1 Film Carrier: The carrier for film adhesive shall be "E" Glass Cloth (See 8.2).
- 3.1.2 Storage Life: Adhesive, stored in waterproof, sealed containers at a temperature not higher than -18°C ('0°F), shall meet the requirements of this specification when tested at any time up to 6 months from date of receipt by the purchaser.

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- 3.2 Properties of Uncured Adhesive: Shall be as follows; tests shall be performed on the product supplied and in accordance with specified test methods:
- 3.2.1 Solids Content of Paste Adhesive: Shall be the qualification value,  $\pm 3\%$ , determined in accordance with 4.5.1.1.
- 3.2.2 <u>Volatile Content of Film Adhesive</u>: Shall be the qualification value, <u>+</u>5%, determined in accordance with 4.5.1.2.
- 3.2.3 Weight of Film Adhesive: Shall be the qualification value,  $\pm 10\%$ , but weight shall not exceed 0.10 lb per sq ft or 0.50 kg/m<sup>2</sup>, including the glass cloth carrier, determined in accordance with 4.5.1.3.
- 2.3 Properties of Cured Adhesive: Shall be as specified in Table I, determined on specimens prepared in accordance with 4.5.2.1 and tested in accordance with specified test procedures; specimens to be tested at room temperature shall be conditioned and tested at standard conditions in accordance with ASTM D618.
- 3.4 Quality: The product shall be uniform in quality and condition, as free from foreign material as commercially practicable, and free from imperfections detrimental to fabrication, appearance, or performance of parts.
- 3.5 Sizes and Tolerances for Film Adhesive: Film thickness and corresponding weight in widths up to 48 in. (1.2 m) shall be as ordered. Individual rolls shall contain not more than 600 sq ft or 56 m<sup>2</sup>, unless otherwise ordered. The following tolerances shall apply:
- 3.5.1 Thickness: +10% measured to the nearest 0.001 in. or 0.03 mm.
- 3.5.2 Width:  $\pm 0.25$  in. or  $\pm 6.4$  mm.
- 4. QUALITY ASSURANCE PROVISIONS:
- 4.1 Responsibility for Inspection: The vendor of adhesive 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 adhesive conforms to the requirements of this specification.
- 4.2 Classification of Tests:
- 4.2.1 Acceptance Tests: Tests to determine conformance to solids content of paste adhesive (3.2.1) or to volatile content (3.2.2) and weight (3.2.3) of film adhesive and to tensile shear strength (Table I, Tests 1 and 2) requirements are classified as acceptance or routine control tests.
- 4.2.2 Qualification Tests: Tests to determine conformance to all technical requirements of this specification are classified as qualification or periodic control tests and may be the basis for approval of the adhesive (See 4.4.1).
- 4.2.2.1 For direct U.S. Military procurement, qualification test material and supporting test data shall be submitted to the cognizant qualification agency as directed by the request for procurement, the procuring activity, or the contracting officer.
- 4.3 <u>Sampling</u>: Sufficient adhesive shall be taken at random, after warming to above the dew point, from each lot to perform all required tests in triplicate or as specified in Table I. A lot shall be all adhesive 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.

## 4.4 Approval:

- 4.4.1 Sample adhesive shall be approved by purchaser before adhesive for production use is supplied, unless such approval be waived. Results of tests on production adhesive 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 adhesive which are essentially the same as those used on the approved sample adhesive. 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 materials and processing and, when requested, sample revised adhesive. No production adhesive made by the revised procedure shall be shipped prior to receipt of reapproval.

## 4.5 Test Methods:

## 4.5.1 For Uncured Adhesive:

4.5.1.1 Solids Content of Paste Adhesive: Shall be determined by heating approximately 3 g of as-received paste adhesive in an aluminum weighing dish in a forced draft oven at 315°C ± 5 (599°F ± 9) for 15 min. ± 1 and calculating the weight of solids remaining as a percentage of the initial sample weight.

# 4.5.1.2 Volatile Content of Film Adhesive:

- 4.5.1.2.1 Cut one 4.0-in. or 100-mm square specimen from each sample and punch a small hole in one corner. Weigh each specimen to the nearest 0.01 g (W<sub>1</sub>). Remove the protective film immediately prior to weighing.
- 4.5.1.2.2 Hang each specimen in a forced-draft oven maintained at 315°C ± 5 (599°F ± 9) for 15 min. ± 1.
- 4.5.1.2.3 Remove specimen from oven, cool to room temperature in a desiccator, and reweigh to the nearest 0.01 g (W2).
- 4.5.1.2.4 Calculate volatile content as follows:

Volatile Content 
$$w_1 - w_2 \le W_1 \le 100$$

where,  $W_1$  = original weight of specimen, g  $W_2$  = final weight of specimen, g

- 4.5.1.2.5 Report individual values and the average value for all specimens.
- 4.5.1.3 Weight of Film Adhesive: Shall be determined on specimens not less than 6 in. or 150 mm square with the protective separator removed immediately before weighing. The weight shall include the glass cloth carrier and shall be determined to the nearest 0.01 g. Report weight in 1b per sq ft or kg/m<sup>2</sup> of individual specimens and the average for all specimens from each lot.

#### 4.5.2 For Cured Adhesive:

#### 4.5.2.1 Preparation of Test Panels:

4.5.2.1.1 Metal Detail Parts: Tensile shear adherends shall be 0.064 in. or 1.60 mm thick aluminum alloy sheet, except that for tensile shear tests No.3 and No. 4 of Table I tensile shear adherends shall be 0.050 in. or 1.30 mm thick titanium alloy sheet. Honeycomb core for flatwise tensile strength tests shall be 5.7-3/16-20P(5052)EX, (minimum weight) or 91-4.8-0.05P(5052)EX, (See 8.2).

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- 4.5.2.1.2 Cleaning of metal detail specimens, priming of cleaned metal surfaces, and the application of adhesive, pressure, and curing schedule shall be in accordance with manufacturer's instructions or the applicable end item fabrication specification.
- 4.5.2.2 <u>Tensile Shear Tests:</u> Panels for tensile shear testing and specimens cut from panels shall be in accordance with ASTM D1002 and shall be conditioned in accordance with ASTM D618 and as specified in Table I. The number of specimens for each test shall be as shown in Table I.
- 4.5.2.3 <u>Flatwise Tensile Tests</u>: Test specimens (2 in. or 50 mm square) and test procedures shall be in accordance with ASTM C297 except that the honeycomb core shall be as specified in 4.5.2.1.1. The number of specimens for each test shall be as shown in Table I.
- 4.5.3 Conditioning: Environmental exposure conditions prior to testing shall be in accordance with ASTM D618 and as shown in Table I. Tests to determine resistance to water shall be performed within two hours of removal of the specimen from the conditioning.

#### 4.6 Reports:

- 4.6.1 The vendor of the product shall furnish with each shipment three copies of a report showing the results of tests made on the product to determine conformance to the acceptance test requirements and stating that the product conforms to the other technical requirements of this specification. This report shall include the purchase order number, material specification number, vendor's material designation, lot number, date of manufacture, thickness, weight, and quantity. Instruction sheets showing the recommended curing time, temperatures, and pressures for each lot of adhesive in the shipment shall also be supplied.
- 4.6.2 The vendor of finished or semi-finished parts shall furnish with each shipment three copies of a report showing the purchase order number, material specification number, contractor or other direct supplier of adhesive, supplier's material designation, part number, and quantity. When adhesive for making parts is produced or purchased by the parts vendor, that vendor shall inspect each lot of adhesive to determine conformance to the requirements of this specification, and shall include in the report a statement that the adhesive conforms, or shall include copies of laboratory reports showing the results of tests to determine conformance.
- 4.7 Resampling and Retesting: If any specimen used in the above tests fails to meet the specified requirements, disposition of the adhesive may be based on the results of testing three additional specimens for each original nonconforming specimen. Failure of any retest specimen to meet the specified requirements shall be cause for rejection of the adhesive represented and no additional testing shall be permitted. Results of all tests shall be reported.

#### 5. PREPARATION FOR DELIVERY:

#### 5.1 Packaging and Identification:

- 5.1.1 Film Adhesive: Shall be supplied in rolls with inside diameter of not less than 3.0 in. or 75 mm and interleaved with a colored, nonadhering separator-film in a manner to provide an outer wrapping with not less than 2.0 in. or 50 mm overlap. Each roll shall be heat sealed in a vapor-barrier bag. Sufficient desiccant shall be added to each bag before sealing, to maintain the dew point at a temperature below -18°C (0°F) for the specified storage life.
- 5.1.2 Paste Adhesive: Shall be packaged and sealed in wide-mouth containers not larger than 1 qt or 1 dm<sup>3</sup> and constructed of a material which is inert to the contents and the storage environment of -18°C (0°F).

5.1.3 <u>Identification</u>: Each bag and container shall be identified with the following information, using characters of such size as to be clearly legible and which will not be obliterated by normal handling:

ADHESIVE, POLYIMIDE, HIGH TEMPERATURE RESISTANT, 315°C or 600°F
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FORM (Film or Paste)
MANUFACTURER'S MATERIAL DESIGNATION
PURCHASE ORDER NUMBER
DATE OF MANUFACTURE
ROLL NUMBER (as applicable)
LOT NUMBER
QUANTITY
THICKNESS AND WEIGHT (for film adhesive)
APPROPRIATE WARNINGS OR PRECAUTIONARY NOTICES
PERISHABLE - STORE BELOW -18°C or 0°F

- 5.1.3.1 <u>Film Adhesive</u>: Shall be identified by marking on the protective separator film or by attached tags.
- 5.1.3.2 Paste Adhesive: Shall be identified on each container.
- 5.1.4 <u>Packing:</u> The protected rolls or containers shall be packed in an exterior container capable of protecting the product adequately during shipment and storage below the specified temperature.
- 5.1.5 Marking of Exterior Package: Each exterior shipping container shall be legibly marked with the following information in such a manner that the markings shall not smear or be obliterated during normal handling and use:

ADHESIVE, POLYIMIDE, HIGH TEMPERATURE RESISTANT, 315°C or 600°F

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FORM (Film or Paste)

PURCHASE ORDER NUMBER

MANUFACTURER'S MATERIAL DESIGNATION

DATE OF MANUFACTURE

LOT NUMBER

QUANTITY

APPROPRIATE WARNINGS OR PRECAUTIONARY NOTICES

PERISHABLE - STORE BELOW -18°C or 0°F

- 5.1.6 Containers shall be prepared for shipment in accordance with commercial practice to ensure carrier acceptance and safe transportation to the point of delivery. Packaging shall conform to carrier rules and regulations applicable to the mode of transportation.
- 5.1.7 For direct U.S. Military procurement, packaging shall be in accordance with MIL-STD-794, Level A or Level C as specified in the request for procurement. Commercial packaging as in 5.1.4, and 5.1.6 will be acceptable if it meets the requirements of Level C.
- 6. <u>ACKNOWLEDGMENT</u>: A vendor shall mention this specification number in all quotations and when acknowledging purchase orders.
- 7. <u>REJECTIONS</u>: Adhesive not conforming to this specification or authorized modification will be subject to rejection.
- 8. NOTES:

- 8.1 CAUTION! These materials may contain an arsenic compound. Ingestion, contact with the skin, and inhalation of vapors should be avoided. Handle only with rubber or plastic gloves. Bonding operations should be carried out in vacuum bags with exhaust vented away from working areas.
- 8.2 Materials for Test Specimen Preparation: Identities of materials used in the preparation of test specimens are as follows; several material specifications are shown for information;

Aluminum Alloy Sheet, 2024-T3: AMS 4037 or QQ-A-250/4 Cloth, "E" Glass: AMS 3824 or MIL-Y-1140 Honeycomb Core, 5052 Aluminum Alloy: AMS 4175 or MIL-C-7438 Titanium Alloy Sheet, 6A1-4V: AMS 4911 or MIL-T-9046, Type III, Comp. C

Ordering Data: For direct U.S. Military procurement, purchase documents should specify the following: 8.3

Title, number, and date of this specification
Thickness and width of film, or size of container for paste, desired
Quantity of adhesive desired
Applicable level of packaging (See 5.1.7).