INCH-POUND

MIL-PRF-46187A
14 August 1998
SUPERSEDING
MIL-P-46187
5 October 1987

PERFORMANCE SPECIFICATION

PREPREG, UNIDIRECTIONAL TAPE, CARBON (GRAPHITE) FIBER HIGH TEMPERATURE RESIN IMPREGNATED, 316°C (600°F)

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification establishes the requirements for carbon fiber/high temperature resin prepreg in the form of unidirectional tape suitable for the fabrication of structural laminates intended for use at temperatures up to and including 316°C (600°F) (see 6.1).

1.2 Classification of material.

1.2.1 High temperature resin. An organic resin either thermoset or thermoplastic which can withstand temperatures up to and including 316°C (600°F) while still retaining required mechanical, chemical and physical properties.

1.2.2 Prepreg. The carbon (graphite) fiber high temperature resin impregnated unidirectional tape conforming to this specification is termed a “prepreg” material (preimpregnated). The unidirectional prepreg tape described in this specification is a composite material in which the polyimide resin is partially cured and the carbon fiber reinforcement is oriented in one direction (unidirectional).

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Director, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, ATTN: AMSRL-WM-M, APG, MD 21005-5069 by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

2.2 Government documents.

2.2.1 Specifications, standards and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS) and supplement thereto, cited in the solicitation (see 6.2).

HANDBOOKS

DEPARTMENT OF DEFENSE

MIL-HDBK-728/6 - Ultrasonic Testing

(Unless otherwise indicated, copies of the above specifications, standards and handbooks are available from the Defense Automated Printing Service (DAPS), 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D695	Standard Test Method for Compressive Properties of Rigid Plastics
ASTM D790	Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials
ASTM D1898	Standard Practice for Sampling of Plastics
ASTM D2344	Standard Test Method for Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short-Beam Method
ASTM D3039/D3039M	Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials
ASTM D3410/D3410M	Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials with Unsupported Gage Section by Shear Loading
ASTM D3800	Standard Test Method for Density of High-Modulus Fibers
ASTM D4018	Standard Test Methods for Tensile Properties of Continuous Filament Carbon and Graphite Fiber Tows
ASTM D4102	Standard Test Method for Thermal Oxidative Resistance of Carbon Fibers

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

SOCIETY OF AUTOMOTIVE ENGINEERS, INC. (SAE)

AMS 3892/7	Tow or Yarn, Carbon Fibers For Structural Composites GF (OX) 400 (2758) Tensile Strength, 33 (228) Tensile Modulus

(Application for copies should be addressed to the Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.)

2.4 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 First article. When specified in the contract or purchase order, a sample shall be subjected to first article inspection (see 4.2.1 and 6.2).

3.2 High temperature resin. The organic resin either thermoset or thermoplastic shall withstand temperatures up to and including 316°C (600°F) while still retaining required mechanical, chemical and physical properties.

3.3 Fiber.

3.3.1 Fiber physical properties. The carbon fibers used in the prepreg shall conform to the minimum physical property requirements of AMS 3892/7 or equivalent with any deviations approved by the procuring activity. Both PAN-base and pitch-base carbon fibers shall be acceptable provided requirements of this specification are met.

3.3.2 Fiber test methods. The ASTM standards D3800, D4018 and D4102 (or approved alternates) shall be used for determining the properties of carbon fibers as specified in AMS 3892/7. Alternate sampling plans and test methods, as agreed upon, shall be used by the fiber supplier in lieu of AMS 3892/7 sampling plans and test methods.

3.3.3 Fiber sizing. Fiber sizing (or finish) shall be compatible with high temperature resin as specified in the contract or purchase order.

3.4 Prepreg. The chemical and physical properties of the carbon fiber/high temperature resin unidirectional prepreg shall be furnished by the contractor.

3.5 Composite laminates.

3.5.1 Physical properties. Carbon fiber/high temperature resin composite physical properties shall be furnished by the contractor.

3.5.2 Mechanical properties. Cured laminate test specimens for determination of mechanical properties shall be ultrasonically “C” scanned prior to mechanical testing. Military Handbook 728/6 is a useful guide to ultrasonic testing. Carbon fiber/high temperature resin composite laminates shall conform to the requirements of table I and table II.

3.6 Shelf life. The product shall meet the requirements of this specification when tested at any time up to 6 months from the date of shipment provided the material has been stored in a heat-sealed vapor barrier bag compatible with thermoplastic or uncured thermoset materials at manufacturer recommended temperatures.

3.7 Prepreg workmanship criteria. The prepreg material shall have a maximum gap width of 0.030 inches. The maximum continuous gap length shall not exceed one

foot. The tows shall have no waves greater than 0.030 inches wide. Tow splices shall be considered a defect. Crimps, cured resin, cured matter, resin-starved areas, wrinkles, and foreign materials are not allowed. Tape shall be parallel within ± 0.5 degrees. Filaments that are not wetted shall be 1 per cent maximum in any 144 square inches (0.093 square meters).

3.8 Identification marking. The manufacturer's code (part) number, contract number, serial number, date of manufacturer, national stock number, type of material, and the number of this specification shall be permanently marked in the location specified by the procuring activity (see 6.2).

4. VERIFICATION

4.1 Verification alternatives. Alternative test methods, techniques, or equipment, including the application of statistical process control, tool control, or cost effective sampling procedures may be proposed by the contractor. Acceptable alternative verification approaches shall be identified in the contract.

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

a. First article inspection (see 4.2.1)
b. Conformance inspection (see 4.2.2)

4.2.1 First article inspection. When required (see 3.1), the first article sample shall be examined for compliance with the requirements and verifications listed in table I and table II. All samples shall be produced with materials and processes proposed for use on the fabrication of structural laminates. Inspection shall be carried out by the contractor under Government surveillance, when specified (see 6.2).

4.2.2 Conformance inspection. Conformance inspection for the acceptance of the fabrication of structural laminates shall be conducted on a recurring basis and include the examinations and tests specified herein (see 4.2.2.1 and 4.2.2.2).

4.2.2.1 Material characterization. When specified (see 6.2), the materials described in this specification shall be analyzed for quality control purposes by the following methods as applicable:

a. Aniline titration
b. Atomic absorption spectroscopy (AA)
c. Differential scanning calorimetry (DSC)
d. Dynamic mechanical analysis (DMA)
e. Gas chromatography (GC)
f. High performance liquid chromatography (HPLC)

These analyses shall be performed in accordance with material suppliers' procedures which are used for production quality control, or in accordance with users' procedures which are used for lot acceptance inspection of incoming materials. The exact procedures to be used shall be agreed upon by supplier and user.

4.2.2.2 Mechanical tests. When specified (see 6.2), mechanical tests for conformance shall consist of those tests listed in table I and table II.

4.2.2.3 Noncompliance. If a sample fails to pass conformance inspection, the manufacturer shall notify the procuring activity and the cognizant inspection activity of such failure, and take corrective action on the materials or processes that are subject to the same failure. Acceptance and shipment of the product shall be discontinued until corrective action, acceptable to the procuring activity has been taken. After the corrective action has been taken,

conformance inspection shall be repeated on additional sample units (all tests and examinations, or the test which the original sample failed, at the option of the procuring activity). Conformance inspections shall be reinstituted, however, final acceptance and shipment shall be withheld until the conformance inspection has shown that the corrective action was successful. In the event of failure after reinspection, information concerning the failure shall be furnished to the cognizant inspection activity and the procuring activity.

4.3 Sampling for inspection. Sampling for inspection shall be performed in accordance with the provisions set forth in ASTM D1898 or as otherwise specified.

4.4 Test methods. Testing of resins, fibers, prepregs and composite laminates for compliance with the requirements of this specification shall be performed in accordance with the applicable test methods described in this specification to the maximum extent practicable, or as otherwise specified by the procuring activity.

4.5 Identification marking. Verify the presence of the required markings.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. The composite material specified herein is military unique because it is the only proven material used in aging aircraft replacement parts. This composite has been increasingly accepted as an engineering material for the design and fabrication of aerospace structural components, particularly aeropropulsion structural components. Material procured in accordance with this specification is intended for use in airframe, aerospace, and similarly related primary or secondary load-bearing structures where high stiffness and strength-to-weight ratios are required. Historically PMR-15 resin has been employed but comparable resin systems may be utilized. Polyimide (PMR-15) composite material has been extensively characterized in the NASA Composites for Advanced Space Transportation Systems (CASTS) Program, and is used in aircraft engine hardware such as cowls, nozzle flaps, and ducts.

6.1.1 Design allowables. A design allowables test program was conducted on graphite/polyimide composites by NASA (NASA CR-165840) to establish material performance over a 116°K (-250°F) to 589°K (600°F) temperature range. Effects of aging, thermal cycling and moisture were also evaluated. Tension, compression and in-plane shear properties were determined for uniaxial, pseudoisotropic and $\pm$ 45 degree laminates. Test results show sufficient strength and stiffness to substantiate graphite/polyimide composites as an acceptable structural material for high temperature structural applications.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number, and date of the specification.
b. Classification of material (see 1.2).
c. Issue of DoDISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.2 and 2.3).
d. When first article is required (see 3.1).
e. Specify location of identification markings (see 3.8).
f. Whether inspection is carried out by the contractor under Government surveillance (see 4.2.1).
g. Conformance inspection (see 4.2.2.1 and 4.2.2.2).
h. Packaging requirements (see 5.1).

6.3 Definitions.

6.3.1 Contractor. The term “contractor” is defined as the organization having a direct contract with the procuring activity.

6.3.2 Manufacturer. The term “manufacturer” is defined as the organization actually performing the operations covered by this specification.

6.3.3 PMR-15. The designation PMR-15 stands for a high temperature resistant polyimide resin system (designated PMR, for polymerization of monomer reactants) developed at NASA-Lewis Research Center. The imide oligomer before curing has a nominal molecular weight of 1500, which gives rise to the designation PMR-15.

6.4 Safety.

6.4.1 Precautions.

a. Some of the materials employed herein are flammable and toxic. Consult the responsible safety and industrial hygiene and security and fire protection organization concerning appropriate facilities, equipment, ventilation, and other requirements for safe operation. For disposal of hazardous waste materials, contact the responsible pollution control monitor for appropriate procedures.

b. Uncured material may cause skin sensitization or other allergic responses. Avoid inhalation of vapor. Use adequate ventilation, particularly if heated or sprayed. Prevent all contact with skin. Do not handle prepregs with bare hands. Use protective gloves such as Viton or equivalent protective coverings for hands and arms. If contact occurs, wash immediately with soap and water.

6.4.2 Safety data. A material safety data sheet conforming to AMS 2825, “Material Safety Data Sheets”, may be furnished to the procuring activity by the supplier prior to, or concurrent with, the initial shipment of material. If the material formulation is subsequently changed within the constraints permitted by this specification, a revised data sheet may be supplied prior to, or concurrent with, the first shipment of material with the revised formulation. Material safety data sheets may be identified with this specification number.

6.5 Subject term (key word) listing.

Laminates
Polyimide
Thermoplastic
Thermoset

Custodians:
Army - MR
Navy - AS
Air Force - 11

Review activities:
Army - AT, AV, IE
Navy - SH
Air Force - 13
DLA - DH

Preparing activity:
Army - MR

(Project CMPS-0161)

TABLE I. Laminate mechanical properties (English units).

	Test Temperature	Minimum Requirements Test Direction 0°	90°	Test Method
Tensile strength ultimate, ksi	RT	185	3.4	ASTM D3039
	550°F	185	1.6	
Tensile modulus, msi	RT	17	1.1	ASTM D3039
	550°F	17	0.82	
Compressive strength ultimate, ksi	RT	150	18.5	ASTM D3410 or ASTM D695
	550°F	85	12.0	
Compressive modulus, msi	RT	18	1.03	ASTM D3410 or ASTM D695
	550°F	18	0.92	
Compressive strength Aged 100 hours at 316°C, ksi	RT	135		ASTM D3410 or ASTM D695
	550°F	78		
Flexural strength, ksi	RT	235		ASTM D790
	500°F	157		
	600°F	157		
Flexural modulus, msi	RT	16		ASTM D790
	500°F	16		
	600°F	16		
Short beam shear strength, ksi	RT	18.8		ASTM D2344
	500°F	11.0		
	600°F	7.8		

TABLE II. Laminate mechanical properties (SI units).

	Test Temperature	Minimum Requirements Test Direction 0°	Minimum Requirements Test Direction 90°	Test Method
Tensile strength ultimate, MPa	RT	1275	23.4	ASTM D3039
	290°C	1275	11.0	
Tensile modulus, GPa	RT	120	7.6	ASTM D3039
	290°C	120	5.7	
Compressive strength ultimate, MPa	RT	1035	127.6	ASTM D3410 or ASTM D695
	290°C	585	82.7	
Compressive modulus, GPa	RT	125	7.10	ASTM D3410 or ASTM D695
	290°C	125	6.34	
Compressive strength Aged 100 hours at 316°C, MPa	RT	930		ASTM D3410 or ASTM D695
	290°C	540		
Flexural strength, MPa	RT	1620		ASTM D790
	260°C	1082		
	316°C	1082		
Flexural modulus, GPa	RT	110		ASTM D790
	260°C	110		
	316°C	110		
Short beam shear strength, MPa	RT	129		ASTM D2344
	260°C	75		
	316°C	54		

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.

2. The submitter of this form must complete blocks 4, 5, 6, and 7.

3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:	**1. DOCUMENT NUMBER** MIL-PRF-46187A	**2. DOCUMENT DATE (YYMMDD)** 980814

3. DOCUMENT TITLE PREPREG, UNIDIRECTIONAL TAPE, CARBON (GRAPHITE) FIBER HIGH TEMPERATURE RESIN IMPREGNATED, 316 DEG C. (600 DEG F.)

4. NATURE OF CHANGE *(Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)*

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME *(Last, First, Middle Initial)*	b. ORGANIZATION	
c. ADDRESS *(Include Zip Code)*	d. TELEPHONE (Include Area Code) (1) Commercial (2) AUTOVON *(if applicable)*	**7.DATE SUBMITTED** (YYMMDD)

8. PREPARING ACTIVITY

a. NAME US ARMY RESEARCH LABORATORY	b. TELEPHONE *Include Area Code)* (1) Commercial (410)306-0725 (2) AUTOVON 458-0725
c. ADDRESS *(Include Zip Code)Commander* WEAPONS & MATERIALS RESEARCH DIRECTORIATE ATTN; AMSRL-WM-M ABERDEEN PROVING GROUND, MD 21005-5069	**IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT:** DEFENSE QUALITY AND STANDARDIZATION OFFICE 5203 Leesburg Pike, Suite 1403, Falls Church, VA 22401-3466 Telephone (703) 756-2340 AUTOVON 289-2340