INCH-POUND

MIL-PRF-46190A <u>22 May 1997</u> SUPERSEDING MIL-P-46190 18 September 1990

PERFORMANCE SPECIFICATION

PREPREGS, WOVEN FABRIC, CARBON FIBER, RESIN IMPREGNATED

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

1. SCOPE

1.1 <u>Scope</u>. This specification establishes the requirements for resin impregnated, high strength, carbon fiber, woven fabric prepress (see 6.1).

1.2 Classification. Prepregs to be of the class specified (see 6.2):

Class A Tow Mechanical properties:

Ultimate tensile strength - 500 thousand pounds per square inch (ksi) (3450 MPa)

Ultimate tensile strain - 1.40 percent

Modulus of elasticity - 32-35 million pounds per square inch (Msi) (220-241 GPa)

Class B Tow mechanical properties:

Ultimate tensile strength - 650 thousand pounds per square inch(ksi) (4481 MPa)

Ultimate tensile strain - 1.40 percent

Modulus of elasticity - 39-42 million pounds per square inch (Msi) (269-289 GPa)

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.

AMSC N/A

AREA CMPS

2. APPLICABLE DOCUMENTS

2.1 <u>General</u>. 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.

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).

HANDBOOK

DEPARTMENT OF DEFENSE

MIL-HDBK-17 - Polymer Matrix Composites, Volume 1 - Guidelines For Characterization Of Structural Materials

(Unless otherwise indicated, copies of the above specifications, standards, and handbooks are available from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094).

2.3 <u>Non-Government publications</u>. 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 C613	-	Standard Test Method for Resin Content of Carbon and Graphite Prepregs by Solvent Extraction
ASTM D123	-	Standard Terminology Relating to Textiles
ASTM D329	-	Standard Specification for Acetone
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 D150	5 -	Standard Test Method for Density of Plastics by the Density-Gradient Technique
ASTM D2344	1 –	Standard Test Method for Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short-Beam Method

ASTM D2734	-	Standard Test Methods for Void Content of Reinforced Plastics
ASTM D3039/ D3039M	-	Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials
ASTM D3379	-	Standard Test Method for Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials
ASTM D3410/ D3410M	-	Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials with Unsupported Gage Section by Shear Loading
ASTM D3531	-	Standard Test Method for Resin Flow of Carbon Fiber-Epoxy Prepreg
ASTM D3532	-	Standard Test Method for Gel Time of Carbon Fiber-Epoxy Prepreg
ASTM D3775	-	Standard Test Method for Fabric Count of Woven Fabric
ASTM D3776	-	Standard Test Methods for Mass Per Unit Area (Weight) of Woven Fabric
ASTM D3800	-	Standard Test Method for Density of High-Modulus Fibers
ASTM D3990	-	Standard Terminology Relating to Fabric Defects
ASTM D4018	-	Standard Test Methods for Tensile Properties of Continuous Filament Carbon and Graphite Fiber Tows
ASTM D4019	-	Standard Test Method for Moisture in Plastics by Coulometric Regeneration of Phosphorus Pentoxide

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

SOCIETY OF AUTOMOTIVE ENGINEERS (SAE)

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- AMS 2825 Material Safety Data Sheets
- AMS 3892 Fibers, Carbon, Tow and Yarn for Structural Composites
- AMS 3892/1 Tow or Yarn, Carbon Fibers for Structural Composites GF 400 (2758) Tensile Strength, 33 (228) Tensile Modulus

(Application for copies should be addressed to Aerospace Material Specifications, 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 <u>First article</u>. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.2.1.

3.2 <u>Recycled, recovered, or environmentally preferable materials</u>. Recycled, recovered, or environmentally preferable materials should be used to the maximum extent possible provided the material meets or exceeds all specified requirements and promotes economically advantageous life cycle costs.

3.3 General material requirements.

3.3.1 <u>Formulation</u>. The contractor shall select the materials, but the materials shall be capable of meeting all the requirements specified herein. A material consisting of carbon tows that have been woven and impregnated with a resin is recommended.

3.3.2 <u>Fiber</u>. The carbon fiber shall comply with the property requirements in table I.

3.3.3 <u>Fiber sizing</u>. The fiber shall be treated to be compatible with the resin in accordance with 4.3.3.

3.4 Woven fabric.

3.4.1 <u>Physical properties</u>. The woven fabric shall conform to the physical properties in table II.

3.4.2 <u>Selvage</u>. The woven fabric shall have selvage edges. The procuring activity shall state the selvage width.

3.4.3 <u>Tracer strands</u>. An x-ray detectable glass tracer yarn shall be placed on 2.0 \pm 0.25 inch centers in the warp direction and 6 \pm 1 inch centers in the weft direction in each ply of material. The tracer yards shall be AR/CK 32.5 I/O, 1 ounce, size 460 white x-ray detectable glass yarn manufactured by Owens Corning Fiberglas Company (Toledo, OH), or an approved equivalent. The tracer yarn materials shall not outgas during processing of the prepreg, or be the cause of thermal microcracking in the composite (see 6.4).

3.4.4 <u>Fabric uniformity</u>. Fabric used for impregnation with the resin shall be produced only after all necessary adjustments have been made on the weaving equipment to provide a uniform product conforming to the requirements of this specification. Nonuniform areas of the fabric at the start or end of the weaving operation shall be removed prior to packaging. The fabric shall be clean and free from creases, cuts, tears, smash or other damage to two or more adjacent warp or fill yarns. Anomalies affecting a single yarn including missing end, mispick, whipped-in filling, loose pick (whether or not resulting

in a loop), slub, broken pick, hang pick, kink and fuzz ball shall not be considered as nonconforming areas provided no more than three such anomalies occur in any linear yard (meter) or 1 meter by 0.2 meter area with no anomaly closer than 6 inches (152 mm) to another anomaly. Anomalies in the cosmetic appearance of the woven fabric which are a result of splices in the yarn shall not be considered as nonconforming areas. A float in the warp direction identified as a result of a single missing end and less than two inches (51 mm) in length shall not be considered as a nonconforming area. A float more than two inches in length or resulting from two adjacent missing ends shall be considered as a nonconforming area.

3.4.5 <u>Materials compatibility</u>. Materials, if any, used on the carbon filaments or yarns or techniques utilized in conjunction with the weaving operation for purposes of assisting the weaving operation shall not interact with or degrade the filament, resin, impregnated fabric or resulting cured and uncured properties, from those specified herein. Such materials or techniques, if used, shall not be changed after first article inspection of the product without written approval by the procuring activity.

3.5 Prepreg.

3.5.1 <u>Requirements</u>. The uncured prepreg material shall meet the physical property requirements of table III. Table IV is optional.

3.5.2 Width. Unless otherwise specified (see 6.2), the prepreg width shall be 1067 \pm 13 mm (42.0 \pm 0.5 inches). Selvage shall not be included in determining prepreg width.

3.5.3 <u>Alignment</u>. The alignment of the warp and fill yarns shall be perpendicular to each other and shall be parallel to the warp and fill directions of the impregnated fabric within 51 mm (2 inches) in any 1219 mm (48 inches) of fabric width or length. The supplier shall, as a minimum, determine the alignment at the start and end of each roll of impregnated fabric. Straightening of impregnated fabric is not permitted when measuring alignment. A tenter frame to correct fiber alignment in impregnated fabric may be used.

3.5.4 <u>Interliner</u>. The prepreg interliner shall be 4 mil (0.1 mm) or greater thick and shall be 12.7 \pm 6.4 mm (0.50 \pm 0.25 inches) wider per side than the prepreg. Removal of the interliner paper or polyethylene film at ambient temperature shall be accomplished with ease and without damage to the prepreg.

3.5.5 <u>Flatness</u>. The prepreg shall contain no buckles or wrinkles. Some wrinkles, buckles and separations between prepreg and release film shall be allowed. The prepreg and its release film shall lay flat with the edges not raising more than 2.5 mm (0.1 inches) above a flat table. The prepreg shall lay flat when removed from the release film with no edge curl visually apparent.

3.5.6 <u>Characterization</u>. Prepreg physiochemical property requirements are to be determined (see table IV).

3.5.7 <u>Storage</u>. The prepreg shall meet the requirements of this specification when tested at any time up to one year from the date of manufacture provided it has been stored at temperatures not exceeding manufacturer's recommendations.

3.6 Composite laminates (properties of cured laminates).

3.6.1 <u>Cure cycles</u>. Cure and postcure cycle procedures, which include layup/bagging procedures and time-temperature-pressure conditions for the fabrication of composite laminates conforming to this specification, shall be specified in a process specification.

3.6.2 <u>Physical properties</u>. The cured woven fabric laminates shall conform to the physical properties of table V.

3.6.3 <u>Mechanical properties</u>. After 21 days (504 hours) at the above conditions the prepreg shall be cured in laminates meeting the mechanical property requirements of table VI.

3.7 <u>Rejection</u>. Carbon fiber woven fabric prepreg not conforming to the requirements of this specification shall be rejected.

3.8 <u>Fluid resistance</u>. When immersed in the fluids specified in table VII for the exposure times and temperatures specified, the cured composite material shall meet the requirements for short beam shear as specified in the applicable specification sheet. In addition, the laminates shall show no visible degradation or damage (i.e. dissolution, gelation, or swelling). Contact by the cured laminate material shall not alter the properties of the fluids as required by their respective specifications.

4. VERIFICATION

4.1 <u>Verification alternatives</u>. 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 <u>Classification of inspections</u>. 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. First article inspection shall be performed on the first production-representative samples of an order when a first article sample is required (see 3.1). The first article sample(s) shall be examined for all the provisions specified by the procuring activity, the contract or the purchase order (see 6.2). This inspection shall consist of the tests specified in tables I, II, III, V, VI, and VII. Table IV is optional.

4.2.2 <u>Conformance inspection</u>. Conformance inspection for acceptance of resin impregnated, high strength, carbon fiber, woven fabric prepregs shall meet all

the provisions specified by the procuring activity, the contract or the purchase order (see 6.2).

4.3 General material requirements.

4.3.1 <u>Formulation</u>. The material shall be visually examined for conformance to paragraph 3.3.1.

4.3.2 Fiber. The carbon fiber property requirements shall be determined in accordance with the test methods in table I.

4.3.3 <u>Fiber sizing</u>. The standard test method for Resin Flow of Carbon Fiber-Epoxy Prepreg, ASTM D3531, shall be used.

4.4 Woven fabric.

4.4.1 <u>Physical properties</u>. The woven fabric physical properties shall be determined in accordance with the test methods in table II.

4.4.2 <u>Fabric characteristics</u>. The woven fabric shall be visually examined for conformance to paragraph 3.4.2 through 3.4.5.

4.5 Prepreg.

4.5.1 <u>Requirements</u>. The uncured prepreg property requirements shall be determined in accordance with the test methods specified in table III.

4.5.2 <u>Dimensional inspection</u>. The prepreg material shall be visually examined for conformance to paragraph 3.5.2 through 3.5.5.

4.5.3 <u>Characterization</u>. Guidance in performing the test methods in table IV may be found in MIL-HDBK-17, Polymer Matrix Composites, Volume 1 - Guidelines For Characterization of Structural Materials. Guidance for one of these test methods is also provided in paragraph 4.5.4.

4.5.4 <u>Rheometric viscosity analysis</u>. The viscosity profile of the prepreg shall be determined using a rheometric dynamic spectrometer (Model RDS-7700 or equivalent). Instrument parameters and test conditions shall be as follows:

- a. Sample plates 25 mm radius
- b. Gap adjusted to accommodate a three prepreg layer of thickness
- c. Starting temperature 50°C (122°F)
- d. Strain 5%
- e. Frequency 10 rads/sec
- f. Heating rate 2°C/min

A rheogram shall be prepared by plotting viscosity versus time to an end point beyond gelation. Gelation is defined as the time/temperature where the storage modulus crosses the loss modulus during the steep viscosity rise.

4.5.5 <u>Drapeability</u>. The prepreg shall be exposed to 24°C (75°F) maximum temperature and a maximum of 50% relative humidity for 5 days (120 hours).

4.5.6 Storage. Examine the prepreg for compliance with the storage requirements specified in paragraph 3.5.7.

4.6 Composite laminates (properties of cured laminates).

4.6.1 <u>Cure cycles</u>. Verify the cure and postcure cycle procedures for the fabrication of composite laminates conforming to this specification.

4.6.2 <u>Physical properties</u>. The cured laminate physical property requirements shall be determined in accordance with the test methods specified in table V.

4.6.3 <u>Mechanical properties</u>. The cured laminate mechanical property requirements shall be determined in accordance with the test methods specified in table VI.

4.7 <u>Rejection</u>. Examine the carbon fiber woven fabric prepreg for compliance with the requirements of this specification.

4.8 <u>Fluid resistance</u>. Examine the cured composite material for compliance with the requirements specified in paragraph 3.8.

5. PACKAGING

5.1 <u>Packaging</u>. 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 carbon fiber woven fabric prepregs covered by this specification are intended for the fabrication of high performance composite structures which are capable of withstanding a temperature environment of -54 to 204°C (-65 to 400°F). Carbon fiber bismaleimide has been used to comply with the requirements of this specification.

6.2 <u>Acquisition requirements</u>. Acquisition documents should specify the following:

- a. Title, number and date of this specification.
- b. Class of prepreg, A or B (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).

d. When first article is required (see 3.1).

- e. Fabric width (see 3.5.2).
- f. Packaging requirements (see 5.1).
- g. Dimensions in SI and English units.

6.3 Definitions. See ASTM D123 and ASTM D3990.

6.4 <u>Prepreg without tracer yarns</u>. Due to the large difference between longitudinal and transverse thermal expansion of carbon fibers, thermal cycling can result in matrix cracking. For this reason, some applications, depending on thermal environment, may require the use of prepreg without tracers.

6.5 Safety.

6.5.1 <u>Precautions</u>. Uncured material may cause skin sensitization or other allergic responses. Adequate ventilation should be used and all contact with skin should be prevented. Established precautions for handling resins and fine fibrous materials should be observed. Operators working with this product should wear clean impervious gloves to reduce the possibility of skin contact, and also to prevent contamination of the material.

6.5.2 <u>Safety data</u>. A material safety data sheet conforming to AMS 2825 should be furnished to the purchaser 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 should be supplied prior to, or concurrent with, the first shipment of material with the revised formulation. A material safety data sheet should be identified with this specification number.

6.6 Subject term (key word) listing.

Aircraft skin components Graphite fiber High temperature (400°F) material Polymer matrix composites

Army - MR Navy - AS Air Force - 11 Review activities: Army - AT, AV, IE Navy - SH Air Force - 13 DLA - DH

Custodians:

Preparing activity: Army - MR

Project No. CMPS-0135

TABLE I. Properties of carbon fibers. 1/ 6 2/

Property	Requirement	Test Methods
Tensile strength, min	3448 MPa (500 Ksi)	ASTM D3379 or ASTM D4018
Tensile modulus, min	227-242 GPa (33-35 Msi)	ASTM D3379 or ASTM D4018
Tensile failure strain, min	1.3%	ASTM D3379 or ASTM D4018
Density	$1.74 - 1.84 \text{ g/cm}^3$	ASTM D1505 or ASTM D3800
Diameter (nominal)	7 microns	As specified

 $\frac{1}{2}$ Perform all tests at room temperature. $\frac{2}{2}$ See AMS 3892

TABLE II.	Physical	properties	of woven	fabric.

Type of	Fabric count <u>1</u> / W X F	Fabric areal weight <u>2</u> /
woven fabric	(yarn count/inch, 25 mm)	(g/m ²)
5HS	12.5 ± 0.5 × 12.5 ± 0.5	370 ± 14

 $\frac{1}{2}$ / Test per ASTM D3776

TABLE III. Physical properties of uncured prepreg materials.

Property	Requirement	Test Method
Volatile content, weight % <u>1</u> / (with volatile reactants) (without volatile reactants)	5 ± 3% 2% max	ASTM C613
Drapeability	Shall bend 180° over 1/8 inch diameter mandrel with no evidence of filament damage when examined at 5X.	Paragraph 4.5.5
Resin flow	As specified	ASTM D3531
Gel time	As specified	ASTM D3532
Moisture content	0.4% max	ASTM D4019

1/ The volatile content may vary widely depending on whether the prepreg does or does not contain volatile reactive constituents. Moisture content testing (ASTM D4019) and gas chromatography (which separates, identifies, and quantifies volatile materials) are recommended options to volatile content testing.

Test Method	Requirement	Paragraph
Differential Scanning Calorimetry (DSC) :		4.5.3
a. Exotherm	TBD <u>1</u> /	
b. Onset Temperature	TBD	
c. Peak Temperature	TBD	
High Performance Liquid Chromatography (HPLC) :		4.5.3
a. Number of major components	TBD	
b. Retention time of components	TBD	
Fourier Transform Infrared Spectroscopy (FTIR)	Report only	4.5.3
Rheometric Viscosity Analysis :		4.5.4
a. Minimum viscosity	TBD	
b. Temperature at minimum viscosity	TBD	
c. Gelation temperature	TBD	

TABLE IV. Prepreg characterization. (optional)

1/ TBD = To be determined

TABLE V. Physical properties of cured woven fabric laminates.

Property	Requirement	Test Method
Void content, volume % (max)	1.5 to 2.0%	ASTM D2734
Glass transition temperature, Tg (wet), (min) $\frac{1}{2}$	As specified	As specified
Cured ply thickness	0.305 ± 0.102 mm (0.012 ± 0.004 in)	As specified

<u>1</u>/ It is recommended that Tg be determined by thermomechanical analysis (TMA) using an expansion probe and a heating rate of 10°C/minute with a 2 gram weight placed over the probe. Sample thickness to be 6 \pm 3 mm.

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Minimum Requirements <u>1</u> /					
Properties	Room Temperature Warp <u>7</u> / Fill		Elevated <u>6</u> / Warp <u>7</u> / Fill		
Tensile properties <u>2</u> / Strength, MPa (ksi)	517 (75)	483 (70)	483 (70)	449 (65)	
Modulus, GPA (Msi)	69 (10)	62 (9)	69 (10)	62 (9)	
Failure strain %	0.65	0.65	0.65	0.65	
Compressive properties <u>3</u> / Strength, ksi (MPa)	541 (62)	403 (58)	448 (65)	276 (40)	
Modulus, GPa (Msi)	62 (9)	49 (7.2)	62 (9) ·	49 (7.2)	
Failure strain, %	0.65	0.58	0.60	0.54	
Flexure properties <u>4</u> / Strength, MPa (ksi)	558 (81)	517 (75)	448 (65)	441 (64)	
Interlaminar shear properties, MPa (ksi) <u>5</u> /	41 (6)	29 (4.8)	31 (4.4) 25 (3.6) <u>6</u> /	26 (3.7) 25 (3.6) <u>6</u> /	

TABLE VI. Mechanical properties of cured woven fabric laminates.

1/ Test five specimens for each condition. Values listed are "S" basis allowables as defined in MIL-HDBK-17, Volume 1.

2/ Test per ASTM D3039.

3/ Test per ASTM D3410 or modified ASTM D695.

4/ Test per ASTM D790 (Span-to-thickness 32:1, 3-point loading). Report mode of failure.

5/ Test per ASTM D2344 (Span-to-thickness of 4:1). Report mode of failure.

- 6/ Coupons for hot-wet tests shall be conditioned in a temperature/humidity cabinet by exposure to 160°F (71°C) and 95-100% relative humidity until a constant weight deviation not greater than 0.05% has been attained for a minimum of 7 days. Test at 350°F (177°C) immediately upon attainment of equilibrium.
- 7/ Use warp data for plain weave cloth. Fill values to be the same.

TABLE VII. Fluid resistance.

Fluid	Specification	Immersion Time and Test Temperature <u>1</u> /
Turbine Fuel	Aviation	90 Days at 75°F (24°C)
Lubricating Oil	Aircraft turbine engine, authentic base with a kinematic viscosity of 4.0 ± 1.0 cs	90 Days at 180°F (84°C)
Anti-Icing Fluid	Deicing fluid (propylene glycol base) with a specific gravity of 1.103 ± 0.003 or ethylene glycol base with a specific gravity of 1.105 ± 0.	30 Days at 32°F (0°C) 005
Hydraulic Fluid	Synthetic Hydrocarbon	90 Days at 75°F (24°C)
Cleaning Fluid	Water dilutable for exterior surfaces of aircraft	7 Days at 75°F (24°C)
Hydraulic Fluid	Petroleum Base	90 Days at 75°F (24°C)
Acetone	ASTM D329	7 Days at 75°F (24°C)

1/ Temperature shall be as specified \pm 6°F (\pm 3°C).

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.

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Previous editions are obsolete.