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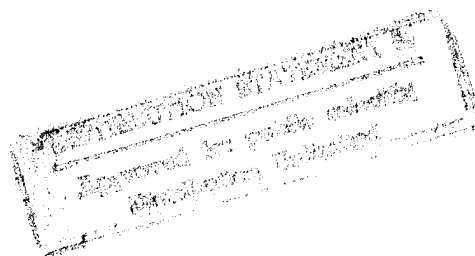
**NASA/Aircraft Industry
Standard Specification for
Graphite Fiber/Toughened
Thermoset Resin Composite
Material**



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Standard Specification for
Graphite Fiber/Toughened
Thermoset Resin Composite
Material**

Compiled by
ACEE Composites Project Office

*Langley Research Center
Hampton, Virginia*

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National Aeronautics
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FOREWORD

A standard specification for a selected class of graphite fiber/toughened thermoset resin matrix material has been developed through a joint NASA/Aircraft Industry effort involving technical personnel from the NASA Langley Research Center and from the three commercial transport producers: Boeing Commercial Airplane Company, Douglas Aircraft Company, and Lockheed-California Company. This standard specification has been compiled to provide uniform requirements and tests for qualifying prepreg systems and for acceptance of prepreg batches. Significant advantages are expected to accrue through the availability and use of the standard specification, both to the using aircraft industry and to the suppliers. Potential advantages to the users include multiple sources of suppliers, one material requirement, more uniform quality, greater availability, and lower costs. Potential advantages to suppliers include uniform testing, quality control, formulation, and processing and improved market opportunities.

The specification applies specifically to a class of composite prepreg consisting of unidirectional graphite fibers impregnated with a toughened thermoset resin that will produce laminates with service temperatures from -65°F to 200°F when cured at temperatures below or equal to 350°F. The specified prepreg has a fiber areal weight of 145 g/m². The specified tests are limited to those required to set minimum standards for the uncured prepreg and cured laminates and are not intended to provide design allowable properties. Qualification and subsequent use of a material through this specification does not constitute or imply endorsement by NASA.

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1 SCOPE

This specification establishes requirements for qualification and batch acceptance of a carbon fiber toughened thermoset resin prepreg that will provide a laminate with service temperature from -65°F to +200°F when cured in an autoclave at temperatures not higher than 350°F and at pressures no greater than 100 psi.

2 CLASSIFICATION

Prepreg materials shall be of the following types, classes, and grades.

2.1 Types

The types shall specify the prepreg nominal resin content.

Type 1 - Resin content 35 percent by weight

2.2 Classes

The class shall specify the prepreg form.

Class 1 - Unidirectional prepreg tape

2.3 Grades

The grades shall specify the areal weight of carbon fibers in grams per meter².

Grade fiber areal weight, g/m ²	Nominal cured ply thickness, in.
Grade 145	0.0056

3 APPLICABLE DOCUMENTS

3.1 ASTM Standards

Standard Recommended Practice for Inplane Shear Stress-Strain Response of Unidirectional Reinforced Plastics. ANSI/ASTM Designation: D 3518 - 76. Part 36 of 1981 Annual Book of ASTM Standards, c.1981, pp. 891-896.

Standard Test Methods of Load Verification of Testing Machines. ASTM Designation: E 4 - 79. Part 35 of 1982 Annual Book of ASTM Standards, c.1982, pp. 1056-1063.

Standard Test Methods for Void Content of Reinforced Plastics. ASTM Designation: D 2734 - 70 (Reapproved 1980). Part 36 of 1982 Annual Book of ASTM Standards, c.1982, pp. 570-573.

Standard Test Method for Compressive Properties of Rigid Plastics. ASTM Designation: D 695 - 80. Part 35 of 1982 Annual Book of ASTM Standards, c.1982, pp. 291-298.

3.2 Military Standards

Military Standard - Surface Roughness, Waviness and Lay. MIL-STD-10A, Jan. 3, 1966. (Supersedes MIL-STD-10, Aug. 2, 1949.)

3.3 NASA Documents

Chen, J. S.; and Hunter, A. B.: Development of Quality Assurance Methods for Epoxy Graphite Prepreg. NASA CR-3531, 1982.

ACEE Composites Project Office, compiler: Standard Tests for Toughened Resin Composites - Revised Edition. NASA RP-1092, 1983. (Supersedes NASA RP-1092, 1982.)

4 DEFINITION OF TERMS

a. Resin batch: Resin mixed with the same lots of ingredients in one continuous operation with traceability to individual component lots.

b. Prepreg batch: Prepreg containing one distinct yarn lot of graphite fiber reinforcements impregnated with one distinct batch of resin in one continuous operation.

c. Yarn or tow lot: The quantity of carbon fibers formed during a single production run having the same continuous process and identical characteristics throughout.

d. Prepreg lot: Prepreg from one batch submitted for acceptance at one time.

e. Storage life: The time in storage at 10°F or below, while contained in a moisture barrier bag made of 6 mil or thicker continuous polyethylene, during which the material maintains its handling life, processing life, and all other requirements of this specification.

f. Processing life: The out-of-refrigeration exposure time that the material can sustain and produce specification-acceptable mechanical properties in laminates when processed by the specified standard cure cycle.

g. Handling life: The out-of-refrigeration exposure time during which the material retains tack, forming, and draping characteristics.

h. Surface resin starvation: Incomplete resin filling of the prepreg surface.

i. Puckers: Areas on prepreg materials that are locally blistered or puckered from the separator film or release paper.

j. Fuzz balls: Balls of fibers that occur when individual filaments are abraded or broken during manufacture of the prepreg. These fibers collect as loose filament bundles or balls which are occasionally incorporated into the prepreg.

k. Roll: Originally produced prepreg roll before slitting.

l. Basic surface roughness symbol: $\sqrt{\quad}$ designates surface roughness.

5 REQUIREMENTS

5.1 Qualification Approval

The materials qualified under this specification shall have passed all qualification requirements specified herein. Qualification requirements shall include all those indicated in tables 1, 2, 3, and 4. After qualification approval, the properties and methods of manufacture shall not be changed without written approval from the buyer. The supplier shall conduct qualification tests on three batches of prepreg material. Suppliers shall provide two copies of all required qualification test results, including individual test values.

5.2 Acceptance Approval

The materials delivered under this specification shall have passed the acceptance tests specified in tables 2, 3, and 4.

5.2.1 Supplier Specifications

Suppliers shall submit their quality control specifications and obtain buyer approval for control of the equipment, processes, test procedures, and raw materials for the manufacture of the product. If necessary, the buyer will stipulate an agreement of secrecy to safeguard the interest of the supplier.

5.2.2 Acceptance Test Results

The supplier shall provide two copies of all required acceptance test results, including individual test values.

5.3 Component Requirements

5.3.1 Fiber

Specifications for graphite fibers used to produce prepreg follow.

5.3.1.1 Fiber classification.- The graphite fiber reinforcements shall be of the following filament count:

3000: shall be 3000 filaments per tow or yarn
6000: shall be 6000 filaments per tow or yarn
12 000: shall be 12 000 filaments per tow or yarn

5.3.1.2 Fiber splices.- The graphite fiber shall contain no more than one splice per pound of tow or yarn with a minimum spacing of 500 ft between splices. The length of any fiber splice shall be 1.50 ± 0.75 in.

5.3.1.3 Fiber requirements.- The graphite fiber reinforcement yarn or tow shall meet the requirements shown in table 1.

5.3.1.4 Fiber certification.- The prepreg supplier shall certify for each prepreg batch that the fiber reinforcement meets the requirements of table 1 and shall provide documentation upon request. The prepreg supplier shall provide a chromatogram of fiber sizing extracted from each yarn lot made by high pressure liquid chromatography (HPLC).

5.3.1.5 Fiber test methods.- Test methods shall be established by the prepreg supplier and documented in a material specification approved by the buyer.

5.3.2 Resin

The resin shall be a toughened thermoset polymer which shall meet the requirements of this specification. Resin processing temperatures shall be no higher than 350°F and processing pressures shall be no higher than 100 psig. Cured laminates made from this material shall be capable of performing continuously at temperatures from -65°F to +200°F.

5.3.3 Prepreg Requirements

Prepreg material shall be tested in accordance with the procedures specified in appendix A and shall meet the requirements specified in table 2.

5.3.3.1 Tape dimensions.- The length and width of the prepreg material shall be as specified by the buyer. The width tolerance shall be ± 0.040 in. over the full length of the roll. The weight of 12-in-wide prepreg material on all but a single roll in a batch shall be between 20 and 70 lb unless otherwise specified. The weight shall be proportionally less for narrower widths.

5.3.3.2 Carrier.- The prepreg shall have a moisture resistant carrier (60-lb kraft paper unless otherwise specified) suitable for use with automated dispensing equipment. The carrier shall be the same width as the prepreg tape ($+0.040$ to 0 in.) specified on the purchase order. The carrier may have a release coating. The carrier shall be easily removable from the material at ambient temperature without transfer of the release coating to the resin and without distortion of the fibers. The prepreg shall contact the carrier at all places on the roll. The carrier shall be on the outside of the roll.

5.3.3.3 Roll core configuration.- Each roll of prepreg shall be supported by a core that does not deform by the material weight. The core shall be supported during shipping and storage in such a way that the material will not be damaged by its own weight. The core inside diameter shall not be less than 8 in. The core length shall be 3 in. longer than the prepreg width.

5.3.3.4 Alignment.- The alignment of the collimated tows or yarns within the prepreg shall not deviate from the material centerline by more than 0.030 in. in a linear foot. The edge of the material shall not deviate from a reference straight line by more than 0.020 in. in any linear foot. The fibers must be flush with the edge of the carrier within 0.025 in.

5.3.3.5 Gaps.- Gaps are defined as the individual open spaces between adjacent, parallel tows or yarns that are 0.010 in. wide or greater. Gaps shall be no more than 0.020 in. wide and not more than 3 in. long. No more than one such gap shall appear in any 50 linear ft of prepreg.

5.3.3.6 Splices.- Yarn or tow splices shall overlap 1.50 ± 0.75 in. and their location shall be clearly indicated on the prepreg or carrier. Not more than one splice shall occur in any 48-in. length of prepreg.

5.3.3.7 Handling characteristics.- In the temperature range 65°F to 85°F, the material shall have the required tack to permit easy removal from the carrier without loss of resin, tearing, shredding, or otherwise becoming damaged. The material shall be capable of being cut without disarray of the filaments or other visible damage.

5.3.3.8 Workmanship.- The prepreg tape shall be free of foreign material, crossed or broken tows, broken tow splices, cured resin particles, fuzz balls, resin rich or starved areas, puckers, and wrinkles which cannot be smoothed out by hand pressure. The material shall be uniform in quality and condition.

5.3.3.9 Tagging of defective areas.- Prepreg rolls containing unacceptable defects shall be tagged on the roll so that the tags are clearly visible before unrolling. The supplier shall provide a linear listing of all defective areas, indicating the length of the defective area and footage at the start and end of the defective area. The supplier shall label the locations of all defects. Rolls containing more than 5 percent defective yardage shall be rejectable. The defective areas shall not be considered as deliverable quantity under the purchase order. Defective areas shall not be closer than 50 linear ft. Prepreg material may be cut to remove defects, but supplied prepreg shall be in lengths of not less than 50 ft. For prepreg supplied for automated layup, all defective material must be removed by cutting and splicing. Splices shall meet the requirement of section 5.3.3.6.

5.3.3.10 Effect of storage.-

1. Storage life shall be 6 months or longer from date of shipment when stored at 10°F or lower in the original sealed shipping package.
2. Handling life shall be 10 days or longer when exposed to room temperature, 80°F maximum in a closed container.
3. Processing life shall be 30 days or longer when exposed to room temperature, 80°F maximum on a tooling surface.

5.3.3.11 Cure.- Material supplied to this specification shall be capable of being cured with the bagging procedure shown in figure 1 and the standard cure cycle specified in figure 2.

5.3.4 Laminate Properties

Test panels fabricated in accordance with section 5.3.3.11 and tested in accordance with the procedures specified in appendix B shall meet all the requirements indicated in tables 3 and 4.

6 QUALITY ASSURANCE PROVISIONS

6.1 Supplier Responsibilities

The supplier is responsible for the performance of all test and inspection requirements specified herein. The supplier may use his own or any other test facilities acceptable to the buyer. Test and inspection records shall be retained for a minimum of 5 years and shall be made available to the buyer.

6.2 Material Qualification

6.2.1 Qualification and Approval

A supplier shall not begin to supply material to this specification until all qualification requirements have been met and approval has been granted by the buyer. Thereafter, the materials and method of manufacture shall not be changed without prior approval by the buyer.

6.2.2 Sample Material Requirement

Qualification shall be based upon the manufacture and successful test of three batches of prepreg. Each prepreg batch shall consist of only one distinct graphite fiber yarn lot and one distinct resin batch. Two separate and distinct fiber lots and two separate and distinct resin batches shall be used to make the three prepreg batches. The supplier shall submit two copies of test data, including individual and average test values, to the buyer, which show that the material meets all of the requirements of the specification.

6.2.3 Audit

Suppliers seeking qualification to this specification shall submit to an audit of their product manufacturing operations, raw material traceability, process

records, test procedures, and Quality Assurance records. Nondisclosure agreements will be signed between the supplier and the buyer if deemed necessary.

6.2.4 Process Plan

The supplier shall have on file a Process Control Document that contains all manufacturing baseline chemical and in-process test information approved by the buyer. No change to approved product formulation, critical raw materials or suppliers, basic methods of manufacture, testing, or geographic location shall be made without prior approval by the buyer. Requalification of a revised material may be required, and a revised supplier designation may be required if the Process Control Document is changed.

6.2.5 Qualification Tests

Materials shall satisfy all qualification requirements listed in tables 1, 2, 3, and 4 before qualification is approved.

6.3 Acceptance

6.3.1 Certification

The supplier shall certify that the components (graphite fiber, resin, and carrier) and processing used in the manufacture of each production batch and lot of material procured under this specification meet the specifications used for qualification.

6.3.2 Test Reports

The supplier shall furnish with each lot of prepreg two copies of a report that states the quantitative results of all acceptance tests and inspections specified in tables 2, 3, and 4. Both individual and average test results shall be included. The report shall include all necessary identification to correlate the inspections and test results with the roll and lot of material and the purchase order or contract.

6.3.3 Acceptance Tests

6.3.3.1 Prepreg resin content.— The supplier shall test every roll of material to verify that the resin content and fiber areal weight meet the specifications in table 2.

6.3.3.2 Chemical characterization.— The supplier shall characterize one roll of each prepreg lot by liquid chromatography.

6.3.3.3 Prepreg properties.— The supplier shall conduct volatile content, tack, viscosity profile, and handling life tests listed in table 2 in accordance with the following schedule:

Material quantity, lb in each lot	Sample selection
1 to 250	Test one roll, random choice
251 to 500	Test first and last roll
500 or more	Test first, every tenth, and last roll

6.3.3.4 Laminate properties.— The supplier shall fabricate panels, by using the methods described in section 5.3.3.11, and shall conduct acceptance tests listed in tables 3 and 4. The properties of each panel shall meet the requirements listed in tables 3 and 4. The following schedule shall be used:

Material quantity, lb in each lot	Sample selection
1 to 250	Test one roll, random choice
251 to 500	Test first and last roll
500 or more	Test first, every tenth, and last roll

In addition, the supplier shall fabricate a processability test panel as specified in section A.10 and perform tests specified in table 4 for each lot of prepreg.

6.3.3.5 Defects.— The supplier shall submit the roll defect log with each shipment and attach a copy inside the roll core.

7 ACCEPTANCE/REJECTION CRITERIA

7.1 Acceptance

Each prepreg lot submitted for acceptance shall meet the requirements of tables 1, 2, 3, and 4.

7.1.1 - If one roll among those tested fails, two additional rolls may be tested for at least the failing property. Both rolls must pass for the lot to be acceptable.

7.2 Rejection

7.2.1 - Material rejected on retest shall not be tested again for acceptance without written authorization from the buyer. Following buyer approval, rejected material may be reworked, retested, and resubmitted for acceptance. However, it shall be accompanied by the data concerning previous rejection, and a detailed description of the action taken to correct the defect or reason for failure.

7.2.2 - The buyer reserves the right to select any prepreg roll from a lot for testing. Failure of these tests to meet acceptance requirements shall be grounds for rejection of the entire lot.

8 PACKING AND SHIPPING

8.1 General

Material shall be packed and shipped in such a manner as to ensure the conformance of the properties and the storage life required by this specification.

8.2 Packing

8.2.1 Interior Packing

The prepreg material shall be packaged in rolls wound on a hollow core 8 in. or larger in diameter. A listing of the defective areas in the tape shall be attached to the inside of the core. A bag of desiccant shall be secured inside the core to absorb moisture. Each roll and the supporting core shall be sealed in a sleeve or bag to prevent moisture entry or loss of volatiles. The roll shall be fully supported from the core ends in such a manner that the prepreg does not telescope and the core end supports shall be at least 1 in. larger than the roll outside diameter. Each roll shall be packaged in a separate box.

8.2.2 Color Coding

Each prepreg roll shall be color coded either by colored carrier or by color marking of the roll core end or center. The color code shall be as follows:

Grade	Type	Class	Color Code
145	1	1	Green

8.2.3 Shipping Container

Clean dry containers constructed so as to ensure acceptance by common or other specified carrier and safe transportation to the place of delivery shall be used. Containers shall be constructed and insulated so that solid carbon dioxide may be packed in sufficient quantities to maintain the material at a temperature not to exceed 10°F (-15°C) for 18 hours, or the time required for shipment, whichever is longer. The gross weight of the container and contents, when packed for shipment, shall not exceed 130 lb.

8.3 Shipping

Material shall be shipped in such a manner that the temperature does not exceed 10°F. If solid carbon dioxide is used to maintain required temperature, a quantity shall be present upon receipt by the buyer. If refrigerated shipment is used, a recorder shall be included to indicate temperature history during shipment.

8.4 Storage

Upon delivery to the buyer, the material shall immediately be placed in storage below 10°F.

8.5 Marking

8.5.1 Package

Each roll of prepreg shall be legibly and permanently marked by means of a tag, securely attached, in such a manner that it remains in place until all material on the roll is completely used. Each package that contains a roll of prepreg shall also be labeled. Information on the tags and labels shall include, but not be limited to, the following items:

Manufacturer's name

Manufacturer's trademark and identification number

Fiber identification and lot number

Number and revision letter of this specification

Type, class, and grade per section 2.

Nominal width

Impregnation date

Lineal feet in roll and net weight

Manufacturer's lot and roll number

CAUTION: Store below 10°F

Storage life expiration date

Purchase order number

8.5.2 Shipping Container

Shipping containers shall be legibly and permanently marked and shall include, but not be limited to, the following information:

Graphite fiber, 350°F cure, 200°F resistant toughened resin prepreg

This specification number

Type number, class number, grade

Manufacturer's name

Material trademark

Manufacturer's lot number

Roll number(s) in container

Nominal width

Nominal length or weight per roll

Shipping and storage requirements

Purchase order

Shipping date

CAUTION: Ship below 10°F

Store below 10°F

APPENDIX A

TEST METHODS FOR UNCURED PREPREG PROPERTIES

Prepreg properties required for qualification and acceptance shall be obtained by tests performed according to the procedures specified in the following sections. CAUTION: The use of solvents is specified herein. Solvents are potential health hazards as they are flammable and are rapidly absorbed through the skin which can produce serious effects when direct contact with hot solvent occurs. Proper ventilation, fire hazard protection, and precautions to avoid skin contact must be assured before these tests are initiated.

A.1 Sampling Procedure

Remove the material from cold storage and store at room temperature for a minimum of 4 hr. Remove the material from the moisture-proof bag and cut enough material to perform all tests listed in table 2. Replace the roll of material in the moisture-proof bag, reseal, and replace in cold storage. All prepreg tests and laminate fabrication should be completed within 24 hr of sampling. If material testing is delayed beyond 24 hr, store sample by placing in a sealed moisture-proof bag and refrigerate.

A.2 Wet Resin Content

The wet resin content by weight of the prepreg material shall be determined by the following procedure.

A.2.1 - For tapes that are at least 12 in. (30 cm) wide, cut three specimens, 3 by 3 in. (7.6 by 7.6 cm) minimum, from equidistant locations across the width of the tape but not closer than 1 in. (2.5 cm) to the edge of the tape. For tapes that are less than 12 in. (30 cm) wide, cut three rectangular specimens that are 9 in² (58 cm²) in area and equal distant from the tape edges. Record specimen location in the tape. Remove the carrier from the back of each specimen. Weight each specimen to the nearest 0.001 g. Record as W_1 . Measure specimen length and width to the nearest 1 mm. Calculate specimen area:

$$\text{Area} = \text{Length} \times \text{Width}, \text{ m}^2$$

A.2.2 - Desolve resin by the method specified in paragraph A.2.2.1 or A.2.2.2 and separate fibers from specimens.

A.2.2.1 - Wash specimens separately in a suitable boiling solvent for 2 min. Time starts when solvent starts to boil. Decant the solvent. The solvent used shall be selected on the basis of being able to completely dissolve the resin under the conditions of this test.

A.2.2.2 - Soak specimens at room temperature in methyl ethyl ketone (MEK) or methylene chloride until resin is completely dissolved.

A.2.3 - Separate fibers from solvent by filtering and rinsing three times with solvent, each time decanting or filtering carefully to retain the fibers.

A.2.4 - Dry fibers by placing them in a circulating-air oven maintained above 225°F until a constant weight is reached. Remove and place in a desiccator. Constant weight shall be defined as a weight change of less than ± 0.005 g.

A.2.5 - After a minimum of 10 min., remove specimens from desiccator and weigh to the nearest 0.001 g. Record as W_2 .

A.2.6 - Calculate wet resin content as follows:

Wet resin content by weight:

$$\text{Resin content} = \frac{W_1 - W_2}{W_1} \times 100 \text{ percent weight}$$

where

W_1 original weight of specimen, g

W_2 final weight of specimen, g

A.2.7 - Record individual and average results.

A.3 Graphite Fiber Areal Weight

The graphite fiber areal weight shall be determined from the data of the uncured resin content specimen by using the following equation. The weight of the solvent cleaned fibers was recorded as W_2 .

$$\text{Fiber areal weight} = \frac{\text{Weight of specimen, g}}{\text{Area of specimen, m}^2} = \frac{W_2}{A} \text{ g/m}^2$$

A.3.1 - Record individual and average results.

A.4 Volatile Content

The volatile content by percent weight of the prepreg shall be determined by the following procedure.

A.4.1 - Cut three specimens from material adjacent to that used for the resin content specimens in paragraph A.2.1. The specimens shall be the same size as the resin content specimens. For tape that is greater than 12 in. (30 cm) wide, the specimens shall not be cut closer than 1 in. (2.5 cm) to the edge of the tape. Record specimen location in the tape. Remove the carrier from each specimen. Weigh each specimen to the nearest 0.001 g. Record as W_3 .

A.4.2 - Place the specimens in a porous Teflon¹ coated fiberglass envelope, or suspended on an aluminum tray (areas of the tray in contact with the specimens shall be covered with a Teflon parting film). Place the specimens in a circulating-air oven, preheated to $350^\circ \pm 10^\circ\text{F}$ and hold for 15 ± 1 min. Hanging specimens in the oven is acceptable, provided there is no resin loss due to dripping.

A.4.3 - Remove the specimen from the oven and place in a desiccator at room temperature for 30 min minimum prior to weighing to the nearest 0.001 g. Record weight as W_4 .

A.4.4 - Calculate the volatile content as follows:

$$\text{Volatile content} = \frac{W_3 - W_4}{W_3} \times 100 \text{ percent weight}$$

A.4.5 - Record individual and average values.

A.5 Tack

The tack at ambient temperature of $75^\circ \pm 5^\circ\text{F}$ of the prepreg shall be determined by the following procedures.

A.5.1 - If the width of the prepreg tape is 5 in. (12.7 cm) or greater, cut one specimen 10 in. (25.4 cm) long by 3 in. (7.6 cm) wide minimum, not closer than 1 in. (2.5 cm) to the edge of the tape. If the width of the prepreg tape is less than 5 in. (12.7 cm), cut one specimen 10 in. (25.4 cm) long by 3 in. (7.6 cm) or full-tape wide, whichever is less, from the tape. Leave the specimen on the carrier until immediately before conducting the test.

A.5.2 - Apply and dry a coat of mold release (Frekote #33 releasing interface or equivalent) to a 2-in-diameter metal mandrel with a surface roughness value of 100 $\mu\text{in. (rms)}$ or better.

A.5.3 - Wrap the prepreg around the mandrel with the fibers oriented in the circumferential direction and the exposed surface in contact with the mandrel. Remove the carrier from the prepreg as it is wrapped on the mandrel and apply light pressure to

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the prepreg. The pressure may be produced either by rolling on a flat table top with downward pressure on the mandrel or by using a squeegee or separate roller over the prepreg.

A.5.4 - Examine the rolled material for proper carrier release as noted in section 5.3.3.7, Handling characteristics.

A.5.5 - After 30 min, examine the rolled material for adherence to the mandrel and to itself.

A.5.6 - Report the results as pass or fail. Failure is defined as inability of the prepreg to adhere to itself for at least 30 min.

A.6 Viscosity Profile

The neat resin melt viscosity profile of the prepreg shall be determined by the following procedure.

A.6.1 Materials

A.6.1.1 Polyimide film (3 to 5 mm thick)

A.6.1.2 Polyimide pressure sensitive tape

A.6.1.3 Certified viscosity standard S30000, calibrated at 50° and 100°C, Cannon Instrument Company or equivalent.

A.6.1.4 Poly(dimethylsiloxane) calibration standard

A.6.2 Equipment

A.6.2.1 Rheometrics Dynamic Spectrometer RDS-7700 or equivalent

A.6.2.2 Disposable plates (50 mm) for rheometer

A.6.2.3 Desiccator

A.6.2.4 Vacuum oven with viewing window

A.6.2.5 Millivolt source and cold junction compensator

A.6.2.6 Extra heavy-duty aluminum foil

A.6.2.7 Heated press, minimum platen size, 8 by 8 in.

A.6.3 Prepreg Resin Sampling

A.6.3.1 - Prepare a unidirectional stack of prepreg; 80 plies, 1.5 by 6 in. This laminate is formed from 1.5- by 12-in. strips of prepreg which are folded with their ends in the center (fig. 3). The laminate is then wrapped with a 6- by 24-in. section of prepreg (fig. 3), by three strips of extra heavy-duty aluminum foil 1 3/4 by 20 in. (fig. 3), and finally by a strip of foil 6 by 20 in. (fig. 3). The aluminum on the edges of the laminate is perforated on each side (fig. 3) at five places evenly spaced. Perforations should be approximately 1 by 2 cm in size.

A.6.3.2 - Preheat the press to $200^{\circ} \pm 5^{\circ}\text{F}$, or other specified temperature agreed upon by the supplier and the buyer, to allow flow without onset of cure.

A.6.3.3 - Place a sheet of polyimide film on the heated lower platen of the press.

A.6.3.4 - Place the aluminum foil covered laminate on the polyimide film in the center of the heated press and cover with another sheet of polyimide film.

A.6.3.5 - Apply a pressure of 500 ± 5 psi and allow the laminate to heat for 3 ± 0.5 minutes, then gradually increase the pressure at 7000 ± 1000 psi/min until $20\,000 \pm 500$ psi is achieved.

A.6.3.6 - Release pressure, remove upper polyimide sheet and discard. Remove the laminate and lower polyimide sheet from the press and separate the laminate from the lower polyimide sheet.

A.6.3.7 - Fold the resin-coated lower polyimide sheet over on itself, seal in a solid polyethylene bag and chill to 0°F or below.

A.6.3.8 - Remove the resin from the polyimide sheet by rapidly flexing the polyimide sheet while the resin is below 0°F .

A.6.4 Degassing of the Resin

A.6.4.1 - Form a 1/2-in-high (13-mm) dam around the circumference of one of the 50-mm disposable plates with polyimide film and seal in place with pressure sensitive polyimide tape.

A.6.4.2 - Preheat plate, with dam affixed, in a vacuum oven at reduced pressure for 20 ± 5 min at a temperature equal to or lower than the temperature employed in paragraph A.6.3.2. Remove plate from oven.

A.6.4.3 - Place approximately 3 g of resin on the plate and return plate to the vacuum oven.

A.6.4.4 - Gradually apply vacuum (5 in. (127 mm) Hg below atmospheric pressure) to degass the resin sample. Do not allow resin to froth over the dam during degassing. Allow sample to remain in the oven at the temperature used for paragraph A.6.4.2 for a total of 15 to 20 min after establishing constant temperature and pressure.

A.6.4.5 - Remove plate from oven, place in a desiccator and allow to cool to room temperature. Remove the polyimide film and tape when the plate has cooled.

A.6.4.6 - Place resin sample in a desiccator and store at 0°F or below until immediately prior to testing.

A.6.5 Initial Calibration of the Dynamic Mechanical Rheometer

A.6.5.1 - The torque output of the transducer is calibrated by the standard methods recommended by the manufacturer.

A.6.5.2 - A parallel plate geometry is employed. The storage (G') and loss (G'') shear moduli of the poly(dimethylsiloxane) viscosity standard are measured as a function of frequency at $26 \pm 1.0^\circ\text{C}$ in the rate sweep mode.

A.6.5.3 - The log of the storage modulus for the poly(dimethylsiloxane) should increase linearly with the log of the frequency between 0.1 and 0.398 rad/sec. When $G' = G''$ the frequency must be 7 ± 0.5 rad/sec. See calibration curve in figure 4.

A.6.5.4 - The thermocouple which monitors the temperature of the sample is calibrated by using a millivolt source and a cold junction compensator.

A.6.5.5 - The gap spacing in the rheometer is adjusted so that, with no sample present, the spacing is zero at 50°C .

A.6.6 Calibration With Certified Viscosity Standard

NOTE: Temperatures given are equipment settings which are accurate to $\pm 1^\circ\text{C}$. Thus, there is no tolerance on the specified temperature.

A.6.6.1 - Place a 2 to 4 mL sample of Cannon Viscosity Standard S30000 on the parallel plate.

A.6.6.2 - Close the gap between the plates until the solution reaches the edge of the plates. Record the gap setting.

A.6.6.3 - Heat the sample to an equipment setting of 50°C .

A.6.6.4 - Set the rheometer to a strain of 40 percent and perform a calibration scan.

A.6.6.5 - During the rate sweep scan, record loss and storage moduli, complex viscosity, temperature, and torque and plot the complex viscosity as a function of frequency.

A.6.6.6 - The measured viscosity must agree with the calibrated value within ± 10 percent.

A.6.6.7 - The calibration shall be repeated on the same sample as described in paragraphs A.6.6.3 through A.6.6.5 except at a temperature of 100°C . The measured viscosity must agree with the calibrated value within ± 10 percent.

A.6.7 Determination of Viscosity

A.6.7.1 - Remove the desiccator containing the 3-g resin sample (see paragraph A.6.4.6) from the 0°F storage and allow to warm to room temperature prior to opening the desiccator.

A.6.7.2 - Set the rheometer to an equipment setting of 50°C.

A.6.7.3 - Transfer the sample from the desiccator to the rheometer in an expedient manner.

A.6.7.4 - Close the gap between the plates until the resin reaches the edge of the plates. Record the gap setting.

A.6.7.5 - Determine a range of maximum strain in which the complex viscosity, loss shear modulus, and storage shear modulus are constant. This range may be achieved by setting the rheometer to a frequency of 10 rad/sec and conducting several runs in the range 0.1 to 50 percent strain. Select a maximum strain value that produces a minimum torque greater than 2 g-cm and meets the other requirements of this paragraph.

A.6.7.6 - Set the rheometer to the maximum strain value selected in paragraph A.6.7.5 and raise the temperature of the sample from 50°C to 177°C (or the recommended curing temperature) at 1°C/min. Exercise care to avoid heating the sample above 177°C, and when 177°C is achieved, maintain constant temperature. Measure and record complex viscosity, loss shear modulus, storage shear modulus, torque, and temperature as a function of time. Terminate the test when the viscosity exceeds 3000 poise. Plot the logarithm of complex viscosity as a function of time.

A.7 HPLC Characterization

A chemical characterization of the prepreg shall be determined by high pressure liquid chromatography (HPLC).

A.7.1 HPLC Analysis Method

The supplier shall develop an HPLC analysis to provide a chromatographic "fingerprint" of key ingredients of the matrix resin. Sample extraction, analysis, and report shall be patterned after the general approach given in NASA CR-3531.

A.7.2 Report

A chromatogram with appropriate peak integration records for standards which can be used to obtain quantitative determinations of major components shall be supplied. The report shall record and compare the batch sample chromatogram with the resin standard chromatogram to detect contaminants or gross change in formulation.

A.8 Handling Life

Cut one piece of material 10 in. long and 3 in. minimum width (as described in paragraph A.5.1) and seal in a moisture-proof bag. After 10 days at room temperature

(80°F maximum) remove from the sealed bag and test for tack as described in section A.5.

A.9 Processing Life

Cut material from the roll and laminate a 3- by 6-in. (minimum dimensions), 16-ply, 0° layup on a prepared tooling surface. Expose the layup for 30 days at room temperature (maximum of 80°F) and then bag and cure the layup by the standard procedure defined in paragraph 5.3.3.11. Test the cured panel for resin and void content as specified in sections B.3 and B.4.

A.10 Processability Test Panels

Laminate processability shall be determined by the following procedures.

A.10.1 Layup

Cut material from an opened roll of prepreg and lay up two panels 24 by 26 in. by 48 plies (0.24 in. nominal thickness) with a quasi-isotropic fiber pattern $[45^\circ/0^\circ/-45^\circ/90^\circ]_{6s}$. Four Teflon disks (0.14 ± 0.01 mm thick) shall be placed in one corner at least 1 in. from the edge and between the two center plies. Two 1/2-in-diameter disks and two 1/4-in-diameter disks shall be used and all disks shall be separated at least 1/4 in.

A.10.2 Bagging

Bag the panels by the method shown in figure 1.

A.10.3 Cure

Cure each panel in an autoclave by using the cure cycle shown in figure 2. The first panel shall have a temperature rise of $2^\circ \pm 0.2^\circ\text{F}/\text{min}$ and the second panel shall have a temperature rise of $8^\circ \pm 0.2^\circ\text{F}/\text{min}$. Temperature shall be measured using thermocouples located at mid-thickness of the panels $0.10^{+0.10}_{-0}$ in. from the panel edge.

A.10.4 Nondestructive Inspection

The process qualification panels shall be inspected for internal defects by an ultrasonic nondestructive inspection (NDI) method approved by the buyer. A record of nondestructive inspection results shall be retained for a minimum of 5 years and shall be made available to the buyer.

A.10.5 Thickness Per Ply

Measure panel thickness and calculate thickness per ply as prescribed in section B.5.3.

A.10.6 Micrographic Analysis

Six specimens 1 in. long by 0.5 in. wide shall be cut from representative areas across the width of each processability laminate. No specimen shall be taken closer than 1 in. to the edge of the laminate. One edge of each specimen shall be polished and observed by light microscopy. Report visual observations, and if a permanent pictorial record is required by the buyer, prepare photomicrographs.

A.10.7 Resin Content, Void Content, and Density

Test a minimum of three samples from each processing panel for resin content, void content, and density as prescribed in sections B.3 and B.4.

APPENDIX B

TEST METHODS FOR CURED LAMINATE PROPERTIES

Laminate properties required for qualification and acceptance shall be obtained by tests specified in the following sections. The laminates shall be fabricated by using the procedures prescribed in section 5.3.3.11.

B.1 Symbols

The symbols used for the calculations in this appendix are defined in this section.

b	specimen width, in.
D_F	density of graphite fiber, g/cm^3
D_L	density of cured laminate specimen, g/cm^3
D_R	density of cured resin (from supplier), g/cm^3
E_C	compression modulus, lb/in^2
E_O	laminate modulus, lb/in^2
E_t	tensile modulus, lb/in^2
F	fiber volume
F_C	compression strength, lb/in^2
F_{CS}	compression interlaminar shear strength, lb/in^2
F_t	tensile strength, lb/in^2
G_C	interlaminar fracture toughness, $\frac{\text{in-lb}}{\text{in}^2}$
G_{LT}	longitudinal shear modulus of unidirectional composite, lb/in^2
L	overlap length, in.
l	gage length of extensometer, in.
m	initial slope of load deflection curve, change in load divided by change in extensometer length, lb/in .
P	ultimate load, lb
P_1	load at 0.001 in/in strain, lb
P_5	load at 0.005 in/in strain, lb
P_6	load at 0.006 in/in strain, lb

t specimen thickness, in.
 W_1 original weight of specimen, g
 W_2 final weight of specimen, g
 Z resin content of cured laminate specimen, percent weight
 ϵ_c strain at onset of delamination
 ϵ_f strain at failure
 ϵ_y transverse strain at 0.005 in/in longitudinal strain

B.2 General

B.2.1 - Laminates shall meet the property requirements indicated in table 3. In addition, laminates shall meet the ply thickness, resin content, void content, and density requirements specified in table 4. The values indicated in table 3 are the minimum required average of the specified replicates. The minimum value for any one test shall equal or exceed 80 percent of the required average value.

B.2.2 - Perform all mechanical property tests with test machines complying with ASTM E 4.

B.2.3 - The unloaded edges of each specimen shall be machined to $\pm 1^\circ$ of the 0° fiber direction. The ends of the specimen that are being loaded or gripped shall be machined to $\pm 1^\circ$ of perpendicular to the 0° fiber direction. For specimens not having 0° fibers, the unloaded edges shall be machined to $\pm 1^\circ$ of the longitudinal axis, and the ends of the specimens that are loaded or gripped shall be machined to $\pm 1^\circ$ of perpendicular to the longitudinal axis.

B.2.4 - Test temperatures shall not vary more than $\pm 10^\circ\text{F}$ from the values indicated in table 3. Room temperature is defined to be 75°F . Specimens to be tested without moisture conditioning at -100°F or 200°F shall be held at the test temperature for 10 ± 3 min prior to testing. Moisture-conditioned specimens to be tested at 200°F shall be held at this temperature for 2 ± 1 min prior to testing.

B.2.5 - Moisture conditioning for specimens to be tested at the wet condition indicated in table 3 shall be achieved by using the following procedure:

B.2.5.1 - Dry specimens in a circulating-air oven at $175 \begin{smallmatrix} + 0 \\ - 10 \end{smallmatrix}^\circ\text{F}$ for $160 \begin{smallmatrix} + 4 \\ - 0 \end{smallmatrix}$ hr.

B.2.5.2 - Measure and record specimen weight after drying.

B.2.5.3 - Soak specimens in demineralized water at $160 \begin{smallmatrix} + 10 \\ - 0 \end{smallmatrix}^\circ\text{F}$ for $340 \begin{smallmatrix} + 4 \\ - 0 \end{smallmatrix}$ hr.

B.2.5.4 - Wipe excess moisture from the surface of the specimen. Measure specimen weight and record weight gain.

B.2.5.5 - Calculate and record specimen moisture content as percent of dry weight.

B.2.5.6 - Test specimens within 30 min after moisture conditioning, or, if not possible, store in a saturated atmosphere (95- to 99-percent relative humidity) at room temperature for no more than 75 hr before testing.

B.3 Resin Content

The resin content of cured laminates shall be determined as follows.

B.3.1 - Carefully cut three specimens weighing between 0.5 and 2.0 g each not closer than 1 in. from the laminate edges. Specimen edges must be free of surface roughness so that an accurate density can be obtained. Do not use broken specimens.

B.3.2 - Dry specimen for a minimum of 1 hr at $300^{\circ} \pm 10^{\circ}\text{F}$ ($149^{\circ} \pm 6^{\circ}\text{C}$), cool in an ambient temperature dessicator for 10 ± 1 min. Weigh each specimen to the nearest 0.001 g. Record weights as W_1 .

B.3.3 - Determine the density of each specimen by any method that is accurate to within 0.005 g/cm^3 . Record the method used. Record the density as D_L . If specimens are submerged in a fluid to determine density, repeat B.3.2 before performing B.3.4.

B.3.4 - Place the specimen in concentrated nitric acid that has been stabilized at a temperature of $140^{\circ} \pm 5^{\circ}\text{F}$ ($60^{\circ} \pm 3^{\circ}\text{C}$). Digest at this temperature for $3 \text{ hr} \pm 10 \text{ min}$. Digestion at $200^{\circ} \pm 5^{\circ}\text{F}$ ($93^{\circ} \pm 3^{\circ}\text{C}$) for $45 \pm 5 \text{ min}$ is also acceptable. Record method used.

B.3.5 - Filter in a suitable device or decant so that all the carbon fibers are retained, and rinse thoroughly with MEK or acetone.

CAUTION: Do not allow MEK or acetone to come in contact with nitric acid.

B.3.6 - Dry for a minimum of 1/2 hr at 300°F (149°C).

B.3.7 - Desiccate for at least 10 min and weigh. Record as W_2 .

B.3.8 - Calculate resin content as follows:

$$\text{Resin content} = Z = \frac{W_1 - W_2}{W_1} \times 100 \text{ percent weight}$$

B.3.9 - Report individual and average values of Z.

B.4 Void Content

Void content shall be determined in accordance with ASTM D 2734, with the following exceptions.

B.4.1 - Resin content and density shall be as determined per section B.3 of this specification.

B.4.2 - Calculate void content as follows:

$$\text{Void content} = \left\{ 1 - D_L \left[\frac{1}{D_F} + \frac{Z}{100} \left(\frac{1}{D_R} - \frac{1}{D_F} \right) \right] \right\} \times 100 \text{ percent volume}$$

B.4.3 - Report individual and average values.

B.5 Fiber Volume and Ply Thickness

B.5.1 Fiber Volume

Calculate fiber volume as follows:

$$\text{Fiber volume} = F = \left(1 - \frac{Z}{100} \right) \times \frac{D_L}{D_F} \times 100 \text{ percent}$$

where the terms are defined in section B.1.

B.5.2 - Report individual and average values.

B.5.3 Ply Thickness

Measure the thickness of the cured laminate in at least five locations, spaced to represent the laminate including the center, but not closer than 2 in. to the edge, with a flat-nose or ball-nose micrometer, to the nearest 0.0001 in. Average the readings and divide by the number of plies. Report as "thickness per ply."

B.6 Tensile Strength and Modulus Tests

B.6.1 - Machine tensile specimens from the same panel in accordance with instructions and dimensions shown in figure 5. Measure and record specimen width and thickness to the nearest 0.001 in.

B.6.2 - Test specimens in a universal test machine at a deflection rate of 0.05 in/min.

B.6.3 - Measure the failure load and the longitudinal strain as a function of applied load with a strain gage or a 1-in. gage length extensometer.

B.6.4 - Calculate the tensile strength F_t :

$$F_t = \frac{P}{bt} \text{ lb/in}^2$$

B.6.5 - If strain was measured with an extensometer, calculate tensile modulus E_t :

$$E_t = \frac{m\ell}{bt} \text{ lb/in}^2$$

B.6.6 - If strain was measured with strain gages, calculate tensile modulus:

$$E_t = \frac{1}{bt} \frac{P_6 - P_1}{(0.005)} \text{ lb/in}^2$$

B.6.7 - Report individual and average values for tensile strength and modulus. Required values are listed in table 3.

B.7 - Compression Strength and Modulus Tests

B.7.1 - Machine the compression modulus and strength specimens from the same panel in accordance with the instructions and dimensions shown in figure 6. Measure and record specimen width and thickness to the nearest 0.001 in.

B.7.2 - Test specimens in a universal test machine by using the ASTM D 695 compression fixture. Test at a deflection rate of 0.05 in/min.

B.7.3 - Definitions of symbols used in calculations are given in section B.1.

B.7.4 - Compression strength shall be determined by testing the tabbed specimen shown in figure 6.

B.7.5 - Calculate compression strength F_C as

$$F_C = \frac{P}{bt} \text{ lb/in}^2$$

B.7.6 - Compression modulus shall be determined by testing the untabbed specimen shown in figure 6. Test the modulus specimen to a minimum strain of 0.006 in/in. Measure the applied load, and the longitudinal strain as a function of applied load with an extensometer or strain gage.

B.7.7 - If strain was measured with an extensometer, calculate the compression modulus E_C as follows:

$$E_C = \frac{m\ell}{bt} \text{ lb/in}^2$$

B.7.8 - If strain was measured with a strain gage, calculate the compression modulus:

$$E_C = \frac{1}{bt} \frac{P_6 - P_1}{(0.005)} \text{ lb/in}^2$$

B.7.9 - Report individual and average values for compression strength and modulus. Required values are listed in table 3.

B.8 Compression Interlaminar Shear Test

B.8.1 - Fabricate compression interlaminar shear specimens as shown in figure 7. Measure and record specimen width, thickness, and notch overlap length to the nearest 0.001 in.

B.8.2 - Test at a deflection rate of 0.05 in/min by using the fixture and procedures of ASTM D 695.

B.8.3 - Calculate compressive interlaminar shear strength as follows:

$$F_{cs} = \frac{P}{bL} \text{ lb/in}^2$$

B.8.4 - Report individual and average values. The required values are listed in table 3.

B.9 Open-Hole Tension Test

B.9.1 - Fabricate open-hole tension specimens as shown in figure 8.

B.9.2 - A hole having a nominal diameter of 0.250 in. shall be drilled and/or reamed so as to avoid any delamination in the test specimen. After machining, measure and record hole diameter and specimen dimensions.

B.9.3 - Test in tension at a deflection rate of 0.05 in/min

B.9.4 - Calculate strength as follows:

$$\text{Open-hole tensile strength} = \frac{P}{bt} \text{ lb/in}^2$$

B.9.5 - Report individual and average values for open-hole tension strength. Required values are listed in table 3.

B.10 Open-Hole Compression Test

B.10.1 - Fabricate open-hole compression specimens as shown in figure 9.

B.10.2 - A hole having a nominal diameter of 0.250 in. shall be drilled and/or reamed so as to avoid any delamination in the test specimen. After machining, measure and record hole diameter and specimen dimensions to the nearest 0.001 in.

B.10.3 - Test specimen by using a fixture such as that shown in figure 10. The longitudinal axis of the specimen must be maintained parallel to the load axis of the machine and centered in the machine, and the side supports on the edges parallel to the loading axis must not constrain transverse deformation due to Poisson's effect.

B.10.4 - Test at a deflection rate of 0.05 in/min.

B.10.5 - Calculate strength as follows:

$$\text{Open-hole compression strength} = \frac{P}{bt} \text{ lb/in}^2$$

B.10.6 - Report individual and average values for open-hole compression strength. In addition, report failure location and description of failure.

B.10.7 - Required values are listed in table 3.

B.11 Compression After Impact Test

B.11.1 - Fabricate the 12.0- by 7.0-in. compression after impact specimen as shown in figure 11.

B.11.2 - The impact test apparatus shall consist of a support fixture shown in figure 12 and an impactor. The impactor shall weigh 10.0 to 12.0 lb and shall have a 0.5-in. hemispherical steel tip on the end that impacts the specimen. A guide tube, lined with Teflon film or equivalent, shall be used to direct the vertical path of the impactor.

B.11.3 - Place the test specimen in the support fixture so that the impact location is at the exact center of the specimen. Clamp the top plate over the test specimen and attach to the base plate by installing nuts on the four tie-down studs and torquing each one to a nominal 20 ft-lb. Locate the guide tube above the test specimen so that the impactor will strike the center of the specimen. Coat the striker end of the impactor with white chalk dust or white grease to allow easy location of the actual impact point. The lower end of the guide tube should be approximately 10 in. above the surface of the specimen. Drop the impactor from a height above the test specimen to generate an impact energy of $20 \begin{smallmatrix} + 0.50 \\ - 0 \end{smallmatrix}$ ft-lb. Care should be taken to arrest the impactor after the strike so that a restrike does not occur. Remove the impacted specimen from the support fixture and visually determine and record the amount of damage to the specimen on the impacted surface and the back surface. Ultrasonically inspect the specimen to determine the extent of internal delamination.

B.11.4 - For each specimen, record the specimen identification number, thickness, front surface and back surface visual damage measurements, total delamination area from the ultrasonic measurement, and maximum width of the delamination from the ultrasonic inspection, measured perpendicular to the longitudinal axis of the specimen.

B.11.5 - Machine a 10- by 5-in. specimen from the impacted laminate as shown in figure 11.

B.11.6 - After machining, the postimpact compression specimen dimensions shall be measured to the nearest 0.001 in. at the locations shown in figure 11. Record individual and average values for thickness, width, and length.

B.11.7 - Install back-to-back axial strain gages on each specimen as shown in figure 11.

B.11.8 - Test the specimens by using a compression test fixture such as that shown in figure 10 so that (1) the longitudinal axis of the specimen is parallel to the load axis of the machine and is centered in the machine and (2) the side supports on the edges parallel to the loading axis do not constrain transverse deformation due to Poisson's effect.

B.11.9 - Test at a deflection rate of 0.05 in/min. Record strain values from all strain gages as a function of load throughout the test.

B.11.10 - Calculate compression strength as follows:

$$F_c = \frac{P}{tb} \text{ lb/in}^2$$

B.11.11 - Report individual and average values of strength. Required values are listed in table 3.

B.12 Longitudinal Shear Modulus

B.12.1 - Fabricate the $[\pm 45]_{3S}$ laminate specimen and install longitudinal and transverse strain gages as shown in figure 5. Measure and record specimen width and thickness to the nearest 0.001 in.

B.12.2 - Test in a universal test machine in accordance with ASTM D 3518 at a deflection rate of 0.05 in/min.

B.12.3 - Measure and record longitudinal and transverse strain as a function of applied load.

B.12.4 - Calculate the longitudinal shear modulus as

$$G_{LT} = \frac{P_5/bt}{0.01 - 2\epsilon_y} \text{ lb/in}^2$$

B.12.5 - Calculate tensile strength of $[\pm 45]$ laminate F_t as

$$F_t = \frac{P}{bt} \text{ lb/in}^2$$

B.12.6 - Report individual and average values for longitudinal shear modulus and tensile strength. Required values are listed in table 3.

B.13 - Edge Delamination Tension Test

B.13.1 - Fabricate edge delamination tension specimens as shown in figure 13.

B.13.2 - Measure the specimen thickness at the six locations along each edge as shown in figure 13 and record the individual and average thickness. Measure the specimen width at the three locations along the specimen length, as shown in figure 13, and record individual and average values.

B.13.3 - Test specimens in accordance with NASA RP-1092, ST-2. Use either a stroke-controlled or a strain-controlled hydraulic test machine. "Stroke controlled" controls crosshead displacement, "strain controlled" controls displacement over the gage length of the strain-measuring device mounted on the specimen. Do not run tests in a load-controlled machine.

B.13.4 - Use an extensometer (clip gage) with an appropriate extender arm to measure strain. The gage length is 4 in. with the gage mounts 1.5 in. from either grip.

B.13.5 - Mount specimen in test machine so as to grip an equal length on each end of the specimen and to expose 7 in. between grips. Emery cloth or tungsten carbide grit insets may be used to improve the gripping surface. If end tabs are used on specimens, the tabs should be squared off, not tapered.

B.13.6 - Test at a deflection of 0.006 in/min.

B.13.7 - Record the extensometer and load cell output on an x-y plotter (real-time analog display). Record deflection on the X-axis and load on the Y-axis.

B.13.8 - Load specimen until visible detection of edge delamination and a corresponding abrupt (not continuous) deviation occur in the load deflection plot as shown in figure 14. Record deflection level at onset of delamination. Note this point on the load deflection curve. If thickness variations greater than 0.003 in. were measured (paragraph B.13.2), record thickness at location closest to the delamination site.

B.13.9 - Calculate the strain at delamination onset ϵ_c as follows:

$$\epsilon_c = \frac{\text{Deflection at delamination onset}}{\text{Gage length of extensometer}}$$

B.13.10 - Continue loading until the specimen fractures into two pieces. Calculate and record the strain at failure ϵ_f :

$$\epsilon_f = \frac{\text{Deflection at failure}}{\text{Gage length of extensometer}}$$

B.13.11 - Determine the laminate modulus E_o from the initial portion of the load deflection curve (before delamination) as follows:

$$E_o = \frac{m\ell}{bt} \text{ lb/in}^2$$

B.13.12 - An exact method for calculating interlaminar fracture toughness G_c is given in NASA RP-1092 as ST-2. This method requires separate tests to measure individual laminate properties. The following method is an approximation to establish a minimum resistance to delamination for materials qualified under this specification. This method assumes that the tension modulus of a completely delaminated laminate is approximately 0.7 of the laminate tension modulus. This approximation is based on data from tests of selected toughened resin laminates and may not represent all materials which may be submitted for qualification. The supplier may, as an alternative to the method given below, determine G_c as specified in NASA RP-1092; if this method is chosen, the supplier shall measure and report all data specified in NASA RP-1092.

B.13.13 - Calculate an approximate interlaminar fracture toughness G_c as follows:

$$G_c = 0.16 \epsilon_c^2 t E_o \frac{\text{in-lb}}{\text{in}^2}$$

B.13.14 - Report individual and average values of G_c , ϵ_c , and E_o .

TABLE 1.- GRAPHITE FIBER REQUIREMENTS

Property	Unit	Value
Ultimate tensile strength ...	psi	5.20×10^6 (minimum)
Tensile modulus	psi	33×10^6 (minimum)
Elongation at failure	percent	1.5 (minimum)
Density	g/cm^3	1.70 to 1.83
Weight/unit length	g/m	
Grade 3000		0.18 (minimum)
Grade 6000		0.36 (minimum)
Grade 12 000		0.72 (minimum)
Sizing ^a	percent	0.0 to 1.6
Twist	turns per inch	0.0 to 0.8

^aThe prepreg supplier shall provide HPLC chromatogram of fiber sizing extract from each yarn lot.

TABLE 2.- UNCURED PREPREG REQUIREMENTS

Property	Requirement	Test method (section)	Qualification requirement	Acceptance requirement
Wet resin content	35 ± 2 percent by weight	A.2	Verify requirements as specified in section 5.2	Test each roll (sec. 6.3.3.1)
Graphite fiber areal weight	145 ± 5 g/m ²	A.3		Test each roll (sec. 6.3.3.1)
Volatile content	0.5 percent by weight maximum	A.4		Test as specified in sec. 6.3.3.3
Tack	Pass	A.5		
Viscosity profile	Test and report	A.6		Test one roll of each lot (sec. 6.3.3.2)
Chemical characterization	Test and report HPLC	A.7		
Storage life at 10°F maximum temperature	Supplier guarantee to meet all specification requirements after 6-month storage			
Handling life	Pass tack test after 10 days at 80°F	A.8		Test as specified in sec. 6.3.3.3
Processing life	Cure laminate after prepreg exposed 30 days at 80°F maximum and passed requirements of sections B.2 and B.3	A.9		

TABLE 3.- LAMINATE MECHANICAL PROPERTY REQUIREMENTS

Test	Ply orientation	Specimen configuration, in.	Required data	Test temperature, °F	Value (a)	Test replicates/batch		Test method (section)
						Qualification ^b	Acceptance ^c	
Tension	(0) ₈	0.5 by 9 tabbed	Strength	-100 RT	270 ksi 270 ksi	3 5	3	B.6
Tension	[±45] _{3s}	1.0 by 9 tabbed	Modulus	-100 RT	18m psi 18m psi	3 3		B.6
			Strength	RT 200	35 ksi 35 ksi	5 3		B.12
Compression	(0) ₈	0.5 by 3.15 tabbed	Shear modulus	RT 200	0.8m psi 0.8m psi	3 3		B.12
			Strength	-100 RT 200 200 wet ^d	200 ksi 200 ksi 180 ksi 160 ksi	3 5 5 3	3 3	B.7
Open-hole tension	[45/0/-45/90] _{2s}	1.5 by 12 0.25 diam. hole	Modulus	-100 RT 200	18m psi 18m psi 18m psi	3 3 3		B.7
			Strength	-100 RT	50 ksi 50 ksi	3 3		B.9
Open-hole compression	[45/0/-45/90] _{2s}	1.5 by 10 0.25 diam. hole	Strength	RT 200 200 wet ^d	33 ksi 30 ksi 26 ksi	3 3 3		B.10
Compression after impact	[45/0/-45/90] _{6s}	5 by 10	Strength	RT	30 ksi	3		B.11
Compression interlaminar shear	[45/0/-45/90] _{6s}	0.5 by 3.15	Strength	RT 200	10 ksi 8 ksi	3 3	3	B.8
Edge delamination tension test	[(±30) ₂ /90/90] _s	1.5 by 10	Interlaminar fracture toughness	RT	1.0 $\frac{\text{in-lb}}{\text{in}^2}$	3		B.13

^aValues are minimum required for average of specified test replicates. Minimum value of any one test shall equal or exceed 80 percent of required average value.

^bAll tests indicated in this table are required for qualification. For qualification requirements see sections 5.1 and 6.2.

^cTests indicated in this column are required for acceptance. For acceptance requirements see sections 5.2 and 6.3.

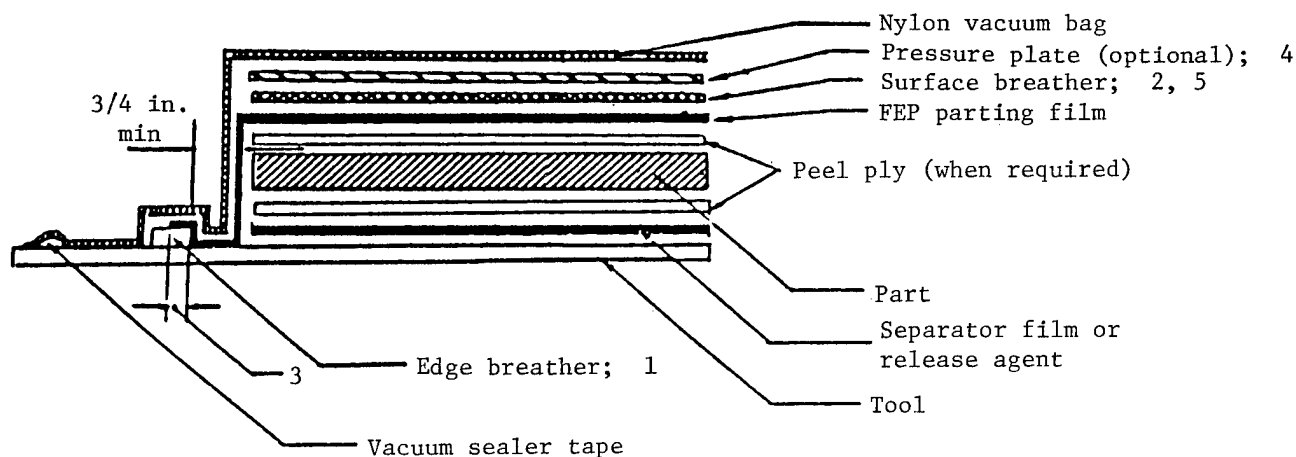
^dWet conditioning: procedure is defined in paragraph B.2.5.

TABLE 4.- LAMINATE PROCESSABILITY REQUIREMENTS

[Processability test panel shall be 24 by 26 in. by 48 plies quasi-isotropic]
fabricated as specified in section A.10

Property	Requirement	Test method	Qualification requirement	Acceptance requirement
Laminate thickness per ply	0.0054 to 0.0056 in.	Section B.5.3	As specified in section 6.2	As specified in section 6.3.3.4
Resin content	29 to 35 percent by weight	Section B.3		
Void content	2 percent maximum	Section B.4		
Density	1.53 to 1.62 g/cm ³	Paragraph B.3.3		

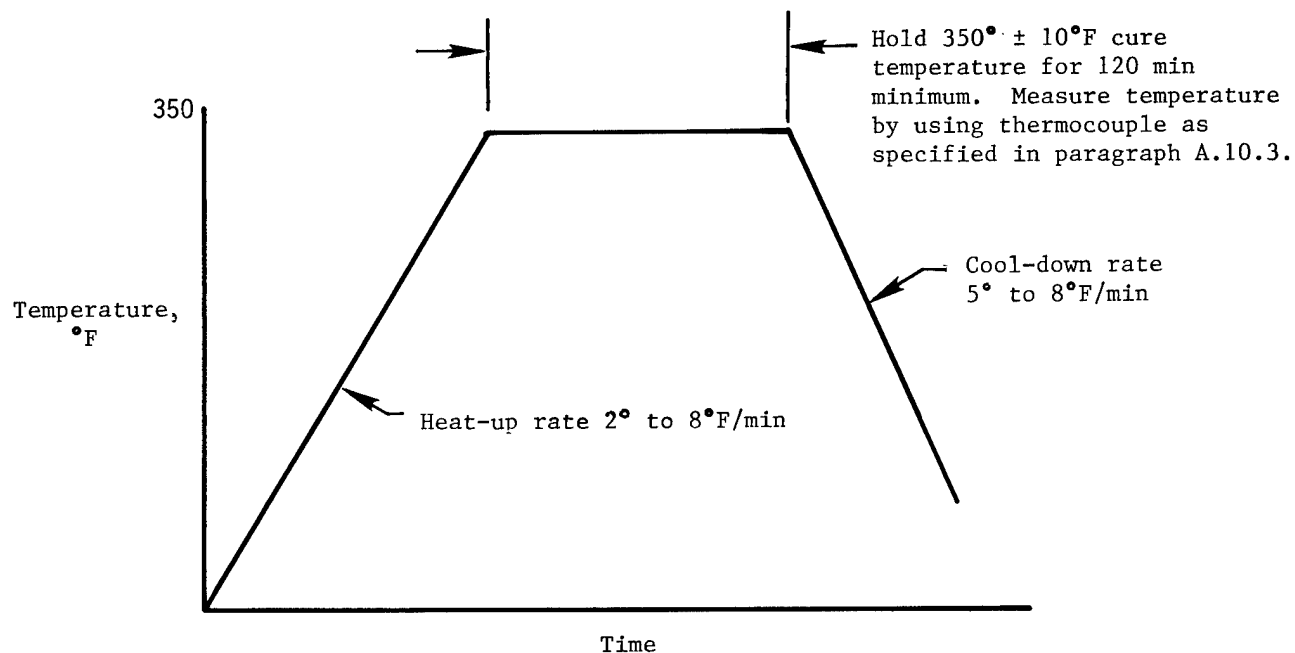
Defects	Limits	Test method	Qualification requirements	Acceptance requirements
Single void area	0.25 in ² maximum	Section A.10.4	Specified in section 6.2.5	As specified in section 6.3.3.4
Total accumulated void area	1.00 in ² in any 1 ft ²			
Single porous area	1.00 in ² maximum			
Total accumulated porous area	4.00 in ² in any 1 ft ²			
Distance between voids	4.00 in. minimum			
Defect distance from finished edge	1.00 in. minimum			



Not to scale

1. 1 in. minimum width with connection to vacuum source; at one corner of the layup, place a single fiberglass yarn between the edge of the layup and the edge breather to allow evacuation of air from the layup. Additional yarns may be required on larger parts to provide adequate removal of trapped air.
2. If fiberglass is used for surface breathers, it shall be net trimmed to the edge of the layup. At one position, connect the surface breather to the edge breather by using a single fiberglass yarn; if AIRTECH Air Weave SS is used for surface breathers, it may extend to connect with the edge breather.
3. Teflon FEP film extends to centerline of edge breather.
4. Pressure plate, 0.20 in. thick minimum.
5. Surface breather required unless pressure plate is used, then breather is not required.

Figure 1.- Standard bagging procedure.

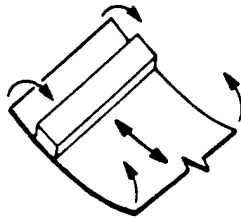
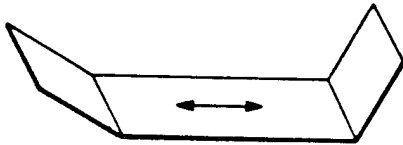


1. Apply 25 in. H_g vacuum minimum.
2. Apply 85 ± 15 psig pressure to laminates.
3. Vent vacuum bag to atmosphere when autoclave pressure reaches 20 psig.
4. Start heat cycle.
5. At completion of heating cycle, when laminate cools to 140°F, release pressure and remove part from autoclave.

Figure 2.- Standard cure cycle.



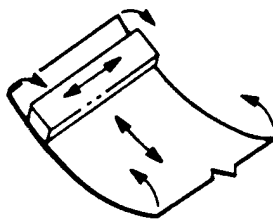
Fold and stack 80 prepreg strips
1.5 by 12 in. with fiber orientation
as indicated by arrow



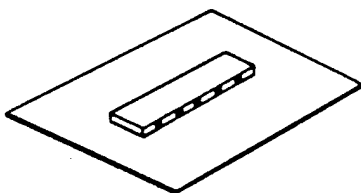
Wrap folded prepreg stack with
6- by 24-in. prepreg sections



Wrap stack in longitudinal direction
with 3 strips of 1 3/4- by 20-in.
aluminum foil



Wrap stack in transverse direction
with 3 strips of 6- by 20-in.
aluminum foil



Perforate 1- by 2-cm holes in aluminum
foil on both sides of stack at five
places evenly spaced; wrap stack with
Kapton sheet

Figure 3.- Prepreg resin sampling specimen.

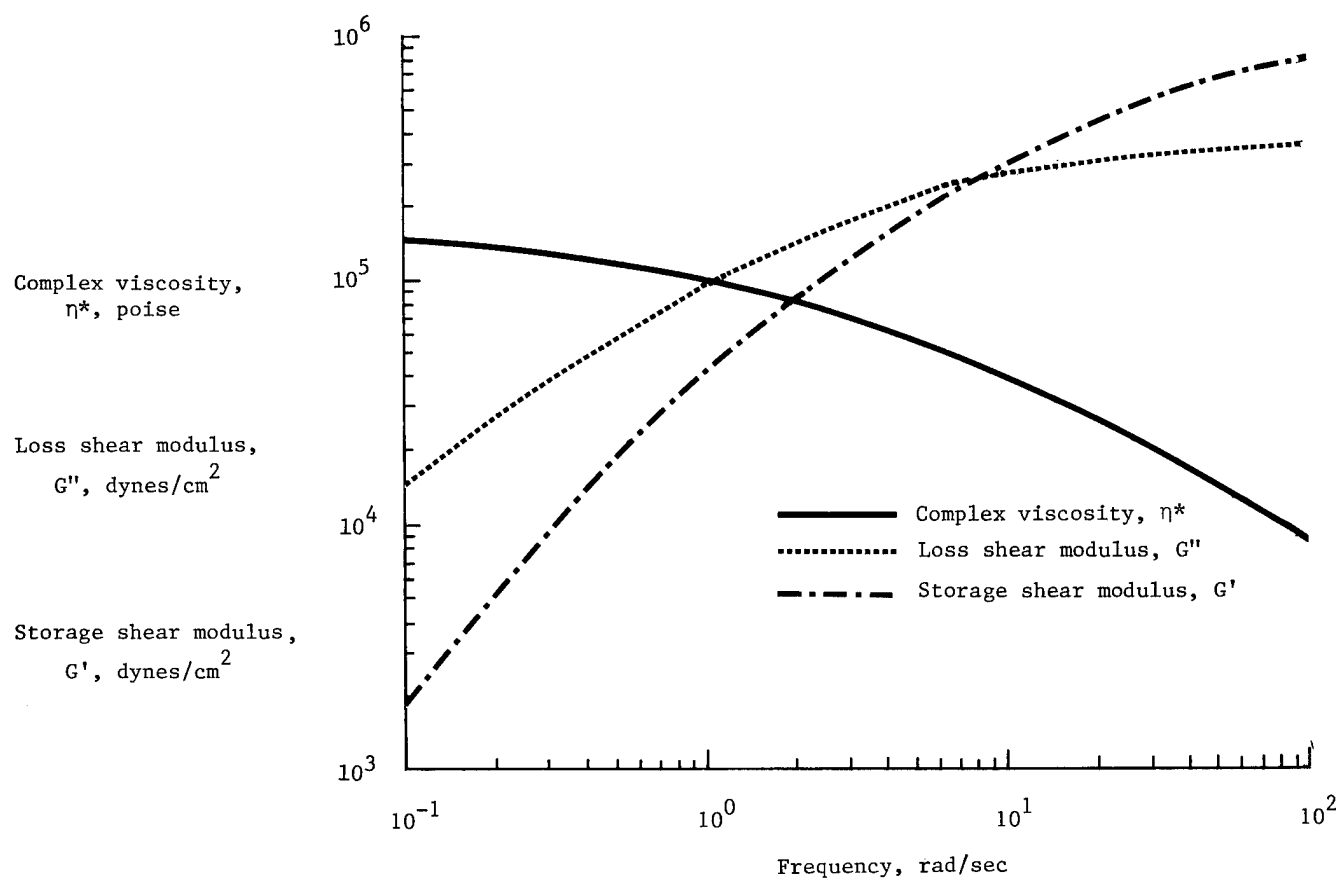
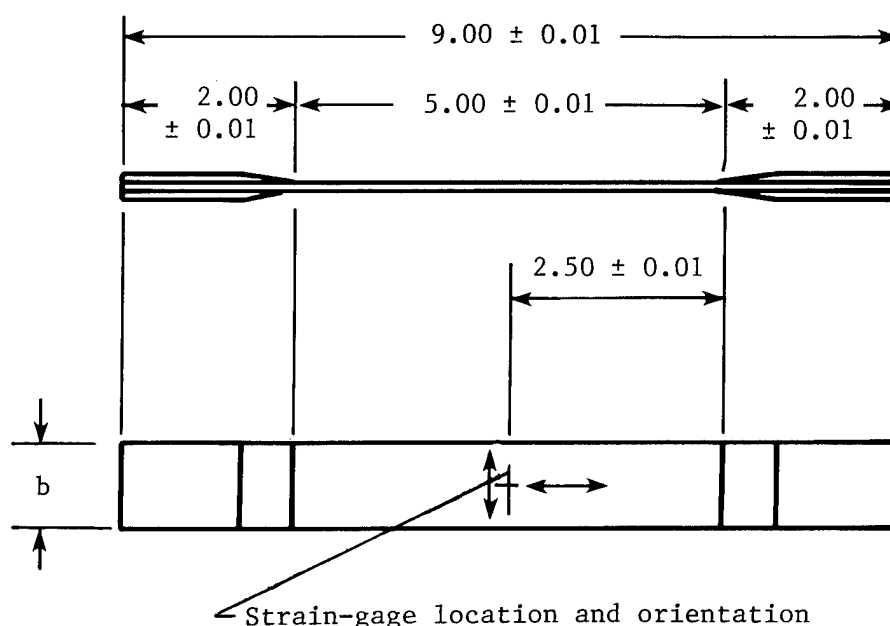


Figure 4.- Calibration data for dynamic mechanical rheometer. Dynamic rate sweep of poly(dimethylsiloxane) at 26°C.



Test	Test method	Ply orientation	Specimen width, b, in.
Tension strength and modulus	Section B.6	(0) ₈	0.500 ± 0.007
Shear modulus	Section B.12	[±45] _{3s}	1.00 ± 0.01

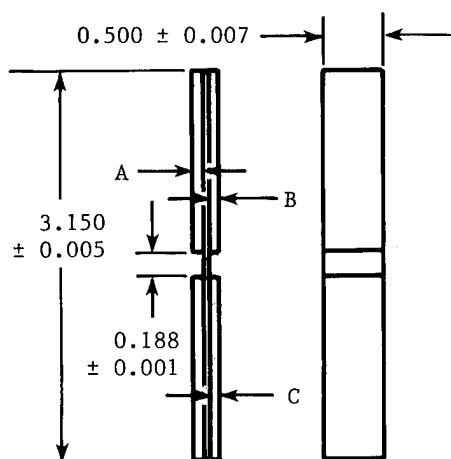
FABRICATION

1. Specimen edge parallel and perpendicular requirements shall be as specified in paragraph B.2.3.
2. Edge finish shall be 32/ in accordance with MIL-STD-10A.
3. Specimen loading tabs shall be fabricated from 5 plies of fiberglass/epoxy prepreg style 181 (1851 or 7781). Taper is achieved by dropping one ply per 0.10 inch or by machining. Prior to bonding tabs, prepare specimen and tab surfaces by hand sanding (No. 150 grit sandpaper) or sandblasting. Clean surface thoroughly with acetone or MEK. Bond tabs to specimen using AF-132, Narmco Metlbond 1133 adhesive, or equivalent.

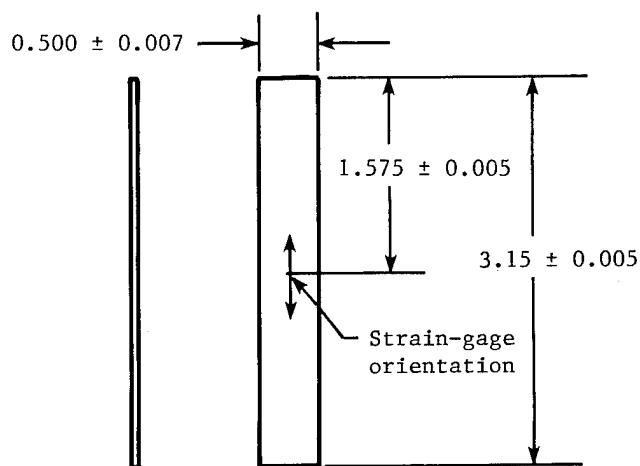
INSTRUMENTATION

1. For the requirements of section B.6, either a longitudinal strain gage or a suitable extensometer may be used to measure longitudinal strain.
2. For the requirements of section B.12, longitudinal and transverse strain gages shall be mounted as shown in the drawing.
3. Locate strain gages adjacent to specimen centerline as indicated on drawing. Strain gage axis shall be aligned within 0.5° of specimen longitudinal or transverse centerline.

Figure 5.- Tension and shear test specimen. All dimensions are in inches.



Compression strength specimen



Compression modulus specimen

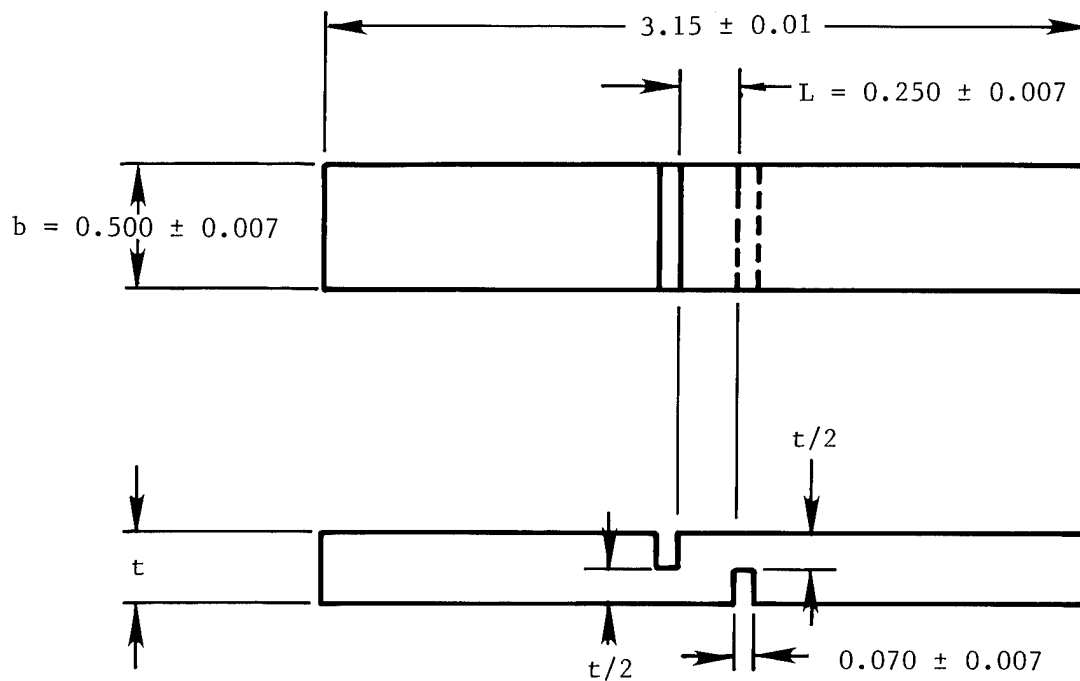
FABRICATION

1. Laminate orientation: $(0)_8$
2. Specimen edge parallel and end perpendicular requirements shall be as specified in paragraph B.2.3.
3. Edge finish shall be 32/ in accordance with MIL-STD-10A.
4. Specimen loading tabs shall be fabricated from the same graphite/resin prepreg as the specimen, 12 plies thick, with the 0° fiber direction parallel to the longitudinal axis within $\pm 1^\circ$. Prior to bonding tabs, prepare specimen and tab bonding surfaces by hand sanding (No. 150 grit sandpaper) or sandblasting. Clean surface thoroughly with acetone or MEK. Bond tabs to specimens with 250°F cure adhesive for room temperature and -100°F testing. Bond tabs to specimens with 350°F cure adhesive for 200°F testing.
5. Tab thickness tolerances: $A = B \pm 0.010$
 $B = C \pm 0.001$

INSTRUMENTATION

1. Either back-to-back strain gages or a suitable extensometer shall be used to measure longitudinal strain on the modulus specimens. Locate strain gage or extensometer on specimen centerline as shown. Strain gage axis shall be aligned within 0.5° of the specimen longitudinal centerline.

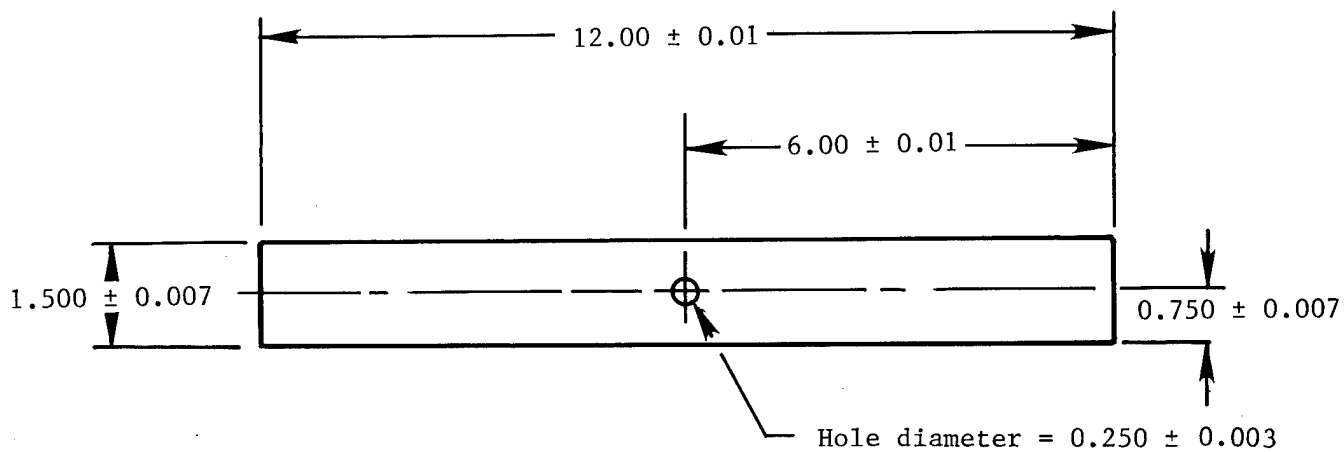
Figure 6.- Compression test specimens. All dimensions are in inches.



FABRICATION

1. Laminate orientation: $[45/0/-45/90]_{6S}$
2. Specimen edge parallel and end perpendicular requirements shall be specified in paragraph B.2.3.
3. Edge finish shall be $32\sqrt{}$ in accordance with MIL-STD-10A.
4. Cut specimen notches with an abrasive wheel such that:
 - Notch depth = $t/2^{+0.010}_{-0.000}$ and notch penetrates centerply of laminate
 - Notch corner radius = $0.005^{+0.001}_{-0.000}$

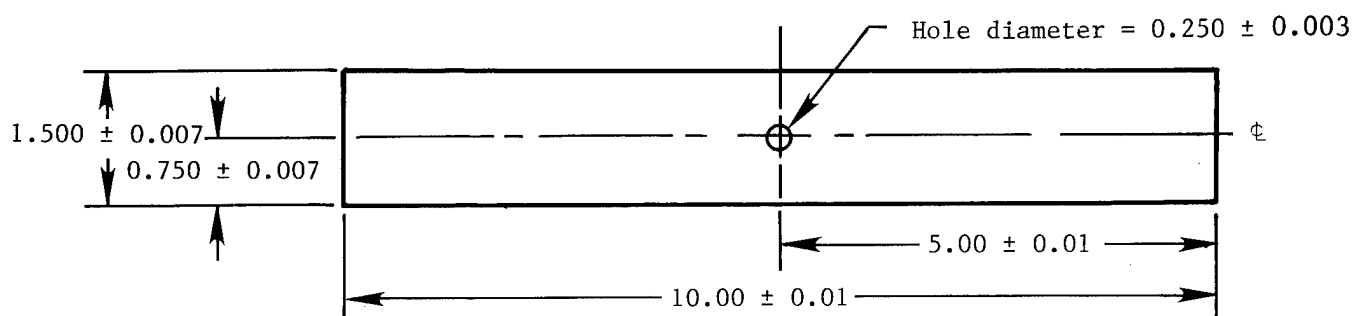
Figure 7.- Compression interlaminar shear specimen. All dimensions are in inches.



FABRICATION

1. Laminate orientation: $[45/0/-45/90]_{2s}$
2. Specimen edge parallel and end perpendicular requirements shall be as specified in paragraph B.2.3.
3. Edge finish shall be $32\sqrt{}$ in accordance with MIL-STD-10A.
4. Drill and/or ream hole as specified in paragraph B.9.2.

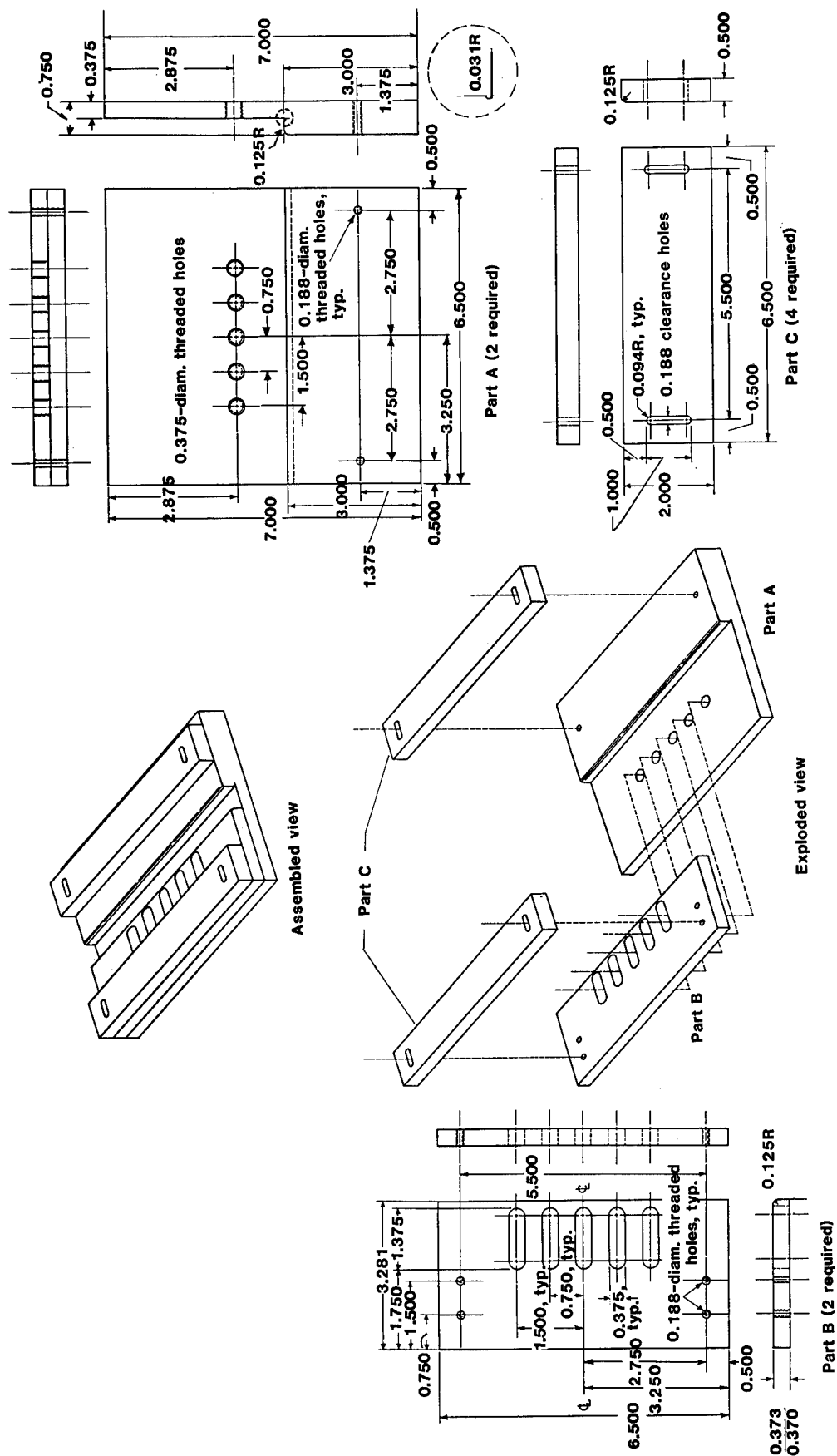
Figure 8.- Open-hole tension specimen. All dimensions are in inches.



FABRICATION

1. Laminate orientation: $[45/0/-45/90]_{2s}$
2. Specimen edge parallel and end perpendicular requirements shall be as specified in paragraph B.2.3.
3. Edge finish shall be $32/\sqrt{}$ in accordance with MIL-STD-10A.
4. Drill and/or ream hole as specified in paragraph B.10.2.

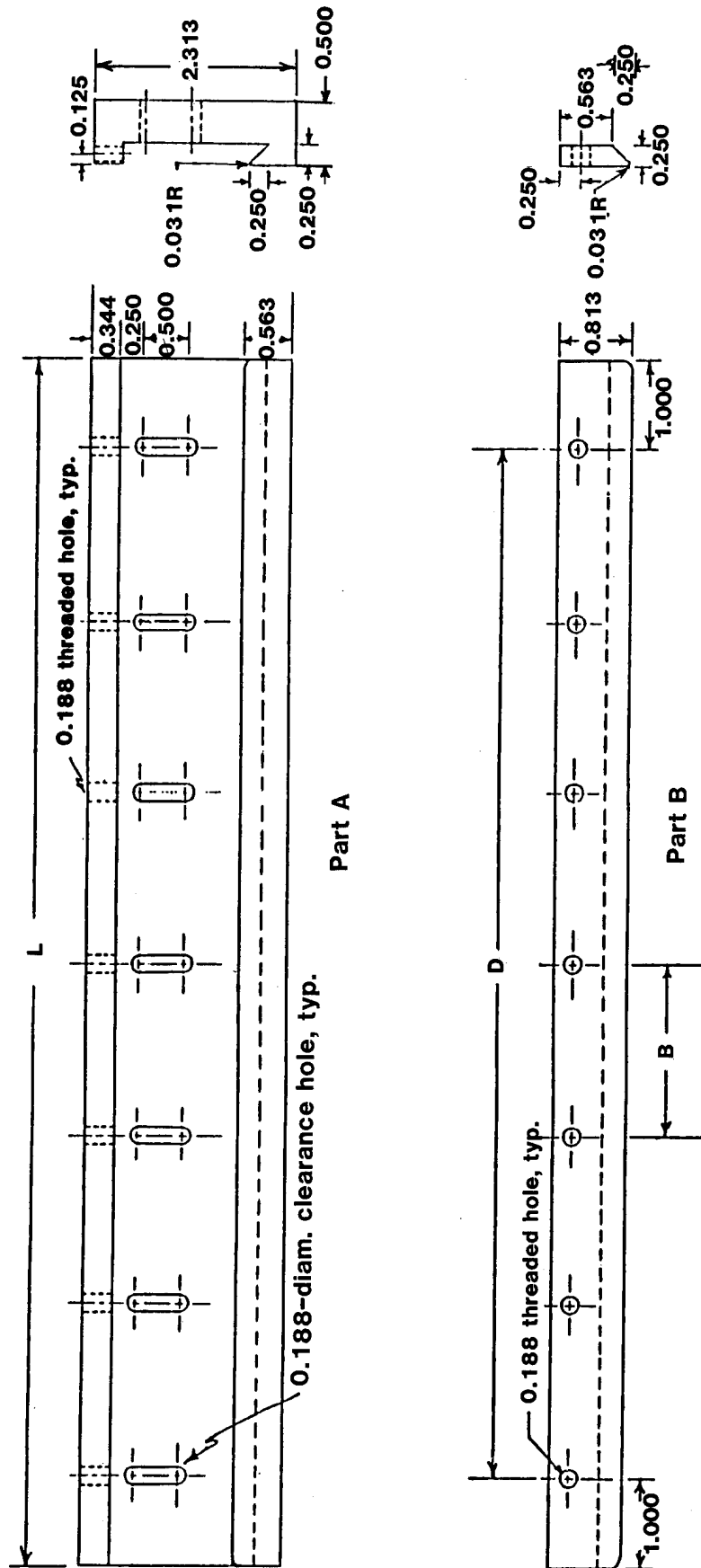
Figure 9.- Open-hole compression specimen. All dimensions are in inches.



(a) Loading plate, top and base, parts and assembly.
Material: 17-4 PH stainless steel.

Figure 10.- Compression test fixture. All dimensions are in inches.

Two needed of each length (both parts)
Rounds 0.313R



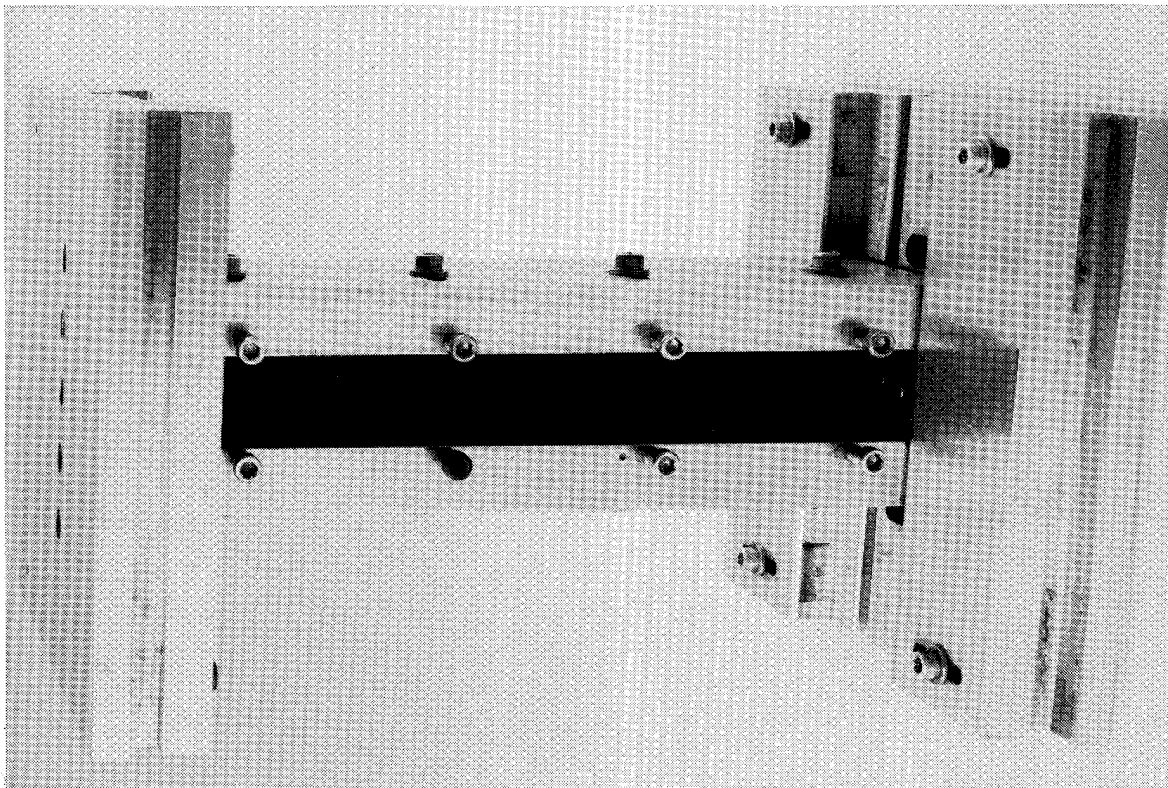
Center bolts and clearance holes equally spaced for each length

For $L = 13.750$, $D = 11.750$, $B = D/6$ or $B = 1.958$ (7 holes total)

For $L = 27.625$, $D = 25.625$, $B = D/13$ or $B = 1.971$ (14 holes total)

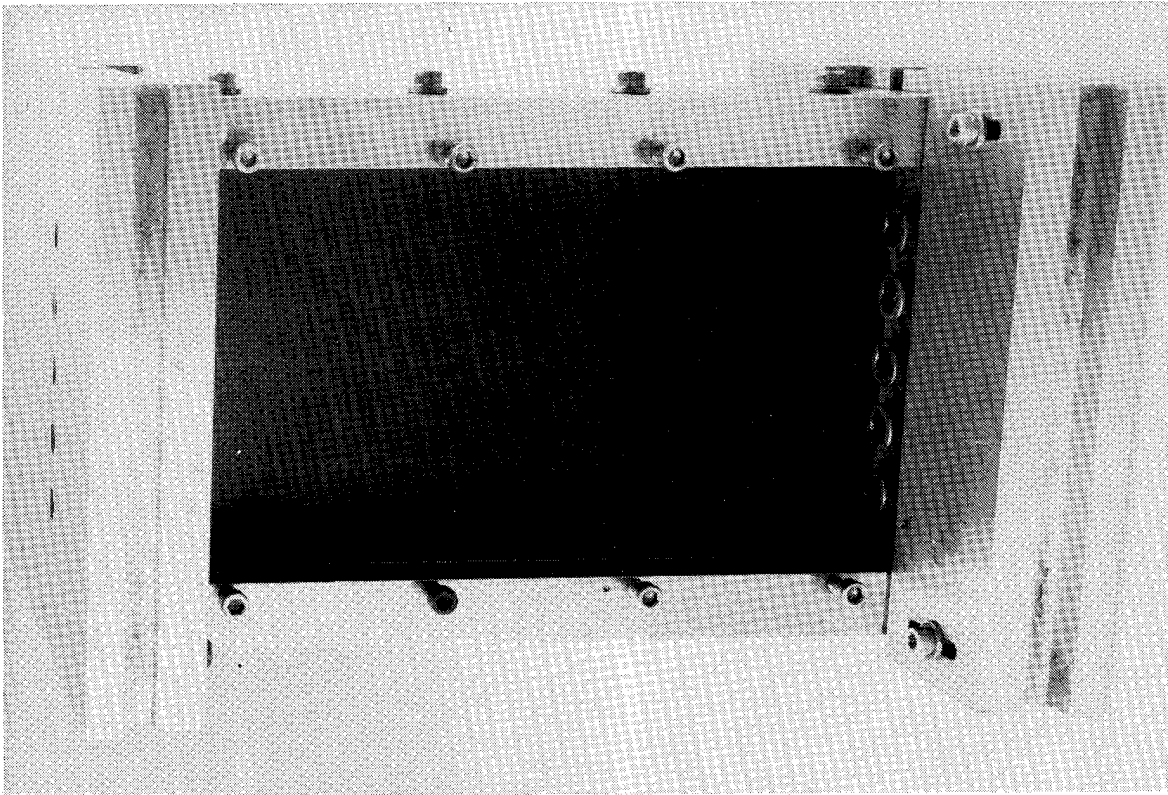
(b) Side supports.

Figure 10.- Continued



L-85-108

(c) Open-hole compression test setup.



L-85-109

(d) Compression after impact test setup.

Figure 10.- Concluded.

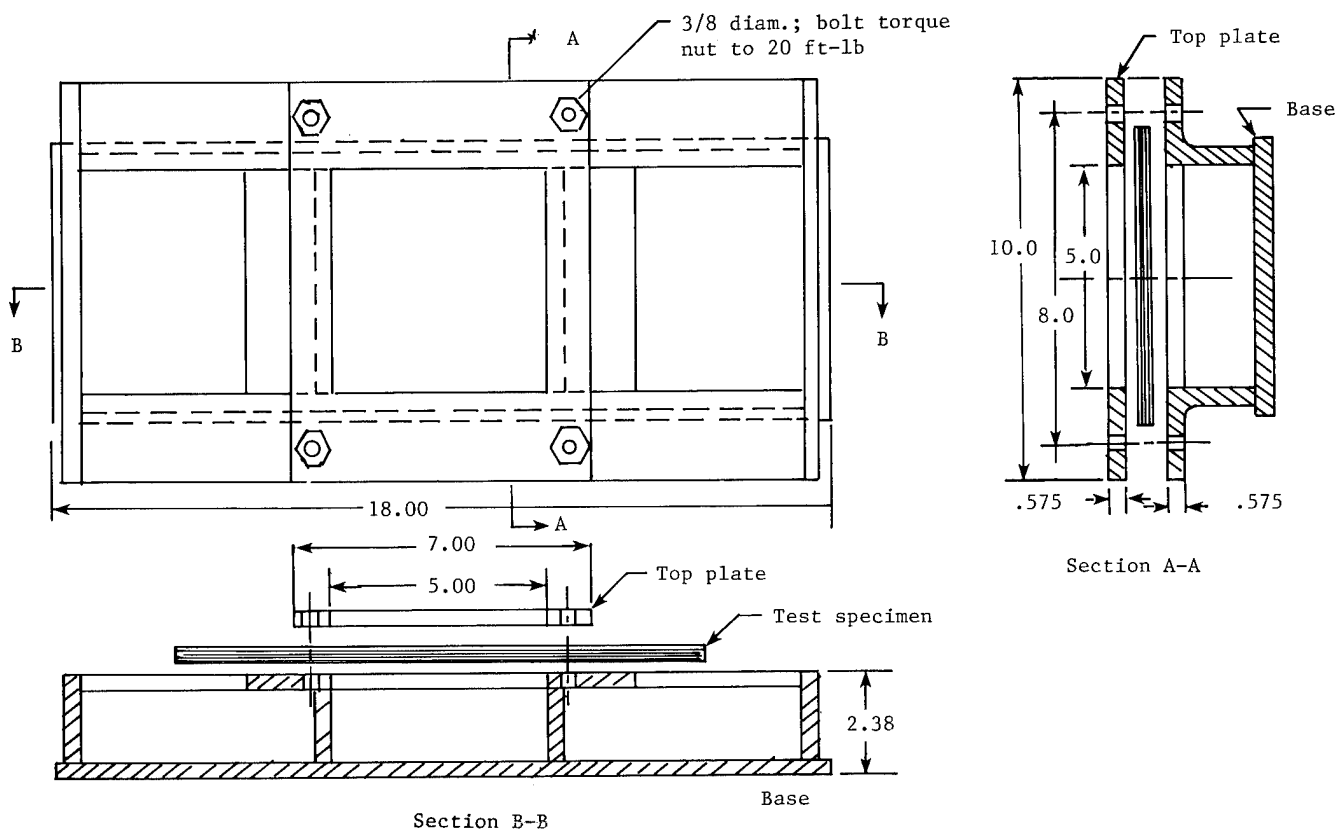
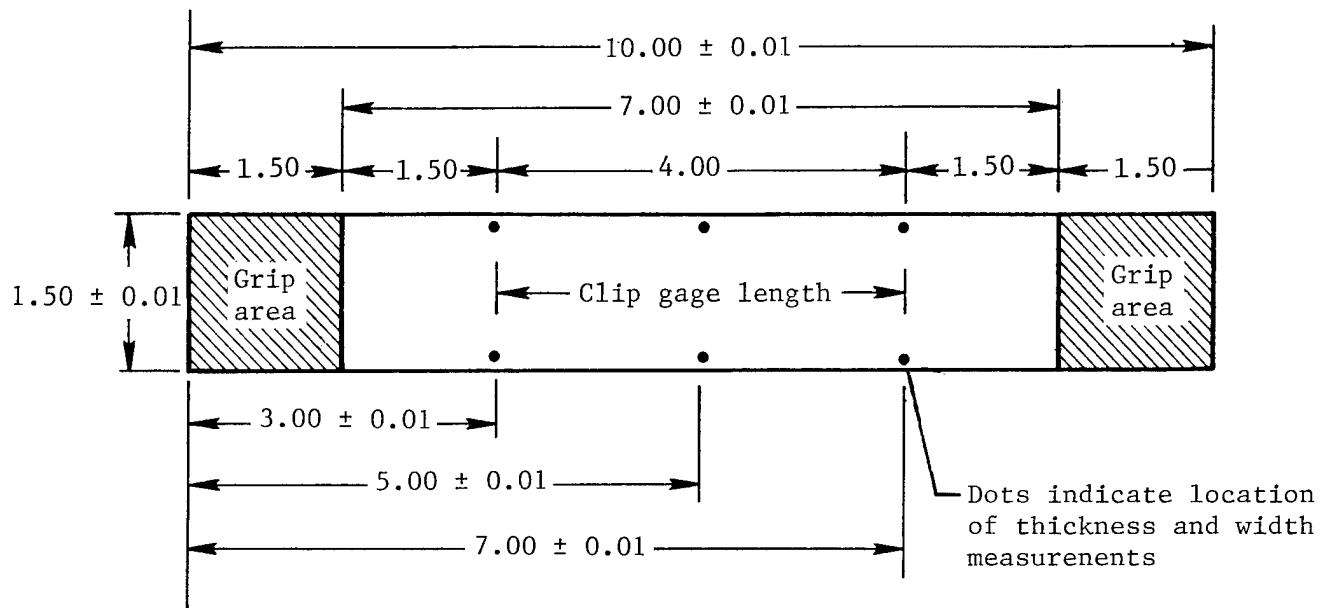


Figure 12.- Impact support fixture. Material: 17-4 PH stainless steel; all dimensions are in inches.



FABRICATION

1. Laminate orientation: $[\pm 30/\pm 30/90/\overline{90}]_s$
2. Specimen edge parallel and end perpendicular requirements shall be as specified in paragraph B.2.3.
3. Edge finish shall be $32/\text{in}$ in accordance with MIL-STD-10A.

Figure 13.- Edge delamination tension specimen. All dimensions are in inches.

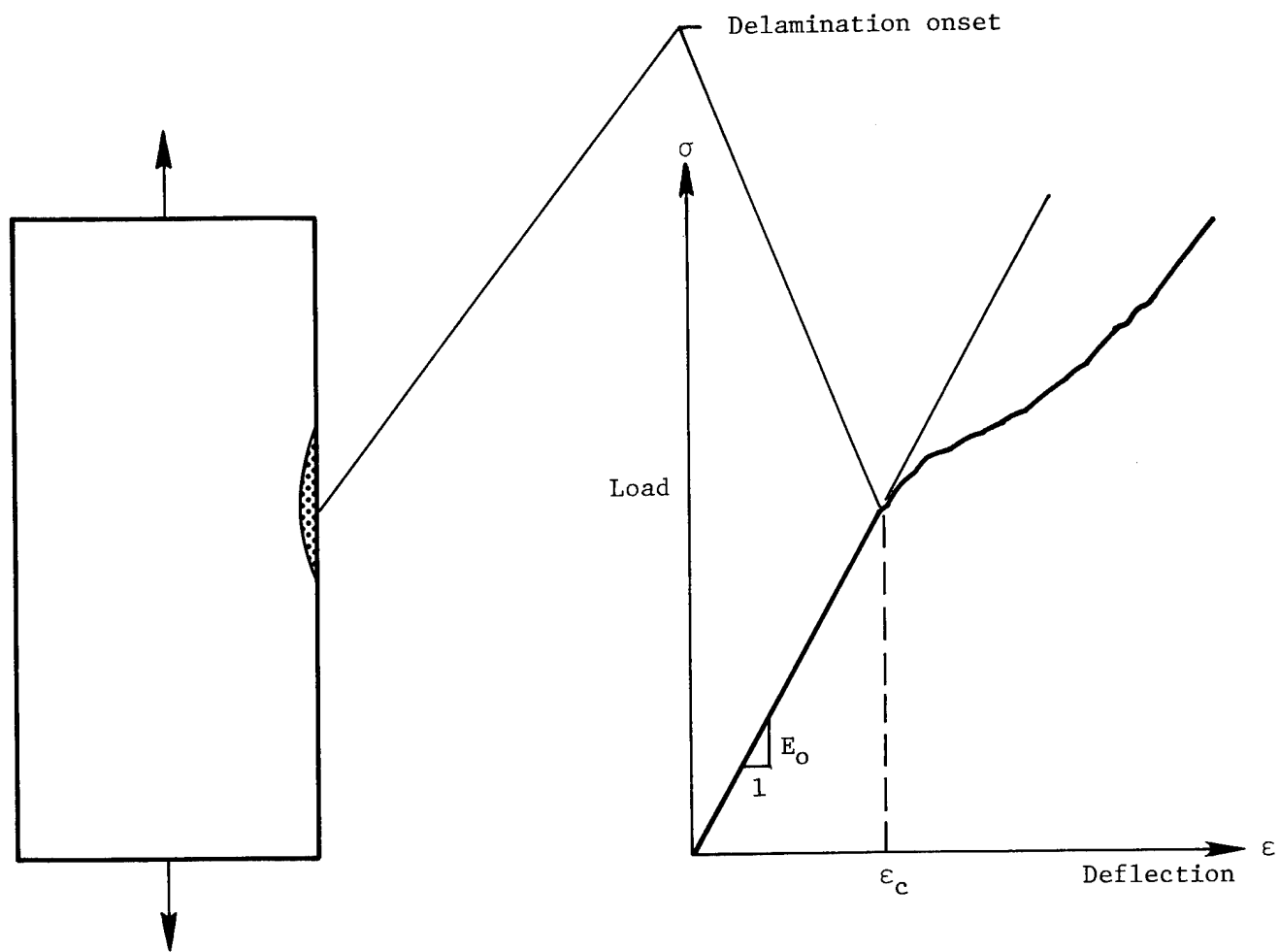


Figure 14.- Load deflection diagram for critical edge delamination determination.

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4. Title and Subtitle NASA/Aircraft Industry Standard Specification for Graphite Fiber/Toughened Thermoset Resin Composite Material				5. Report Date June 1985	
				6. Performing Organization Code 534-06-13	
7. Author(s) Compiled by ACEE Composites Project Office				8. Performing Organization Report No. L-15946	
9. Performing Organization Name and Address NASA Langley Research Center Hampton, VA 23665				10. Work Unit No.	
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12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, DC 20546				13. Type of Report and Period Covered Reference Publication	
				14. Sponsoring Agency Code	
15. Supplementary Notes					
16. Abstract A standard specification for a selected class of graphite fiber/toughened thermoset resin matrix material has been developed through joint NASA/Aircraft Industry effort. This specification has been compiled to provide uniform requirements and tests for qualifying prepreg systems and for acceptance of prepreg batches. The specification applies specifically to a class of composite prepreg consisting of unidirectional graphite fibers impregnated with a toughened thermoset resin that will produce laminates with service temperatures from -65°F to 200°F when cured at temperatures below or equal to 350°F. The specified prepreg has a fiber areal weight of 145 g/m ² . The specified tests are limited to those required to set minimum standards for the uncured prepreg and cured laminates, and are not intended to provide design allowable properties. Qualification and subsequent use of a material through this specification does not constitute or imply endorsement by NASA.					
17. Key Words (Suggested by Author(s)) Composite materials Material qualification Material acceptance				18. Distribution Statement Unclassified - Unlimited Subject Category 24	
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