Recommended Criteria and Guidelines for the Development of a Material Specification For Carbon Fiber/Epoxy Unidirectional Prepregs to be Used on FAA Certified Structures
### Technical Report Documentation Page

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Peter Shyprykevich
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Appendix A – Future Needs
1.0 Introduction

This document recommends guidance and criteria for the development of material specifications for carbon fiber reinforced materials to be used on aircraft structures. These recommendations were prepared by a team of industry experts that have extensive experience with material specifications, part processing, qualification programs and design allowables. Starting with Section 3.0 of this document, the sections parallel the typical sections found in a material specification. A list of areas needing improvement and enhancement is given in Appendix A.

The purpose of this report is to establish a definitive set of recommendations to guide the development of new and revised composite prepreg material specifications. This is intended to advance the work that has been done through previous FAA programs such as Advanced General Aviation Transport Experiment [AGATE]. These programs have established methodology for developing design allowable data, control of the establishment of them and sharing the database developed. In the current work, a generalized approach to the development of a shared composite material database is proposed. It is intended to remove the restrictions placed on those methods developed for General Aviation to allow a broader market to utilize the shared database.

This document can also be used to develop an industry approach such that the following goals can be achieved:

- Greatly reduce the number of material and process specifications for identical composite material systems.
- Develop property databases that uniquely define a given material
- Establish material batch testing and process monitoring sufficient to minimize variability and preclude property changes over time.
- Reduce costs through common documentation and shared databases of basic material properties

Background

Unlike structural parts that use metallic materials in the manufacturing process, the material properties of a composite are manufactured into the structure as part of the fabrication process. Therefore, it is essential that material and process specifications used to produce composite materials contain sufficient information to ensure that critical parameters in the fabrication process are identified to facilitate production and adherence to standards in the final engineered part. Due to the wide variety of composite aircraft structures now emerging for certification at the same time composite applications are expanding for other industries, control of the materials is rapidly becoming a vital issue with respect to the overall assurance of safety.

In recent years, NASA, Industry and the Federal Aviation Administration [FAA] have worked together to develop a cost-effective method of qualifying composite material systems by sharing central material qualification databases such as Mil-Handbook-17 and AGATE. Through these shared databases a manufacturer can select an approved composite material system to fabricate parts and perform a smaller subset of testing to a specific application. For materials to be accepted into these shared databases, it is required that the raw materials be manufactured in accordance with a material specification which impose control of the key physical, chemical...
Recommended Guidelines for CFRP Tape Prepreg Material Specifications

and mechanical properties, and processed in accordance with a process specification that controls key processing parameters.

Steady growth in the use of composites has continued in transport aircraft and rotorcraft. General aviation has emerged recently with the growth of new composite aircraft and composite material applications in primary structures. Several new composite aircraft are undergoing the certification process and many more aircraft are currently undergoing the design and development processes that take advantage of composite materials for primary structural applications. With this growth of composite applications, certifications issues have emerged with respect to the exact philosophy of quality control and quality assurance methods needed to guarantee a safe and consistent material supply.

1.1 Proposed Specification Format

For consistency and standardization purposes, a general format for composite prepreg material specifications should be followed. The following is a recommended format.

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2.0 Development of Material Controls

This section outlines the differences between qualification to an industry standard versus an end user material specification, the responsibilities of an end user related to establishing the suitability of a particular material and part fabrication process, and the steps required to establish a material specification, “qualify” the material, demonstrate equivalency of the end-user part fabrication process to the qualification database, and to perform on-going batch acceptance testing.

2.1 Industry Material Specification

The recommendations in this document are particularly applicable to a material specification that will be released as an industry standard. The process envisioned for such a specification would involve the development of an initial material database by the supplier. The material supplier is the actual manufacturer of the prepreg. The material supplier controls the incoming raw materials and processes to produce a consistent product. A distributor does not produce the final product: it repackages large batches of prepreg into smaller units to be resold to end users.

The initial material database will result from testing conducted to an FAA approved and witnessed test plan and will provide sufficient data to establish the material specification requirements and batch acceptance limits. In this scenario, the material supplier would calculate proposed specification requirements, and bring the material test results along with the proposed specification requirements to an industry committee for approval. The committee would review the data, and finding it satisfactory, would approve a slash sheet to the base specification. This slash sheet would uniquely define the material, and will include specific property requirements and batch acceptance limits.

With this industry specification approach, the traditional process of “qualifying” a material to an existing material specification (containing either target requirements or requirements from a previously qualified material) is no longer applicable. The specification requirements will be determined based on the properties of the specific material. Specifications will be issued for any material for which the minimum dataset and process control and documentation requirements have been met.

Material Qualification is defined as the process of evaluating a material, using a prescribed series of tests, to establish its characteristics as produced by the baseline manufacturing process, and using the evaluation results to define new material specification requirements. A material qualification is performed initially for a new material; it is repeated in part or in whole when manufacturing processes need to be reevaluated. The scope of a previous qualification may also need to be expanded when requirements for additional characteristics are either added to an existing application or result from using the material in a new application. For material characteristics that have never been qualified, a material specification will typically contain "target" values in place of requirements; in this case, following qualification, the target values are updated to requirements based on the evaluation results.

It will be the responsibility of the material supplier to continually test and evaluate the material to fully populate the database on an ongoing basis to ensure that the material has not changed.
2.2 End-User Material Specification

The traditional approach in the aerospace industry is for each end-user to prepare their own material and process specifications. After qualification of a material to these specification, the end user then purchased the prepreg and manufactured a part. This approach has involved the qualification of a material to an existing material specification (either in draft or released form). In many cases, multiple, different, materials have been "qualified" to the same set of specification requirements, even though the properties of the materials may be been significantly different. This approach can result in less than desirable levels of control over the properties of the individual materials qualified to the specification.

Since it may be several years before the industry standard specifications are in place, this document includes recommendations for the preparation of end-user material specifications, and the qualification of materials to these specifications, to meet the goals stated above.

2.3 End-User Responsibilities

It is the responsibility of the end user to "qualify" the material for use in a particular aircraft/rotorcraft application. This process by the end user may involve additional tests to characterize the material and/or validate specific design details. These tests will be conducted to fully populate the certification database and, then on a reduced frequency basis, to ensure that the design allowables stay current. The end user is also responsible for validating that multiple materials are acceptable for the application, if this is so desired.

If the end user decides to use the property information in databases previously developed for the material in the end user’s certification project, the end user will need to perform “equivalency” tests to demonstrate an “understanding” of the associated material and process specifications. This “understanding” essentially involves demonstrating that the end user can produce test panels and specimens that give results that are statistically equivalent to the values in the existing database. The equivalency testing should first be done using the material supplier’s baseline cure process prior to attempting to demonstrate equivalency to an end user’s alternate cure cycle.

Further, it is the responsibility of the end user to validate that any deviations from the baseline laminate cure cycle given in the material and process specifications that are desired for the end user’s production process do not result in statistically significant changes to design allowables derived from data using the baseline process. Successful demonstration of “equivalency” to an existing shared database will allow the end use to avoid additional material qualification tests and to use the material allowables derived from the shared database.

Much of this document is limited to recommendations and guidance on the development of a material specification. Additional guidance on the end user’s responsibilities for validating the use of a specific material with a specific fabrication process can be found in the accompanying report on the development of a process specification (DOT/FAA AR/02-XX).

2.4 Material Qualification Process – Industry Material Specification

The following outlines the process of material qualification, end-user demonstration of equivalency for their part fabrication process, and the on-going batch acceptance testing. It assumes that an industry base material specification is in place, and that the new material will “qualify” to that specification. This process also assumes that the FAA will issue a Technical
Recommended Guidelines for CFRP Tape Prepreg Material Specifications

Standard Order (TSO) that approves the material specification and material characterization data for use on aircraft certification programs.¹

In this process, it is anticipated that the material supplier will:

- Develop a new material for potential market requirements or want to qualify an old material to an industry specification.
- Stabilize the prepreg production process parameters through laboratory trials.
- Establish and document the baseline cure process parameters for the material. This cure process will be used to generate the qualification database.
- Submit a qualification test plan to the FAA ACO office responsible for the material supplier. The ACO will review and approve the test plan. This step is part of the TSO issuance process.
- Perform the minimum “qualification” tests, as defined in the base material specification. A minimum of three (3) batches of prepreg material will be produced for the manufacture of test panels. Panel fabrication and testing will be witnessed by an FAA DMIR and DER, respectively.
- Upon completion of the testing, calculate proposed material batch acceptance limits, material equivalence limits and B-basis allowables values. Specification limits will be calculated using procedures documented in the DOT/FAA AR/00-47 and the industry material specification; allowables will be calculated using procedures documented in Mil-Handbook-17.
- Send the test data and proposed specification limits to the industry committee responsible for the industry material specification. The committee will review the data, and upon a finding of satisfactory data, will issue a slash sheet to the base material specification for the specific material.
- Submit the test data, material specification slash sheet, baseline cure process documentation and the prepreg process control document (PCD) to the FAA ACO. The ACO will review the data and specification values, and if acceptable, will issue a TSO for the material. The TSO will approve the use of the material specification and allowables data for future aircraft certification projects.
- Based on marketing requirements, develop a test plan for additional material characterization tests. Submit the test plan to the FAA ACO office responsible for the material supplier. The ACO will review and approve the test plan. Perform the

¹ The formal regulatory process to implement a TSO for composite raw materials has not been completed. The implementation of standard industry practices for review and acceptance of composite databases, which have associated material and process specifications, may also preclude the need for a TSO. Under such circumstances it is still envisioned that the FAA will be involved in the acceptance processes, as has been the case to date for AGATE shared databases.

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tests per the test plan. Panel fabrication and testing will be witnessed by a FAA DMIR and DER, respectively.

- Upon completion of the testing, calculate proposed material equivalence limits for the additional tests, along with B-basis allowables values.

- Send the test data and proposed specification limits to the industry committee responsible for the industry material specification. The committee will review the data, and upon a finding of satisfactory data, will revise the slash sheet with the material equivalence requirements for the additional test conditions.

- Submit the additional test data and revised material specification slash sheet to the FAA ACO. The ACO will review the data and specification values, and if acceptable, will revise the TSO for the material.

- Submit the material and accompanying data, material specification and allowables to a potential end-user.

At this point the end user will:

- Develop a test plan for the equivalency tests specified in the base material specification to verify that the user’s process(es) for fabricating test panels and production parts can produce equivalent properties as compared to the baseline test database developed by the material supplier.

- Submit the test plan to the FAA ACO office responsible for the end user. The ACO will review and approve the test plan. Perform the tests per the test plan. Panel fabrication and testing will be witnessed by an FAA DMIR and DER, respectively.

- Submit the test data for the ACO, and compare the results of the equivalency tests to the material equivalence limits published in the material specification slash sheet. If all test data meets the requirements then the end-user can use the material allowables developed from the supplier’s baseline database in the design and certification of the end-user’s structure.

- If equivalency is not demonstrated, the end-user can either a) modify their fabrication process and re-run the equivalency test program, or b) perform additional tests to develop design allowables specific to their fabrication process.

- In either case, the end-user will perform additional design verification and certification tests to validate specific configurations and design details of their structure. Upon completion of all certification tests and analyses, the FAA ACO will approve the design, which specifies the materials, for a Type Certificate.

Once the end-user’s design is approved for production, the material supplier will then:

- Produce material to the PCD referenced on the industry material specification slash sheet and approved by the corresponding TSO.

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• Perform the batch acceptance tests specified in the material specification.

• For the first N (TBD) batches of production prepreg, perform the additional “periodic” material characterization tests as specified in the material specification. At the completion of the testing for the Nth batch, recalculate the material batch acceptance limits and material equivalency limits.

• Send the batch test data and revised specification limits to the industry committee responsible for the industry material specification. The committee will review the data, and upon a finding of satisfactory data, will revise the slash sheet with the new limits. Submit the batch test data and revised material specification slash sheet to the FAA ACO. The ACO will review the data and specification values, and if acceptable, will revise the TSO for the material.

• For every subsequent M (TBD) batches after the first N batches, perform the additional “periodic” material characterization tests as specified in the material specification. At the completion of the testing for the Lth (TBD) set of periodic test batches, recalculate the material batch acceptance limits and material equivalency limits.

• Send the batch test data and revised specification limits to the industry committee responsible for the industry material specification. The committee will review the data, and upon a finding of satisfactory data, will revise the slash sheet with the new limits. Submit the batch test data and revised material specification slash sheet to the FAA ACO. The ACO will review the data and specification values, and if acceptable, will revise the TSO for the material.

Once the prepreg material is in production, should it be necessary to make a “major change” to the material (such as new or modified raw ingredients, a facility resite, new prepreg line, etc.) the material supplier will:

• Develop a test plan for the equivalency tests specified in the base material specification and DOT/FAA AR/00-47 to verify that the material produced with the revised process will have equivalent properties as compared to the baseline test database originally developed and updated by the material supplier.

• Submit the test plan to the FAA ACO office responsible for the end user. The ACO will review and approve the test plan. Perform the tests per the test plan. Panel fabrication and testing will be witnessed by a FAA DMIR and DER, respectively.

• Submit the test data to the ACO, and compare the results of the equivalency tests to the material equivalency limits published in the material specification slash sheet. If all test data meets the requirements then the revised prepreg process will be approved. The TSO will be revised to reference the new PCD revision letter. Submit the test data to the industry committee. The committee will revise the slash sheet to reference the new PCD revision letter.

• If equivalency is not demonstrated, the prepreg production process change will not be allowed. The supplier has the option of running additional tests to validate the
change, or of foregoing the change. If the former option is selected, the supplier will work with the ACO, industry specification committee and all end-user’s of the material to develop the required test plan.

2.5 Material Qualification Process – End-User Material Specification
The following outlines the process of material qualification, end-user demonstration of equivalency for their part fabrication process, and the on-going batch acceptance testing. It assumes that the new material will “qualify” to a material specification written and maintained by an end-user. If also assumes that all qualification and design related testing will be performed by the end-user.

In this process, it is anticipated that the material supplier will:

• Develop a new material for potential market requirements.
• Stabilize the prepreg production process parameters through laboratory trials.
• Establish and document the baseline cure process parameters for the material. This cure process will be recommended to potential end-users.
• Submit the material and accompanying data, material specification and allowables to a potential end-user.

At this point the end-user will:

• Submit a qualification and design allowables test plan, and draft material specification to the FAA ACO office responsible for the end-user. The ACO will review and approve the test plan.
• Perform the qualification and allowables tests, using the end-user’s planned production cure process. A minimum of three (3) batches of prepreg material will be used for the manufacture of test panels. Panel fabrication and testing will be witnessed by an FAA DMIR and DER, respectively.
• Upon completion of the testing, calculate proposed material batch acceptance limits and B-basis allowables values. Specification limits will be calculated using procedures documented in the DOT/FAA AR/00-47; allowables will be calculated using procedures documented in Mil-Handbook-17.
• Submit the test data, material specification, and cure process documentation to the FAA ACO. The ACO will review the data and specification values, and if acceptable, will approve the use of the material specification and allowables data for the end-user’s aircraft certification project.
• The end-user will perform additional design verification and certification tests to validate specific configurations and design details of their structure. Upon completion of all certification tests and analyses, the FAA ACO will approve the materials and design for a Type Certificate.
Once the end-user’s design is approved for production, the material supplier will then:

- Produce material to the PCD referenced on the end-user’s material specification.
- Perform the batch acceptance tests specified in the material specification.
- For the first N (TBD) batches of production prepreg, perform the additional “periodic” material characterization tests as specified in the material specification. At the completion of the testing for the Nth batch, recalculate the material batch acceptance limits and material equivalency limits.
- For every subsequent M (TBD) batches after the first N batches, perform the additional “periodic” material characterization tests as specified in the material specification. At the completion of the testing for the Lth (TBD) set of periodic test batches, recalculate the material batch acceptance limits and material equivalency limits.

Once the prepreg material is in production, should it be necessary to make a “major change” to the material (such as new or modified raw ingredients, a facility resite, new prepreg line, etc.) the material supplier will:

- Develop a test plan for the equivalency tests specified in the material specification and DOT/FAA AR/00-47 to verify that the material produced with the revised process will have equivalent properties as compared to the baseline test database originally developed by the end-user. The material supplier will have to do this for each and every end-user that uses the material.
- Submit the test plan to the FAA ACO office responsible for each end user. The ACOs will review and approve the test plan. Perform the tests per the test plan. Panel fabrication and testing will be witnessed by an FAA DMIR and DER, respectively.
- Submit the test data to the ACOs, and compare the results of the equivalency tests to the material equivalency limits published in the material specification slash sheet. If all test data meets the requirements then the revised prepreg process will be approved.
- If equivalency is not demonstrated, the prepreg production process change will not be allowed. The supplier has the option of running additional tests to validate the change, or of foregoing the change. If the former option is selected, the supplier will work with the ACOs, and all end-user’s of the material to develop the required test plan.

3.0 Scope

This section of the material specification should include a general description of the product and its area of application to guide the prospective user. General temperature use limits and cure

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conditions should be stated. If the product is to be supplied in various resin contents, cured ply thickness and product forms, i.e. wide tape and slit tape, then a system must be defined to distinguish the various types, classes, grades etc. Those products to be controlled by this specification are required to be listed here.

It is recommended that:

- **Form** - defines the basic material form; tape, slit tape
- **Type** - defines the resin content of the prepreg
- **Grade** - defines the areal weight of the prepreg
- **Class** - defines the specific fiber used in the prepreg (fiber type, manufacturer, facility)

### 4.0 Applicable Documents

This section of the material specification should include appropriate drawings, specifications, standards and methods that will form a key part of the specification. The material supplier is encouraged to use existing documentation available to the public and developed or approved by industry organizations. Test methods can come from ASTM and SACMA. Government recommended processes and procedures should be referenced and followed such as DOT/FAA/AR-00/47, Material Qualification and Equivalency for Polymer Matrix Composite Material Systems and MIL-HDBK-17, Composite Material Handbooks. Supplier internal documents such as special test procedures should be referenced and included in the process control document (PCD).

Examples include:

- ASTM C393-00 Standard Test Method for Flexural Properties of Sandwich Constructions
- ASTM D792 Specific Gravity (Relative Density) and Density of Plastics by Displacement
- ASTM D2344 Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short-Beam Method
- ASTM D2734 Void Content of Reinforced Plastics
- ASTM D3039 Tensile Properties of Polymeric Matrix Composite Materials
- ASTM D3171-99 Standard Test Method for Constituent Content of Composite Materials

******************************************************************************

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<table>
<thead>
<tr>
<th>ASTM Standard Number</th>
<th>Standard Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>D3532-99</td>
<td>Standard Test Method for Gel Time of Carbon Fiber-Epoxy Prepreg</td>
</tr>
<tr>
<td>D3800-99</td>
<td>Standard Test Method for Density of High-Modulus Fibers</td>
</tr>
<tr>
<td>D4018-99</td>
<td>Standard Test Methods for Properties of Continuous Filament Carbon and Graphite Fiber Tows</td>
</tr>
<tr>
<td>D3479-96</td>
<td>Standard Test Method for Tension-Tension Fatigue of Polymer Matrix Composite Materials</td>
</tr>
<tr>
<td>D5379</td>
<td>Shear Properties of Composite Materials by V-Notched Beam Method</td>
</tr>
<tr>
<td>D5467-97</td>
<td>Standard Test Method for Compressive Properties of Unidirectional Polymer Matrix Composites Using a Sandwich Beam</td>
</tr>
<tr>
<td>D5687-95</td>
<td>Standard Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation</td>
</tr>
<tr>
<td>D5766-95</td>
<td>Standard Test Method for Open Hole Tensile Strength of Polymer Matrix Composite Laminates</td>
</tr>
<tr>
<td>D5961-01</td>
<td>Standard Test Method for Bearing Response of Polymer Matrix Composite Laminates</td>
</tr>
<tr>
<td>D6264-98</td>
<td>Standard Test Method for Measuring the Damage Resistance of a Fiber-Reinforced Polymer-Matrix Composite to a Concentrated Quasi-Static Indentation Force</td>
</tr>
<tr>
<td>D6484-99e1</td>
<td>Standard Test Method for Open-Hole Compressive Strength of Polymer Matrix Composite Laminates</td>
</tr>
<tr>
<td>D6641-01e1</td>
<td>Standard Test Method for Determining the Compressive Properties of Polymer Matrix Composite Laminates Using a Combined Loading Compression (CLC) Test Fixture</td>
</tr>
<tr>
<td>D6742-01</td>
<td>Standard Practice for Filled-Hole Tension and Compression Testing of Polymer Matrix Composite Laminates</td>
</tr>
<tr>
<td>D5528-01</td>
<td>Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites</td>
</tr>
</tbody>
</table>
### ASTM D6115-97
Standard Test Method for Mode I Fatigue Delamination Growth Onset of Unidirectional Fiber-Reinforced Polymer Matrix Composites

### ASTM D6415-99e1
Standard Test Method for Measuring the Curved Beam Strength of a Fiber-Reinforced Polymer-Matrix Composite

### ASTM D6671-01
Standard Test Method for Mixed Mode I-Mode II Interlaminar Fracture Toughness of Unidirectional Fiber Reinforced Polymer Matrix

### ASTM D3544-76(1996)
Standard Guide for Reporting Test Methods and Results on High Modulus Fibers

### ASTM D3878-01
Standard Terminology Composite Materials

### ASTM D6507-00
Standard Practice for Fiber Reinforcement Orientation Codes for Composite Materials

### ASTM E1309-00
Standard Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases

### ASTM E1434-00
Standard Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases

### ASTM E1471-92(1998)

### ASTM E168
General Techniques of Infrared Quantitative Analysis

### ASTM E1252
Standard Practice for General Techniques for Quantitative Infrared Analysis

### SACMA SRM 1R-94
Compressive Properties of Oriented Fiber-Resin Composites

### SACMA SRM 18R-94
Glass Transition Temperature (Tg) Determination by DMA of Oriented Fiber-resin Composites

### SACMA SRM 20R-4R
High Performance Liquid Chromatography of Thermoset resins

### SACMA SRM 22R-94
Resin Flow of Preimpregnated "B" Staged Material.

### SACMA SRM 23R-94
Resin Content and Fiber Areal Weight of Thermoset Prepreg with Destructive Technique.

### SACMA SRM 25R-94
Onset Temperature and Peak Temperature for Composite System resins Using Differential Scanning Calorimetry (DSC)

---

**5.0 Definitions**

This section of the material specification should include definitions for terms or abbreviations that are used with some degree of frequency. The definitions are to provide clarity for the supplier and the procurer. Material properties, quality, and defects will need to be defined such that batches following the original qualification will be made the same. For example, unitape defects such as slitter litter, fuzz balls, creases, foreign material, dry spots, gaps, alignment, splices, edge deviation from a straight line should have a specific definition. Since the ability to

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be laid up and cured dependably into a part can be affected by the degree of advancement of the resin, storage life, out life, and handling life must be clearly defined. Where possible, definitions from industry standards such as MIL-HDBK-17 should be used.

6.0 Material Requirements

It is strongly recommended that the prepreg material supplier establish a process control document (PCD) that documents all aspect of the material fabrication, lists all raw material ingredients, defines key process parameters and establishes statistical process control (SPC) procedures and requirements. The PCD will be maintained by the material supplier and should be made available for review by material end-users and certification agencies. It should be referenced by the material specification.

6.1 Prepreg Ingredient and Process Requirements

This section of the material specification should include requirements that define the specific raw ingredients and processes for producing the prepreg. For this specification it is assumed that the resin mixing, filming, and prepregging is conducted without solvent (hot melt prepreg process). A similar methodology can be applied to solvated systems.

The prepreg specification should include only a single resin formulation and a single fiber class. The specification can include multiple forms, types and grades.

The key process parameters for filming and prepregging must be established and documented in the PCD. Process conditions should have maximum and minimum limits that are monitored, recorded and reviewed per the SPC procedures.

The resin filming process limits should not only control the chemical signature of the resin, but film quality aspects such as fish eyes, mottling, and film thickness down the length and across the width. Limits should be defined for film thickness, including a target thickness value and range.

The backing paper and film must be controlled like any other critical raw material.

The prepreg process limits will result in control of the resin advancement as well as degree of resin impregnation, puckers, gaps etc. The production of prepreg with consistent handling characteristics such as tack and drape, with consistent thickness, and with consistent resin content and fiber areal weight are critical for subsequent part manufacture.

The capability of the prepreg to be cured within the time and temperature limits specified by the manufacturer must be demonstrated. Film and prepreg made to conditions that result in minimal thermal history should be combined with the shortest, lowest temperature, fastest ramp time cure cycle claimed by the manufacturer for characterization testing. Conversely, prepreg made to conditions that result in maximum thermal history (except maximum storage life, which is not practical) should be combined with a cure cycle with the longest time, slowest ramp rate, and maximum temperature. Validation will be through the testing of key elevated temperature mechanical properties after fluid and water exposure. Physical tests such as DMA (dynamic mechanical analysis) can be used to evaluate degree of cure. DSC (differential scanning calorimetry) is another useful technique for assessing if the resin is fully reacted.

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6.1.1 Resin Requirements

This section of the material specification should include requirements that define the specific chemical and physical properties of the resin.

The resin used in the manufacture of prepreg to this specification shall be an epoxy based thermosetting polymer. The designation of the resin must be specified and must refer to only one combination of ingredients processed via one mixing regime. The resin composition and mixing process will be defined prior to qualification. Proposed limits of ingredient weighing accuracy and process times and temperatures will be validated through physical and chemical testing. Mixing process includes premix step(s), final mix step(s), ingredient handling, mixed resin cooling, mixed resin storage, mixed resin reheating and feed process to the filming step. The limitations of in process tests must be understood. Current industry practice is to use resin viscosity and/or gel time as quick methods to validate the resin mixing step. These quick tests typically allow a wide range of acceptable values and may not be an accurate measure of resin consistency. If blending of mixed resin batches is to be allowed, the nature and type of blending will be validated through chemical analysis. Blending of blended resins is not preferred unless it can be demonstrated that there is no impact on prepreg out-life and cure kinetics. Process limits defined and validated by the above tests must be documented in the PCD or specification.

Resin components and their manufacturers must be specified in the PCD. If multiple sources of an ingredient are planned, the use of each component must be validated through chemical analysis. Raw ingredients can be blended as long as storage and handling requirements for the raw materials are met. Documentation must establish that departures from the raw material manufacturer’s recommendations for handling and storage are valid.

Resin requirements that measure key attributes of the final mix or premix(es) shall be identified in the PCD. These may include gel time, viscosity, and analytical signature such as IR or HPLC. In addition, the resin cure kinetics and rheology should be well characterized. It is recommended that the resins used for the initial material data base be used to derive a cure model using isothermal and temperature ramped DSC. These same resins should also be used to evaluate the rheology of the resin as it is subjected to the proscribed cure cycles. If possible, the resin samples should be taken from the beginning and end of a film set to capture the accumulation of processing effects and thermal history. It would be valuable to conduct the kinetic and rheological studies on resins made to the limits of ingredient ratios allowed by the mix procedure and weighing errors. At one extreme, the curative would be at its lowest concentration and the epoxies at their highest concentration. At the other extreme, the curative would be at its highest concentration and the epoxies at their lowest.

The diffusion and absorption of moisture and environmental fluids in the cured resin should be evaluated via moisture uptake versus time and by plasticization leading to lowering of elastic modulus and Tg. The resistance of the cured resin to thermal microcracking over the range of use temperatures and cycles, both as cured neat resin and cured composite should be assessed.

All of the above mentioned data should be documented by the material supplier, and be made available to potential users of the material and to the industry committee responsible for the industry specification for the material, if applicable. Table 1 summarizes the resin property data discussed above.
Table 1: Recommended Minimum Set of Neat Resin Properties

<table>
<thead>
<tr>
<th>Resin Property</th>
<th>Test Condition/Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity</td>
<td>TBD</td>
</tr>
<tr>
<td>Gel Time</td>
<td>TBD</td>
</tr>
<tr>
<td>IR</td>
<td>TBD</td>
</tr>
<tr>
<td>HPLC (ingredient ratios)</td>
<td>TBD</td>
</tr>
<tr>
<td>Cure Kinetics</td>
<td>TBD</td>
</tr>
<tr>
<td>Rheology</td>
<td>TBD</td>
</tr>
<tr>
<td>Moisture Diffusion</td>
<td>TBD</td>
</tr>
<tr>
<td>Moisture Absorption</td>
<td>TBD</td>
</tr>
<tr>
<td>Thermal Induced Microcracking</td>
<td>TBD</td>
</tr>
</tbody>
</table>

6.1.2 Fiber Requirements

This section of the material specification should include requirements that define the specific chemical and physical properties of the fiber.

The carbon fiber to be used in the prepreg should be purchased to a separate, existing fiber material specification. The carbon fiber specification must uniquely define the fiber type, manufacturer and facility. Attributes that affect prepreg processing such as fuzz and spreadability should also be specified.

The carbon fiber must be capable of meeting the requirements of the prepreg specification when impregnated with the specified resin and processed per the specified cure procedure. The prepreg specification must define the specific fiber to be used; if multiple fiber sources are to be included in the prepreg specification, then each fiber source must have a unique prepreg designation (e.g. Class). It is not acceptable for a prepreg specification to refer to the carbon fiber by a trade name without specifying the specific manufacturer and facility that produces the fiber.

The fiber specification must define the average values and ranges for all fiber mechanical and physical properties including tensile strength, tensile modulus and density. The prepreg specification must specify the fiber form and tow count (e.g. 12K flat tow). The fiber sampling plan and test methods for fiber properties and quality must be documented in the PCD.

The fiber size material, method of size application, and size content are considered to be an integral part of the carbon fiber. The fiber sizing is to be unique and there should be a shelf life requirement if the sizing ages during storage. Changes to the size, application and/or content will require equivalency testing or the establishment of a new material designation.

Fiber batch blending is allowed as long as there is traceability of each fiber batch. For prepreg materials used to establish the initial material database, each prepreg batch must contain a single and unique fiber batch.

Table 2 summarizes the fiber property data discussed above.
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Table 2: Recommended Minimum Set of Carbon Fiber Properties

<table>
<thead>
<tr>
<th>Fiber Property</th>
<th>Test Condition/Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Form</td>
<td>TBD</td>
</tr>
<tr>
<td>Tow Count</td>
<td>TBD</td>
</tr>
<tr>
<td>Size Content</td>
<td>TBD</td>
</tr>
<tr>
<td>Tensile Modulus</td>
<td>TBD</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>TBD</td>
</tr>
<tr>
<td>Density</td>
<td>TBD</td>
</tr>
<tr>
<td>Spreadability</td>
<td>TBD</td>
</tr>
</tbody>
</table>

6.2 Uncured Prepreg Requirements

6.2.1 Uncured Prepreg Properties

This section of the material specification should include requirements for uncured prepreg physical and chemical properties. Table 3 outlines the minimum set of uncured prepreg properties needed for characterization of the material. The requirements and test methods for each property shall be documented in the specification.

Testing should be conducted on prepreg made to the extreme upper and lower limits of the resin mixing, filming and prepregging processes in order to capture the variability expressed by the raw materials and processes used to manufacture the prepreg.

Testing should be conducted alternately on the start and end of spools and should also be conducted on the sides and center of the spool. This is intended to capture down-the-length and across-the-width variability introduced in the filming and prepregging process steps.

Table 3: Recommended Minimum Set of Uncured Prepreg Properties – Prepreg Tape Materials

<table>
<thead>
<tr>
<th>Uncured Prepreg Property</th>
<th>Test Condition/Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber Content, areal weight</td>
<td>TBD</td>
</tr>
<tr>
<td>Resin Content, % by weight</td>
<td>TBD</td>
</tr>
<tr>
<td>Volatile Content, % by weight</td>
<td>TBD</td>
</tr>
<tr>
<td>Flow, % by weight</td>
<td>TBD</td>
</tr>
<tr>
<td>Gel Time, minutes</td>
<td>TBD</td>
</tr>
<tr>
<td>HPLC (ingredient ratios)</td>
<td>TBD</td>
</tr>
<tr>
<td>IR (Ingredients Chemical Signature)</td>
<td>TBD</td>
</tr>
<tr>
<td>Chemical Reactivity and Degree of Advancement via DSC</td>
<td>TBD</td>
</tr>
<tr>
<td>Tack</td>
<td>TBD</td>
</tr>
<tr>
<td>Drape</td>
<td>TBD</td>
</tr>
<tr>
<td>Roll Edge Alignment</td>
<td>TBD</td>
</tr>
<tr>
<td>Prepreg Width</td>
<td>TBD</td>
</tr>
<tr>
<td>Puckers/Pimples</td>
<td>TBD</td>
</tr>
</tbody>
</table>
6.2.2 Roll Characteristics
The roll size, weight, width, core type, and width and length tolerances will be defined and can be specified on a purchasing document.

6.2.3 Visual Defect Limitations and Dimensions
This section of the material specification should include limitations on visual defects in the prepreg. Criteria for continuous defects such as gaps, fiber alignment, and edge alignment must be established and documented in the PCD. Allowable defect limits can be based on generally accepted industry standards. Procedures for closing gaps such as through the use of rollers can be documented in the PCD. Edge alignment can be corrected by re-spooling if procedures are established to control and document material out life.

Criteria for discontinuous defects such as puckers, fuzz balls, splices, foreign material should be defined in the specification. Where surface defects such as foreign material and fuzz balls can be removed by scraping or picking, these procedures can be used if documented.

Procedures for continuous inspection of the prepreg shall be defined in the specification or PCD. It is recognized that since release paper is typically found on one side of the prepreg, only the top surface can be inspected. Should the same defects be detected by the procurer on the bottom of the prepreg when the part is being laid up, the same criteria for allowable defect limits and correction shall be followed.

The specification should require that each prepreg defect outside the allowable limits be identified and marked by a flag positioned at the edge of the material. The type, location and length of each defect shall be recorded for each roll and the record attached to the roll. Defects can be removed by splicing per documented procedures and criteria for maximum number and minimum spacing of splices. The splicing technique must be easily identified by the procurer to avoid incorporation of the splice into a part. The time out of cold storage during defect removal will be recorded and used to adjust the remaining out life.

6.2.4 Storage, Handling and Out-Life
This section of the material specification should include definitions and limitations for storage life under specified conditions, handling life under ambient conditions, and out life capability of laid up material. These requirements should be based on specific test data and/or experience with similar materials.

It is recommended that a tracking policy be implemented at the material supplier to document storage/out life of material from date of manufacture to arrival on dock at the purchaser. Tracking should include resin intermediates, mixed resin, and film.

Any testing to re-establish the acceptance of materials that have been subjected to storage upsets such as a freezer breakdown will need to demonstrate that key cure related attributes are within the normal range.

A distributor should practice the same documentation of storage life and conditions as the material supplier and purchaser. If original packaging is to be opened to allow for respooling into smaller units, the prepreg should be allowed to warm in the unopened package until moisture does not condense on the prepreg. All out life accumulated during warming, respooling, and
Recommended Guidelines for CFRP Tape Prepreg Material Specifications

repackaging will need to be subtracted from that specified by the material supplier, and this information documented to the purchaser.
6.3 Cured Prepreg Property Requirements

This section of the material specification should include requirements for the cured prepreg in a laminate form. These requirements should be based on specific data obtained for the material.

6.3.1 Baseline Cure Process

It is recommended that the material supplier establish a baseline cure cycle to be used to produce laminates for the initial material database and batch acceptance testing. Reasonable tolerances on heat-up rates and time at temperature shall be established and documented. The cure process shall be capable of producing cured laminates of consistent, high quality. This cure cycle should be used for all acceptance testing by the material supplier and purchaser.

6.3.2 Cured Laminate Physical Properties

It is recommended that the material specification include, as a minimum, requirements for the cured laminate physical properties listed in Table 4. The limits and test methods for each property shall be documented in the specification.

It is recommended that these tests be conducted on prepreg from the start and end of rolls, as well as prepreg from the sides and center of rolls.

Table 4: Recommended Minimum Set of Cured Laminate Physical Properties – Prepreg Tape Materials

<table>
<thead>
<tr>
<th>Cured Laminate Physical Property</th>
<th>Test Condition/Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cured Ply Thickness</td>
<td>TBD</td>
</tr>
<tr>
<td>Laminate Density</td>
<td>TBD</td>
</tr>
<tr>
<td>Fiber Volume, % by volume</td>
<td>TBD</td>
</tr>
<tr>
<td>Resin Content, % by volume</td>
<td>TBD</td>
</tr>
<tr>
<td>Void Content, % by volume</td>
<td>TBD</td>
</tr>
<tr>
<td>Glass Transition Temperature, Tg</td>
<td>Room Temp/Ambient Hot/Wet</td>
</tr>
</tbody>
</table>

6.3.3 Cured Laminate Mechanical Properties

A minimum set of mechanical property data will be required to adequately characterize the material and to provide a database for future material equivalency evaluations. The tests must be able to detect changes in the fiber, resin, and the response of the prepreg to the baseline cure process. A subset of these tests is required for acceptance testing for each batch.

In addition to the batch acceptance tests, it is recommended that a second set of tests be run at some frequency on an ongoing basis to keep the test methodology current and to further populate the database (see Section 7.2). The results from these tests would be used to update the material acceptance and equivalency requirements in the specification. They would also assist in detecting material changes or an increase in variability.

The following paragraphs present a series of recommended tests for development of a material property database. It is expected that this database will be developed over time, as the market for the material expands, and specific applications require additional data. The first set of tests represent the minimum tests required to establish a material specification. This test matrix is

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very similar to the AGATE test matrix in DOT/FAA/AR-00/47, and is intended for applications that have simple layup configurations and do not involve mechanically fastened joints or highly loaded structure. An additional set of open hole laminate tests is recommended for inclusion in specifications for materials intended for more general applications.

Further sets of recommended tests for an expanded database are then presented. These tests are optional with regard to inclusion in the material specification, but may be required for the design and certification of an end user’s product. There is a potential for cost savings if these additional tests could be shared amongst several end users. Therefore, for marketing purposes, the material supplier may elect to perform tests to expand the shared database, either by themselves or in conjunction with one or more end users. The expanded database could include the tests recommended below, other design specific tests and/or other environmental related tests (e.g., flammability, moisture diffusion, thermal cycling). In each step of the database development, its utility is limited until more data is collected, but the intent is to let market conditions drive the expansion of the database.

### Recommended Laminate Tests

To establish a material specification, the tests in Table 5a are recommended as a minimum set for material characterization of a prepreg tape material. The additional open hole tests in Table 5b are recommended for materials expected to be used in more general applications that will contain mechanical fastened joints or will be designed with notched laminate properties. The requirements and the test methods for each property shall be documented in the specification.

All of the tests should be robust in that material variability, rather than test variability, will be evaluated. Recommended test methods for each property are given in Chapters 3 through 7 of Volume 1 of Mil-Handbook-17. Moisture conditioning should be conducted per the procedures given in Mil-Handbook-17 and ASTM D5229. Cross-ply [0/90] tension and compression tests are recommended over the traditional unidirectional [0] tests because the [0/90] layup has been demonstrated to be less sensitive to test variables. The ASTM D6641 (CLC) test method is recommended for compression testing due to its superior performance (reduced variability) and lack of requirement for tabs on the test specimens. It is expected that industry material specifications to be developed in the near future will require the use of the cross-ply test configurations.

Batch acceptance tests are recommended in Table 5a (as shown with FL1). These batch acceptance tests are recommended based on the following rationale:

- The room temperature tension test is included to monitor the fiber and fiber-resin interface properties.
- The hot compression test is included to primarily monitor the resin properties.
- The apparent shear strength by short beam test is included to monitor the fiber-resin interface properties.
### Table 5a: Recommended Minimum Set of Cured Laminate Mechanical Properties – Prepreg Tape Materials

<table>
<thead>
<tr>
<th>Layup</th>
<th>Test Type and Direction</th>
<th>Property</th>
<th>Lowest Temperature/Ambient</th>
<th>70F/Ambient</th>
<th>Highest Temperature/Ambient</th>
<th>Highest Temperature/Wet</th>
</tr>
</thead>
<tbody>
<tr>
<td>[0]n</td>
<td>0 Tension</td>
<td>Modulus</td>
<td>3 x 6 FL2</td>
<td>1 x 6</td>
<td>1 x 6</td>
<td>1 x 6</td>
</tr>
<tr>
<td>[0]n</td>
<td>0 Compression</td>
<td>Modulus</td>
<td>3 x 6 FL2</td>
<td>1 x 6</td>
<td>1 x 6</td>
<td>1 x 6</td>
</tr>
<tr>
<td>[90]n</td>
<td>90 Tension</td>
<td>Ultimate Strength and Modulus</td>
<td>3 x 6 FL2</td>
<td>1 x 6</td>
<td>3 x 6</td>
<td>3 x 6</td>
</tr>
<tr>
<td>[90]n</td>
<td>90 Compression</td>
<td>Ultimate Strength and Modulus</td>
<td>3 x 6 FL2</td>
<td>1 x 6</td>
<td>3 x 6</td>
<td>3 x 6</td>
</tr>
<tr>
<td>[90/0]ns</td>
<td>0 Tension</td>
<td>Ultimate Strength and Modulus</td>
<td>3 x 6 FL1</td>
<td>3 x 6</td>
<td>3 x 6</td>
<td>3 x 6</td>
</tr>
<tr>
<td>[90/0]ns</td>
<td>0 Compression</td>
<td>Ultimate Strength and Modulus</td>
<td>3 x 6 FL2</td>
<td>3 x 6</td>
<td>3 x 6</td>
<td>3 x 6</td>
</tr>
<tr>
<td>[+45/-45]ns</td>
<td>In-plane Shear</td>
<td>Ultimate Strength and Modulus</td>
<td>3 x 6 FL2</td>
<td>3 x 6</td>
<td>3 x 6</td>
<td>3 x 6</td>
</tr>
<tr>
<td>[0]n</td>
<td>Short Beam Shear</td>
<td>Ultimate Strength</td>
<td>3 x 6 FL1</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

FL1 - Batch acceptance tests (see section 9.4)
FL2 - Property sampling tests (see section 7.2)

### Table 5b: Recommended Additional Cured Laminate Mechanical Properties for General Applications – Prepreg Tape Materials

<table>
<thead>
<tr>
<th>Layup</th>
<th>Test Type and Direction</th>
<th>Property</th>
<th>Lowest Temperature/Ambient</th>
<th>70F/Ambient</th>
<th>Highest Temperature/Ambient</th>
<th>Highest Temperature/Wet</th>
</tr>
</thead>
<tbody>
<tr>
<td>[45/0/-45/0]ns</td>
<td>Open Hole Tension FL3</td>
<td>Ultimate Strength</td>
<td>3 x 6 FL2</td>
<td>3 x 6 FL2</td>
<td>3 x 6 FL2</td>
<td>3 x 6</td>
</tr>
<tr>
<td>[45/0/-45/90]ns</td>
<td>Open Hole Compression FL3</td>
<td>Ultimate Strength</td>
<td>3 x 6 FL2</td>
<td>3 x 6 FL2</td>
<td>3 x 6 FL2</td>
<td>3 x 6</td>
</tr>
</tbody>
</table>

FL2 - Property sampling tests (see section 7.2)
FL3 - Open hole test configuration: 0.25 inch hole diameter, 1.5 inch width
Recommended Guidelines for CFRP Tape Prepreg Material Specifications

The tests listed in Table 6a are some recommended optional tests for an expanded material database. These tests will provide data for additional material design allowables that are commonly used to design and certify aircraft structures. The recommended test conditions for each test are shown with a checkmark. The number of batches to be tested will depend on the acceptable level of conservatism for the allowable values, on the criticality of the structure for which the data will be used, and on acceptance by the responsible FAA ACO.

Many aircraft applications involve more than solid laminate construction. In Table 6b some tests are recommended for honeycomb sandwich panels. The tests are selected as sensitive indicators of any change in the constituent materials. This application will require that separate specifications exist for the honeycomb and the adhesive used to bond the prepreg to the core.

The recommended optional tests listed in Table 7 are some of those that may be required to show that the material will be suitable for the intended aircraft/rotorcraft application. These include testing of cured laminates after exposure of the laminates to solvents that the part will be subjected to in actual service. Recommended fluids for testing are:

Extended Contact:
- 100 Low Lead Aviation Fuel
- JP-4 Jet Fuel
- MIL-H-5606 Hydraulic Oil
- MIL-H-83282 Hydraulic Oil
- Engine Lubricating Oil MIL-L-7808
- Engine Lubricating Oil MIL-L-23699

Short Duration Contact:
- MEK washing Fluid. ASTM D740
- Polypropylene Glycol Deicer (Type I) MIL-A-8243
- Isopropyl Alcohol Deicing Agent (TT-I-735)

The test method to evaluate solvent sensitivity must to be sensitive to the expected effect on the laminate. Shear tests are best for detecting the effect of solvent exposure on epoxy resins. The solvent exposure and subsequent testing will be conducted at the temperatures expected during service.

It is recommended that fatigue testing be conducted to confirm that the parts will be durable over the expected service life. Post-impact residual strength evaluation for damage tolerance is recommended for primary structure applications.

The recommended test conditions for each test are shown with a checkmark in Table 7. The number of batches to be tested will depend on the acceptable level of conservatism for the allowable values, on the criticality of the structure for which the data will be used, and on acceptance by the responsible FAA ACO.
Table 6a – Optional Cured Laminate Mechanical Properties for Expanded Database – Prepreg Tape Materials

<table>
<thead>
<tr>
<th>Layup</th>
<th>Test Type and Direction</th>
<th>Property</th>
<th>Test Temperature/Moisture Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Lowest Temperature/Ambient</td>
</tr>
<tr>
<td>[45/0/-45/90]ns</td>
<td>Unnotched Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/-45/90/45/-45/0/45/-45/0]ns</td>
<td>Unnotched Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/90/-45/0/0/45/0/-45/0]ns</td>
<td>Unnotched Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/0/-45/90]ns</td>
<td>Unnotched Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/90/-45/0/0/45/0/-45/0]ns</td>
<td>Unnotched Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/-45/90/45/-45/45/-45/0/-45/0]ns</td>
<td>Unnotched Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/0/-45/90]ns FL3</td>
<td>Open Hole Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/-45/90/45/-45/45/-45/0/-45/0]ns FL3</td>
<td>Open Hole Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/90/-45/0/0/45/0/-45/0]ns FL3</td>
<td>Open Hole Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/-45/90/45/-45/45/-45/0/-45/0]ns FL3</td>
<td>Open Hole Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/0/-45/90]ns FL4</td>
<td>Filled Hole Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/90/-45/0/45/0/-45/0]ns FL4</td>
<td>Filled Hole Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/-45/90/45/-45/45/-45/0/-45/0]ns FL4</td>
<td>Filled Hole Tension</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/0/-45/90]ns FL4</td>
<td>Filled Hole Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/90/0/45/0/-45/0/-45/0]ns FL4</td>
<td>Filled Hole Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/-45/90/45/-45/45/-45/0/-45/0]ns FL4</td>
<td>Filled Hole Compression</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/0/-45/90]ns FL5</td>
<td>Single Shear Bearing</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/90/-45/0/45/0/-45/0]ns FL5</td>
<td>Single Shear Bearing</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[45/-45/90/45/-45/45/-45/0/-45/0]ns FL5</td>
<td>Single Shear Bearing</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[0]n</td>
<td>Compression Interlaminar Shear</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
</tbody>
</table>

---

FL3 - Open hole test configuration: 0.25 inch hole diameter, 1.5 inch width
FL4 - Filled hole test configuration: 0.25 inch hole diameter, 100° Tension Head Countersunk Fastener, 1.5 inch width
FL5 - Single shear bearing configuration: 0.25 inch hole diameter, 1.5 inch width, one protruding head fastener, stabilization fixture
<table>
<thead>
<tr>
<th>Layup</th>
<th>Test Type and Direction</th>
<th>Property</th>
<th>Test Temperature/Moisture Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Lowest Temperature/Ambient</td>
</tr>
<tr>
<td>[0/90/90/0/core/0/90/90/0]</td>
<td>Sandwich Long Beam Flexure</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[0/45/90/-45/45/90/45/0]</td>
<td>Sandwich Long Beam Flexure</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[0/45/90/-45/45/90/45/0]</td>
<td>Sandwich Long Beam Flexure, with Open Hole</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[0/45/90/-45/45/90/45/0]</td>
<td>Sandwich Long Beam Flexure, with 30 in-lb impact</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
<tr>
<td>[0/45/90/-45/45/90/45/0]</td>
<td>Sandwich Long Beam Flexure, with 120 in-lb impact</td>
<td>Ultimate Strength</td>
<td>✓</td>
</tr>
</tbody>
</table>
### Table 7: Recommended Tests for Durability and Service Life Confirmation—Prepreg Tape Materials

<table>
<thead>
<tr>
<th>Layup</th>
<th>Test Type and Direction</th>
<th>Property</th>
<th>Test Temperature/Moisture Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>+45/-45</td>
<td>In-plane Shear</td>
<td>Ultimate Strength and Modulus</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[0]n</td>
<td>Mode I Fracture Toughness</td>
<td>G1c</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[0]n</td>
<td>Mode II Fracture Toughness</td>
<td>G2c</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[45/0/-45/90]ns FL3</td>
<td>Open Hole Fatigue, R=-1 (Tension/Compr.)</td>
<td>Fatigue Life</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[45/90/-45/0/0/45/0/0/-45/0]ns FL3</td>
<td>Open Hole Fatigue, R=-1 (Tension/Compr.)</td>
<td>Fatigue Life</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[45/-45/90/45/-45/45/-45/0/45/-45]ns FL3</td>
<td>Open Hole Fatigue, R=-1 (Tension/Compr.)</td>
<td>Fatigue Life h</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[45/0/-45/90]ns</td>
<td>Compression After Impact, 270 in-lbs impact</td>
<td>Ultimate Strength</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[45/0/-45/90]ns</td>
<td>Compression After Impact, 540 in-lbs impact</td>
<td>Ultimate Strength</td>
<td><img src="icon" alt="" /></td>
</tr>
<tr>
<td>[45/0/-45/90]ns</td>
<td>Compression After Impact, 1080 in-lbs impact</td>
<td>Ultimate Strength</td>
<td><img src="icon" alt="" /></td>
</tr>
</tbody>
</table>

FL3-Open hole test configuration: 0.25 inch hole diameter, 1.5 inch width

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7.0 Material Characterization

7.1 Initial Material Qualification
This section of the material specification should include procedures and requirements for initially characterizing the material in order to establish the specification requirements.

Industry Material Specification

In order to develop the information needed to “qualify” a material to an industry specification, tests will be conducted to establish an initial database. The testing can be performed by a material supplier, an end user or an industry consortium (a supplier and multiple users, e.g., AGATE). The results from the testing are used to establish the initial material specification and batch acceptance limits. The decision on whether the specification becomes an industry standard or an end-user proprietary specification is for the developers of the database to determine.

A request for the initial qualification shall be reviewed by the FAA. The organization that will conduct the tests shall submit a test plan, material specification and process specification prior to the actual qualification.

End-User Material Specification

For qualification to an end-user specification, the material supplier and end-user typically will negotiate as to which party will fund and conduct the qualification tests. The end-user will be responsible for submitting a test plan, material specification and process specification to the FAA prior to the actual qualification. The results from the qualification testing are used to establish the initial material specification and batch acceptance limits.

Minimum Level of Testing

It is recommended that the initial material database include the minimum required properties listed in sections 6.2 and 6.3. At the discretion of the developers, the database may include the additional recommended tests listed in sections 0 and any additional tests desired by the prospective purchasers of the material.

A minimum of three different material batches consisting of a minimum of two different fiber batches and three different resin batches shall be used for the initial database.

Laminates for mechanical property data for each batch shall be processed using two independent cure cycles. One cure process shall use material that has the least thermal history (minimum process, storage and out life history) and shall use the fastest, shortest cure cycle. The other cure process shall use material that has the highest thermal history (maximum process and out life) and shall use the slowest, longest cure cycle. The data from the two processing cycles can be considered separate batches when calculating design allowables from the data.
Recommended Guidelines for CFRP Tape Prepreg Material Specifications

The statistical procedures given in DOT/FAA/AR-00/47 and/or Mil-Handbook-17 can be used to calculate the material property equivalency requirements and material batch acceptance limits. When using these procedures, the equivalency requirements shall be calculated using an alpha = 0.05, while the batch acceptance limits shall be calculated using an alpha = 0.01. The equivalency requirements for all tested properties, and the acceptance limits for specified properties shall be listed in the specification. These requirements and acceptance limits shall be established as:

- Maximum average, minimum average and minimum individual values for all strength properties.
- Maximum and minimum average values for all stiffness properties.
- Maximum and minimum average values for thickness, resin content, areal weight, Tg, etc.
- Maximum average values for volatile content, void content, etc.

7.2 Periodic Property Testing

Since the initial material qualification testing is performed on only three batches of prepreg (often containing only two batches each of resin and fiber), and since the qualification batch material is often produced using processes that are not completely representative of full-scale prepreg production, it is strongly recommended that the material specification contain the following requirements:

1. For the first N (TBD – 12?) batches of production prepreg material, the additional periodic property sampling tests listed in Section 0 shall be performed for each batch. After the Nth batch of production material is tested, the material equivalency and batch acceptance requirements will be recalculated, and the specification revised with the updated values.

2. For every subsequent M (TBD – 30?) batches after the first N batches perform the additional periodic testing.

3. Upon accumulation of each additional L (TBD – 10?) sets of test results (either from the periodic testing or from intervening material equivalency testing), the material equivalency and batch acceptance requirements will be recalculated, and the specification revised with the updated values. For those properties tested for each batch of material, the data from all batches shall be included in the recalculation.

The reasons for recommending this additional and periodic testing are:

- The additional data will provide a more robust database (better estimate of the population means and variability’s) for calculating the material batch acceptance and material equivalency requirements. This is expected to result in fewer material batch rejections and fewer failures of material equivalency programs. It is also expected that there will be a greatly reduced chance of the control specimens in an equivalency test program failing the equivalency requirements.

- Provides an on-going validation of structural properties, thereby minimizing the chance of “surprise” changes in material properties.

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Will result in a larger database, thereby providing the potential for higher allowables.

Will result in a database with additional structural properties, thereby potentially reducing the amount of end-user testing.

7.3 Additional Characterization Testing for Specific Design Applications

Depending on the intended application for the material, additional tests at the laminate, element and subcomponent levels may be required to fully characterize the material. This testing would include evaluations of process and configuration variations, such as for cocured sandwich structures. These tests could also include evaluations of solvent resistance, impact damage resistance and residual strength, fracture toughness, and bolted and bonded joint strength.

These tests can be performed at the discretion of the supplier if a common database of properties is desired by the supplier; otherwise the tests can be left to the individual end-users of the material.

8.0 Changes to Qualified Materials

This section of the material specification should include the procedures and requirements for establishing the equivalency of future material data to the baseline database.

Material Equivalency is the process of determining whether two materials or processes are similar enough in their characteristics and properties that they can be used without distinction and without additional evaluation. Statistical tests are used to determine whether data from the same material processed in two different manners are significantly different. Equivalency is limited to the evaluation of minor changes in a material’s constituents, manufacturing process, or to changes in the fabrication (e.g., curing) process used with a material. Two materials that meet the same minimum material specification requirements but have statistically different mean properties are not considered “equivalent”.

Mil-Handbook-17, Volume 1, Section 8.4.1 “Tests for Determining Equivalency Between an Existing Database and a New Dataset for the Same Material” gives statistical procedures which can be used to determine whether there is a statistical difference between the data from the two materials or fabrication processes. For two materials to be truly equivalent, their population means and distributions for every property of interest must be essentially identical. However, in practice, this will almost never be achieved, so engineering judgement will be required when equivalency determinations are necessary.

Since processes and material undergo continual evolution and change, it is necessary to establish that the prepreg remain true and consistent to the original database and allowables. It is the responsibility of the material supplier to conduct testing to demonstrate that the current material, when processed to the baseline process specification will generate composite properties statistically equivalent to the properties of the original materials.

Any material changes that result (or can be expected to result) in a change to the material allowables, or to the acceptance limits shall be considered to be a major change under 14 CFR §FAR 21.611. The following sections describe five levels of material changes and the testing and notification requirements associated with these levels.

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8.1 Level 0 Changes
These are changes that do not effect the material. Some examples of these changes are typographical error corrections to the specification or PCD, changes to the names of incoming materials due to company name changes, and use alternate of storage facility locations using identical storage conditions. No notification to the end users or FAA is necessary for these changes. Level 0 changes are considered to be minor changes under 14 CFR §FAR 21.611.

8.2 Level 1 Changes
These changes are minor changes that have been tested internally at the supplier beyond normal batch acceptance testing on the same or similar material, and have been found not to affect the material. Some examples of Level 1 changes are:

- Changes to backing paper.
- Change in release paper or other process aid.
- Alternate vendor for chemically identical raw materials.
- Changes to packaging methods and materials.

Current end users shall be notified of these changes, but approval is not required by the FAA, or by the end user prior to incorporation. A new revision of the applicable material specification is required. Level 1 changes are considered to be minor changes under 14 CFR §FAR 21.611.

8.3 Level 2 Changes
These are major changes that must be validated through testing conducted under an FAA approved test plan prior to incorporation. Due to the type of change involved, these changes are not subjected to the full equivalency test plan required for a Level 3 change. These changes will require that the material supplier conduct testing to an extent which establishes the requirements listed in the material and process specifications will not change. Some examples of Level 2 changes are:

- Change in feedstock or precursor to resin ingredients.
- Change in feedstock or precursor to fiber ingredients.
- Second source of chemically similar raw materials.
- Changes to test methods that reduce variability.
- Modifications to process equipment or process that do not change key characteristics or key process parameters.
- Addition of new similar equipment.
- Expansion of existing facilities, including start up of additional production facility machines.

The type of change and the testing that demonstrates no significant effect must be documented in the appropriate part of the supplier PCD. It is recommended that side-by-side testing of the original material or method and the new material or method be conducted.

Using the material equivalency requirements contained in the specification, the statistical procedures given in DOT/FAA/AR-00/47 can be used to verify that the data from the altered material is equivalent to the baseline database for the material.
Recommended Guidelines for CFRP Tape Prepreg Material Specifications

A new revision letter for the applicable material specification is required when the change is incorporated. Current end users shall be notified of these changes, and approval is required by the FAA, and by the end users prior to incorporation. End user approval is only required for those users receiving material to the new revision of the process specification. Level 2 changes are considered to be major changes under 14 CFR §FAR 21.611.

8.4 Level 3 Changes

These changes are major changes that are subjected to the full equivalency test program, as defined in DOT/FAA/AR-00/47. Level 4 major changes are those that have the possibility of changing either the part processing characteristics or the cured lamina properties such that there is a shift away from the average values established for the material. A request for approving and witnessing the testing to demonstrate material equivalency will be submitted to the responsible FAA ACO. The request will contain a test plan, a description of the change, and the original material and process specifications. Some examples of Level 3 changes are:

- Change in fiber manufacturing process, size type or size level.
- Change in resin chemical characteristics (e.g. alternate resin ingredient).
- Change in manufacturing site for fiber or resin.
- Change in resin mixing, filming and prepregging equipment, process and key process parameters
- Change in cure cycle (e.g., temperature, dwell time, pressure)
- Change in tack
- Change to/from autoclave to/from vacuum pressure cure
- Change in fiber sizing, finish, or coupling agents
- Change in resin content

Testing to validate major changes will require a minimum of three batches of prepreg containing 3 batches of resin and 2 batches of fiber. The material batch acceptance tests and the property sampling tests listed in Section 0 are the minimum required testing for demonstration of equivalency. In addition, any other critical properties that are expected to be affected by the change must be included in the test plan.

Using the material equivalency requirements contained in the specification, the statistical procedures given in DOT/FAA/AR-00/47 can be used to verify that the data from the altered prepreg is equivalent to the baseline database for the material. If equivalency to the original data cannot be confirmed, then the change will not be allowed, or a new material specification designation will be required for the altered material (see Level 4 changes below).

A new revision letter for the applicable material specification is required. Current end users shall be notified of these changes, and approval is required by the FAA, and by the end users prior to incorporation. End user approval is only required for those users receiving material to the new revision of the process specification. Level 3 changes are considered to be major changes under 14 CFR §FAR 21.611.

8.5 Level 4 Changes

Level 4 changes require a new product identification (new specification designation) and a new qualification test program. Level 3 or lower material changes which fail to demonstrate equivalency will typically be considered Level 4 changes. Some changes will be considered

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Level 4 changes regardless of the results of equivalency results, due their significant potential effect on material properties or on part fabrication processing. Some examples of Level 4 changes are:

- Change in resin composition, or viscosity of major components.
- Change in fiber areal weight (e.g., 145 to 190 gm/m²)
- Change in fiber type (e.g., T300 to AS4)
- Change in fiber manufacturer (e.g., Toray to Amoco).
- Change in type of fabric weave (e.g., plain weave to 8 harness satin)
- Change in nominal number of fibers per tow (e.g., 3000 fibers per tow to 6000)

Because Level 4 changes are considered a new material, existing end users will not be effected unless they elect to purchase the new material. An end user who wishes to use the new material must perform sufficient tests to qualify and certify the use of the material in the intended aircraft/rotorcraft application.

9.0 Quality Assurance

This section of the material specification should define the batch sampling plan and establish which testing will be the responsibility of the material supplier and which will be conducted by the purchaser. Key characteristics such as resin content and carbon fiber areal weight will require SPC control. These key characteristics will be monitored on a continuing basis to build a database and to allow for detection of variations and anomalies.

Material Acceptance is the process of determining, by test and/or inspection, whether a specific batch of material meets the requirements of the applicable procurement specification. ("Material acceptance" is also called "quality conformance" in specifications which conform to U.S. DoD MIL-STD-490/961 practices.) Normally a subset of the material qualification tests are selected and designated as "acceptance tests" (or "quality conformance" tests). These tests should ideally be representative of key material/process characteristics such that significant changes in the test result indicate a potential change in the material. The material specification defines sampling requirements and limits for these acceptance tests. Statistical methods are used to determine the material specification requirements using the qualification data and subsequent production batch data. The sampling requirements for acceptance testing normally vary with the maturity and confidence in the process - larger and more frequent samples are selected when the process has a greater likelihood of changing, and conversely, smaller and less frequent samples are selected when increased process maturity and property stability has been demonstrated. Modern production practices emphasize statistical quality control tools using acceptance testing data (as well as process control tests) to monitor production trends and make real-time (or near-real-time) process corrections.

9.1 Supplier Site Qualification

The prepreg manufacturing site shall demonstrate the capability to conduct raw material testing, final product testing, record maintenance, calibrations and statistical process control. Training programs and records will be in place to assure that personnel are capable of conducting testing, running equipment, and assembling and interpreting test results. Adequate and consistent document control will be demonstrated. Major equipment maintenance and modification records will be available. Appropriate organizational structure will exist to assure that each major function (i.e. operations and quality assurance) can perform their functions.

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9.2 Statistical Process Control

The quality assurance department will maintain the procedures and requirements for statistical process control (SPC) based on key characteristics (KC) and key process parameters (KPP). The KC’s will be those properties detailed in the uncured and cured prepreg material requirement tables. KPP’s are those process parameters that have a significant influence on the KC’s. KPP’s will be determined prior to qualification and be documented in the process control document (PCD). Average values, ranges, limits, and sampling frequency will be established and documented in the PCD.

The procedures used by the quality assurance department to conduct SPC analysis of the KC’s and KPP’s will be documented in the PCD. The procedures used to establish and calculate the control limits will be documented in the PCD. It is expected that control charts will be maintained on the KC’s and will be available for inspection. There shall be an effective program to collect, plot, analyze and act on KC data. It is expected that action will be taken when the criteria for non-random data trends are met. Action will be taken while the data is still within the upper and lower control limits established during the initial data base generation and subsequent production batches.

If a KC is out of control, the cause of variation will be identified and eliminated re-establishing statistical control. The supplier’s quality assurance department will document all corrective actions affecting the process and monitor if the corrective action has been effective.

9.3 Reduced Testing

Reduced testing may be established based on the capability of the KCs and KPPs. Reduced testing will require approval by the FAA and the purchaser(s) prior to being implemented. The reduced testing plan will be documented in the PCD. If KCs are found to be out of control, testing will return to the original level for a period of time until confidence in the control of the material is re-established. The reduced testing may take the form of a lower frequency of supplier testing or a reduction of purchaser testing. A prerequisite for reduced testing is adherence to monitoring of control charts.

9.4 Product Certification

This section of the specification should define the material acceptance testing to be performed by the material supplier and the end-user (purchaser).

9.4.1 Supplier Certification Testing

The material supplier must perform material acceptance testing as designated in Sections 6.2 and 6.3. Certification reports must be prepared for each prepreg batch. The test report must show that the batch meets all of the uncured and cured prepreg requirements. All records for each prepreg batch and the original baseline database shall be kept on permanent file. Records of raw material receiving inspection, in-process materials testing, SPC required by the PCD, and full prepreg batch traceability shall be kept for a minimum period of 10 years. The supplier quality department will review the certification test results prior to shipment to a purchaser. Materials that fail can undergo a review process. Retest or replacement of test data is allowed only if:

a. An abnormality is observed or can be reasonably deduced to have occurred during testing.

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b. Data is a statistically outlier.
c. The test has been conducted on materials that have not been prepared or conditioned properly (e.g. machining errors on laminates)

9.4.2 Purchaser Testing
Purchaser quality department shall perform the specified acceptance tests in Section 6.0 on each prepreg batch. The quality department must review the test results and allow the prepreg to be released to manufacturing only upon satisfactory demonstration that the material meets the specification requirements. The purchaser should hold to the same record keeping requirements and retest criteria as the material supplier.

An end user must conduct the acceptance testing if the material is bought directly from the manufacturer or through a distributor. The original certification testing conducted by the prepreg manufacturer will be made available by the distributor for a specific batch sold to the end user.

In cases where the material has demonstrated a high level of SPC control and capability for the material, it may be possible to reduce and/or eliminate the purchase acceptance testing. The FAA ACO will evaluate requests for reduced purchaser testing on a case-by-case basis. If purchaser testing is reduced or eliminated, then provisions for monitoring the thermal exposure history of each shipment of material between the supplier and end-user (including all transit periods and storage periods at a distributor) will be required.

10.0 Material Test Methods
Recommended test methods for each property are given in Chapters 3 through 7 of Volume 1 of Mil-Handbook-17. In general, ASTM Standard Test Methods are recommended. Deviations from industry standard methods must be clearly detailed in the specification. In the event that purchaser testing is required by the specification, it is recommended that the material supplier and purchaser conduct round-robin test evaluations to reduce test result differences.

It is recommended that all testing be conducted by a lab certified to conduct the tests to the specified methods; this certification applies to supplier, purchaser and/or independent test labs. Certified means the lab follows established policies and procedures such as training of test technicians, having written procedures for performing tests, documenting the dimensional accuracy of the test fixture, and tracing calibration to NIST standards. The specification should define the requirements and procedures for certifying a test lab. It is recommended that for an industry specification, a third-party organization be used to certify the test labs.

11.0 Test Panel Fabrication
The baseline cure process to be used to produce test panels for initial database and subsequent acceptance and property sampling tests should be clearly defined in the material specification. Both the material supplier and end users must be capable of performing the cure process on a routine basis. Process aids such as vacuum bag material, vacuum sealant, breathers, caul plates and edge dams must be defined in the PCD. Acceptable nominal values and ranges for temperature ramp rates and holds must be defined.
12.0 Packaging and Shipping
The product must have suitable identification and the packaging and handling during shipping must result in the product being capable of its full handling and out-life when received by the purchaser.

If repackaged by a distributor, the new packaging must be functionally equivalent to the original packaging and the labeling requirements met. Any decrease in storage life, out-life, and handling life must be documented by the distributor.

12.1 Material Identification
The batch number and roll number must be on two labels, one on the inside of the core, the other on the outside of the shipping wrapper. The label should also include the material designation, name of manufacturer, specification number, and date of manufacture.

12.2 Interleaf or Release Layer
The prepreg release film or paper should be easily removable, non contaminating and controlled specified by the PCD. Changes to a different release paper must demonstrate that no contaminants are transferred from the paper to the product.

12.3 Packaging
The prepreg should have a core that adequately supports the weight of the roll without deformation. The roll should be in a sealed bag that prevents the ingress of moisture and contaminants. Additional packaging, such as a box, may be needed to ensure that the bag is not torn during shipment. Provisions are needed to deal with rolls that are received with damage.

12.4 Shipping
The shipping and storage temperatures will need to be established. If freezer temperatures are needed to maintain product quality, time-temperature recording devices should be used to document the temperature exposure history of the shipment. Materials that have exceeded recommended limits will require a disposition process.
Appendix A - Future Needs

The following are some areas that are recommended for further research and development:

- Improved laminate test methods with lower testing variability to reflect material variability.
- Refined chemical test methods to quantitatively measure resin ingredient concentration in prepreg, e.g. IR and HPLC.
- Improved prepreg rheology test methodology. Current prepreg flow measurements are too subjective and have high variability.
- Development of in-line measurements for resin content and fiber areal weight for use during prepregging.
- Automated visual systems for detection of inline prepreg defects, including gaps, puckers and dry fiber areas.
- Development of accurate sensors to measure key attributes during resin mixing, such as rheology and chemical advancement. These measurements must be done in real time. Current test methods are quick to perform, but are of limited value due to high variability.