DOT/FAA/AR-02/109

Office of Aviation Research
Washington, D.C. 20591

Guidelines and Recommended Criteria for the Development of a Material Specification for Carbon Fiber/Epoxy Unidirectional Prepregs

March 2003

Final Report

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U.S. Department of Transportation
Federal Aviation Administration

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Technical Report Documentation Page

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
DOT/FAA/AR-02/109		

4. Title and Subtitle	5. Report Date
GUIDELINES AND RECOMMENDED CRITERIA FOR THE DEVELOPMENT OF A MATERIAL SPECIFICATION FOR CARBON FIBER/EPOXY UNIDIRECTIONAL PREPREGS	March 2003
	6. Performing Organization Code

7. Author(s)	8. Performing Organization Report No.
William McCarvill, Stephen Ward, Gregg Bogucki, and John Tomblin	

9. Performing Organization Name and Address	10. Work Unit No. (TRAIS)
National Institute for Aviation Research Wichita State University 1845 North Fairmount Wichita, KS 67260-0093	11. Contract or Grant No.

12. Sponsoring Agency Name and Address	13. Type of Report and Period Covered
U.S. Department of Transportation Federal Aviation Administration Office of Aviation Research Washington, DC 20591	Final Report
	14. Sponsoring Agency Code AIR-100

15. Supplementary Notes

The William J. Hughes Technical Monitor was Curtis Davies.

16. Abstract

This report establishes recommendations to guide the development of composite prepreg material specifications. This is intended to advance the work that has been done through previous Federal Aviation Administration and National Aeronautics and Space Administration programs such as the Advanced General Aviation Transport Experiment. These programs have established methodologies for developing design allowable data, control of the data, and sharing the resulting database. 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 general aviation methods to allow a broader market to use the shared database.

This document recommends guidance and criteria for the development of material specifications for carbon fiber/epoxy unidirectional prepreg tape materials to be used on aircraft structures. These recommendations were prepared by a team of industry experts. The guidelines and recommendations are meant to be a documentation of current knowledge and application of sound engineering principles to the development and implementation of composite material procurement specifications. A list of material control areas needing improvement and enhancement is given in appendix A. This document can also be used to develop common industry specifications. This document is limited to recommendations and guidance on the development of the material specification. Additional guidance on the development of process specifications, instructions, and controls for making high-quality laminates can be found in the companion report Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber-Reinforced Polymer Composites, DOT/FAA/AR-02/110.

17. Key Words	18. Distribution Statement
Composite, Specification, Prepreg, Carbon fiber, Epoxy, Test methods, Testing, Qualification, Certification	This document is available to the public through the National Technical Information Service (NTIS), Springfield, Virginia 22161.

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	64	

ACKNOWLEDGEMENTS

This document was assembled with help from many individuals across many areas of expertise. The following Federal Aviation Administration personnel are acknowledged for their individual contributions to this document:

Curtis Davies
Larry Ilcewicz
Evangelia Kostopoulos
Peter Shyprykevich
David Swartz

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LIST OF ACRONYMS

AC	Advisory Circular
ACO	Aircraft Certification Office
AGATE	Advanced General Aviation Transport Experiment
AMS	Aerospace Material Specification
ASTM	American Society for Testing and Materials
CACRC	SAE Commercial Airplane Composite Repair Committee
CFR	Code of Federal Regulations
CTD	Cold Temperature Dry
DAR	Designated Airworthiness Representative
DER	Designated Engineering Representative
DMIR	Designated Manufacturing Inspection Representative
DSC	Differential scanning calorimetry
ETD	Elevated Temperature Dry
ETW	Elevated Temperature Wet
DER	Designated Engineering Representative
DMIR	Designated Manufacturing Inspection Representative
FAA	Federal Aviation Administration
IR	Infrared
HPLC	High-Pressure Liquid Chromatography
KC	Key Characteristics
KPP	Key Process Parameter
MIDO	Manufacturing Inspection District Office
MOL	Material Operational Limit
MRB	Material Review Board
NIST	National Institute of Standards and Technology
OEM	Original Equipment Manufacturer
PCD	Process Control Document
QA	Quality Assurance
RTD	Room Temperature Dry
RTW	Room Temperature Wet
SACMA	Suppliers of Advanced Composite Materials Association
SAE	Society of Automotive Engineers
SPC	Statistical Process Control
TSO	Technical Standard Order

EXECUTIVE SUMMARY

A widely acknowledged validation process used within the composite aircraft industry for the substantiation of composite structure is called the building block approach. This approach is a process using analysis and associated tests of increasing structural complexity. The building block approach is integrated with supporting technologies and design considerations. MIL-HDBK-17F, Volume 3, Chapter 4 contains a complete description of the building block approach. A key element supporting the building block approach is material and process specifications.

The material and process specifications are interwoven throughout the certification validation process. Material specifications are used to define the material's attributes and define the qualification characterization tests. Materials used within the building block tests are purchased in accordance with the material specification. The material specification is used for the procurement of production material. This ensures the delivered materials are of the same quality and performance standards used in the certification and validation process. Process specifications define and control the processes used for the fabrication of materials into structural components. It is widely known that the performance properties of composite laminates are directly affected by the specific process used for their fabrication. It is critical that the test specimens fabricated through the various levels of the building block approach use the same process, which is representative of the one that will be used in the fabrication of production aircraft and rotorcraft.

This report establishes 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 Federal Aviation Administration and National Aeronautics and Space Administration programs such as the Advanced General Aviation Transport Experiment. These programs have established methodology for developing design allowable data, control of the data, and sharing the resulting database. 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 general aviation methods to allow a broader market to utilize the shared database.

This document recommends guidance and criteria for the development of material specifications for carbon fiber/epoxy unidirectional prepreg tape materials to be used on aircraft structures. These recommendations were prepared by a team of industry experts. The guidelines and recommendations are meant to be a documentation of current knowledge and application of sound engineering principles to the development and implementation of composite material procurement specifications. A list of material control areas needing improvement and enhancement is given in appendix A. This document can also be used to develop common industry specifications. This document is limited to recommendations and guidance on the development of material specifications. Additional guidance on the development of process specifications, instructions and controls for making high quality laminates can be found in the companion report Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber-Reinforced Polymer Composites, DOT/FAA/AR-02/110.

1. INTRODUCTION.

1.1 OBJECTIVE.

This document recommends guidance and criteria for the development of material specifications for carbon fiber/epoxy unidirectional prepreg tape materials to be used on aircraft structures. These recommendations were prepared by a team of industry experts who have extensive experience with material specifications, part processing, qualification programs, and design allowables. Starting with section 3 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. This document is limited to recommendations and guidance on the development of a material specification. Additional guidance on the development of process specifications, instructions, and controls for making high-quality laminates can be found in the companion report Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber-Reinforced Polymer Composites, DOT/FAA/AR-02/110.

The purpose of this report is to establish recommendations to guide the development of composite prepreg material specifications. This is intended to advance the work that has been done through previous Federal Aviation Administration (FAA) and National Aeronautics and Space Administration (NASA) programs such as the Advanced General Aviation Transport Experiment (AGATE). These programs have established methodologies for developing design allowable data, control of the data, and sharing the resulting database. 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 general aviation methods to allow a broader market to use the shared database.

The guidelines and recommendations contained in this document should not be viewed as FAA policy or as the only acceptable method for composite material specifications and qualification. They are meant to be a documentation of current knowledge and application of sound engineering principles to the development and implementation of composite material procurement specifications.

This document can also be used to develop an industry approach so 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.

1.2 BACKGROUND.

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. Many more aircraft are currently undergoing the design and development processes that take advantage of composite materials for primary structure applications. With this growth of composite applications, certification issues have emerged with respect to the philosophy of quality control and quality assurance methods needed to guarantee a safe and consistent material supply.

The material properties of a composite are manufactured into the structure as part of the fabrication process (process intensive material). 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, control of the materials is rapidly becoming a vital issue with respect to the overall assurance of safety.

In recent years, the aerospace industry, the National Aeronautics and Space Administration, and the FAA have worked together to develop a cost-effective method of qualifying composite material systems by sharing material qualification databases such as MIL-HDBK-17 and AGATE. By using shared databases, a manufacturer can select an approved composite material system to fabricate parts and validate with a smaller subset of testing for a specific application. For materials to be accepted into these shared databases, the raw materials are required to be manufactured in accordance with a material specification which imposes control of key characteristics (physical, chemical, and mechanical properties) and be processed in accordance with a process specification that controls key characteristics (processing parameters).

1.3 CERTIFICATION PROCESS.

The objective of the composite aircraft structure certification process is to validate that the design meets the applicable configuration requirements. In this context, the design validation process (to establish by proof) is accomplished through verification (to prove by evidence) and qualification (to define attributes or characteristics) of the materials, processes, and analysis tools. Verification is simply to prove by evidence, usually by test data, that the proposed design is acceptable. Material qualification is the verifying of a materials attributes and characterizations, which are typically determined through testing.

A widely acknowledged validation process used within the composite aircraft industry for the substantiation of composite structure is called the building block approach. This approach uses analysis and associated tests of increasing structural complexity. The building block approach is integrated with supporting technologies and design considerations. Refer to MIL-HDBK-17F, Volume 3, Chapter 4 for a complete description of the building block approach. Key elements supporting the building block approach are the material and process specifications.

The material and process specifications are interwoven throughout the certification and validation process. Material specifications are used to define the material's attributes and to define the qualification characterization tests. Materials used within the building block tests are purchased in accordance with a material specification. The material specification is used for procurement of production material. This ensures the delivered materials are of the same quality and performance standards used in the certification validation process. Process specifications define and control the processes used for the conversion of materials into structural parts. It is widely accepted that the performance properties of composite laminates are directly determined by the specific process used for their fabrication. It is critical that the test specimens fabricated through the various levels of the building block approach use the same process, which is representative of the one that will be used in the fabrication of production aircraft and rotorcraft.

Material qualification is a key element of the validation process, which occurs during the coupon level of the building block approach. It is during qualification that the composite material is fully defined and characterized. Qualification tests are planned and conducted to

- establish key material attributes,
- establish material performance properties, and
- verify material characteristics will work in the intended application.

The objective in defining material attributes is to establish the material property limits. Examples of attributes in which limits are set include

- resin content,
- fiber areal weight,
- cured per ply thickness, and
- fiber volume.

These attributes define the material and control its resulting performance properties. Other attributes that are often overlooked are related to the physical structure of the material, which affects processing characteristics. Example attributes of this type include

- fiber-sizing level and type,
- level of impregnation,
- resin impregnation method (hot-melt film or solution),
- width tolerance, and
- backing material selection.

Performance properties are established, or made stable, through statistically significant amounts of testing. It is imperative that the material's natural variability is captured at this time. The objective is not to meet a desired level of performance, but rather, establish the true performance range of the material. Mechanical properties are typically thought of as the only performance properties.

There are other performance-related properties that have a direct bearing on the more familiar mechanical properties, which include tack or handling characteristics, kinetic behavior,

rheological behavior, sensitivity to ambient moisture and temperature (out-time effects), effect of freezer storage, and resistance to fluids and solvents. Multiple material batches (typically three) are tested to establish the material variability. Results obtained from these tests are used to establish minimum and maximum values within the material specification.

1.4 RECOMMENDED SPECIFICATION FORMAT.

For consistency and standardization purposes, a general format for composite prepreg material specifications should be followed. The following is a recommended format that follows the standard format of SAE AMS specifications; other formats with the same content are acceptable to the FAA.

1. Scope
2. Applicable Documents
3. Technical Requirements
   - 3.1 Definitions
   - 3.2 Material Requirements
   - 3.3 General Prepreg Requirements
     - 3.3.1 Resin Requirements
     - 3.3.2 Fiber Requirements
     - 3.3.3 Roll Characteristics
     - 3.3.4 Visual Defects
     - 3.3.5 Storage, Handling, and Out-Time
   - 3.4 Uncured Prepreg Requirements
   - 3.5 Cured Prepreg Requirements
     - 3.5.1 Baseline Cure Process
     - 3.5.2 Cured Laminate Physical Properties
     - 3.5.3 Cured Laminate Mechanical Properties
   - 3.6 Material Characterization
     - 3.6.1 Initial Material Qualification
     - 3.6.2 Equivalency Baseline Database Testing
     - 3.6.3 Additional Characterization Testing for Specific Design Applications
4. Quality Assurance
   - 4.1 Changes To Qualified Materials
     - 4.1.1 Level 0 Changes
     - 4.1.2 Level 1 Changes
     - 4.1.3 Level 2 Changes
     - 4.1.4 Level 3 Changes
     - 4.1.5 Level 4 Changes
   - 4.2 Supplier Site Qualification
   - 4.3 Statistical Process Control
   - 4.4 Product Certification
     - 4.4.1 Supplier Certification Testing
     - 4.4.2 Purchaser Testing
   - 4.5 Test Methods
   - 4.6 Test Panel Fabrication

5. Preparation for Delivery
   - 5.1 Material Identification
   - 5.2 Interleaf
   - 5.3 Packaging
   - 5.4 Shipping
6. Acknowledgement
7. Rejection
8. Notes

2. DEVELOPMENT OF MATERIAL CONTROLS.

Before the initiation of a qualification program, the sensitivities of the material to variations in the tolerances set on the material chemical and physical properties and processing should be investigated. This investigation should explore the characteristics of the material as the various limits are reached. These will determine the suitability of control limits in establishing the required reliability for the normal production phase of a qualified composite material.

This investigation can be performed in a structured design of experiments that will give the relative sensitivities to the process variables with minimum testing. These parametric studies should be performed well before the qualification batches are run to allow time for any required adjustment to settle out in the manufacturing process.

The rest of 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 and qualify the material. The following two sections discuss different types of material procurement specifications: (1) an industry standard specification established by an industry committee and (2) the traditional specification established by an individual end-user.

The authors of this document believe that the full benefits of shared material databases can only be achieved through the use of industry standard material specifications. However, it is recognized that there will continue to be cases where end-user material specifications are appropriate or required. This section discusses the qualification steps for both approaches. It is intended that the remaining sections of the document (starting at section 3) apply to both specification types.

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 material supplier. The material supplier is the manufacturer of the prepreg in this discussion. 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.

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 material specification requirements. A material qualification is performed initially for a new material; it is repeated in part or in whole when changes to materials or manufacturing processes need to be evaluated. 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 may contain target values in place of requirements; in this case, following qualification, the target values are updated to requirements based on the evaluation results.

The initial material database will result from testing conducted to an FAA-approved and FAA-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 (such as SAE AMS Committee P, SAE CACRC, MIL-HDBK-17, and ASTM Committee D-30) for development of an industry specification. The committee would review the data, and finding it satisfactory and needed, would approve a development of a specification. This specification 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. It is envisioned that specifications will be issued for any material for which the minimum dataset, process control, and documentation requirements have been met. End-users desiring multiple material sources for an application can either callout the acceptable materials on the part drawings, on a substitution document, or on an internal specification once they have validated that all of the materials are acceptable for the design(s). However, the materials would be purchased and accepted to the requirements of the industry specification.

It will be the responsibility of the material supplier to continually test and evaluate the material to 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 material and process specifications. After qualification of a material to these specifications, the end-user then purchases the prepreg and manufactures 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, different materials have been qualified to the same set of specification requirements, even though the properties of the materials may be significantly different. This approach can result in less than desirable levels of control over the properties of the individual materials qualified to the specification. This may then translate into less than optimum control over structure made with these materials.

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 in section 2.1.

2.3 END-USER RESPONSIBILITIES FOR MATERIAL USE IN STRUCTURAL DESIGN.

It is the responsibility of the end-user to qualify the material for use in a particular aircraft or rotorcraft application (see section 5.6.1 for additional information). This process by the end-user may involve additional tests to characterize the material and 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 remain valid. The end-user is also responsible for validating that many materials are acceptable for the application, if this is so desired.

If the end-user decides to use the property information in previously developed databases 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.

In order to reduce the risk of failing the equivalency demonstration, it is recommended that the equivalency testing first be done using the material supplier's baseline cure process prior to attempting to demonstrate equivalency to an end-user's modification to the cure cycle. The end-user has the option of skipping the first step and directly demonstrating the equivalency of their cure cycle to the baseline database and cure cycle. However, the two-step process is recommended, since it is further recommended that the end-user perform the purchaser batch acceptance tests on panels cured using the material supplier's baseline cure process.

Further, it is the responsibility of the end-user to validate any deviations from the baseline laminate cure cycle given in the material and process specifications. The end-user's production process must not result in statistically significant changes to design allowables established by using the baseline process. Successful demonstration of equivalency to an existing shared database will allow the end-user to avoid additional material qualification tests and to use the material allowables derived from the shared database. Once an equivalency evaluation is performed by an end-user for one application, it does not have to be repeated by that end-user for follow-on applications that use the same cure process.

This document is limited to recommendations and guidance on the development of a material specification. Additional guidance on the development of process specifications, instructions, and controls for making high-quality laminates can be found in the companion report DOT/FAA/AR-02/110.

2.4 MATERIAL QUALIFICATION PROCESS WHEN USING AN 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. Details

of the industry committee procedures, acceptance limit and allowables calculation procedures, and FAA involvement and procedures for this material qualification process will be defined at a later date.

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 through production trials.

- establish and document the cure process parameters for the material. This cure process will be used to generate the qualification database.

- perform the minimum qualification tests, as defined in the material specification. A minimum of three batches of prepreg material will be produced for the manufacture of test panels.

- upon completion of the testing, develop statistical material batch control limits and B-basis allowables values.

- send the test data and specification limits to the industry committees responsible for the industry material procurement specification, material processing specification, and database approval.

- make the process control document (PCD) available for on-site review by the industry committee and customer personnel who have executed proprietary agreements with the supplier.

- the industry committee will review the data and specification values, and if acceptable, will issue appropriate notifications (documents, web announcements, etc.).

- based on marketing requirements, develop a test plan for additional material characterization tests. Perform the tests, calculate material equivalence limits and allowables, and submit the data and calculated values to the industry committee.

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

At this point, the end-user will

- perform equivalency tests to the material procurement specification, material processing specification, and property database to verify that the user's processes for fabricating test panels and production parts can produce equivalent properties as compared to the industry-approved database developed by the material supplier.

- compare the results of the equivalency tests to the published material database. If all test data meets the requirements then the end-user can use the material allowables developed from the supplier's database in the design and certification of the end-user's structure.

- if equivalency is not demonstrated, the end-user can either (1) modify their fabrication process and rerun the equivalency test program or (2) perform additional tests to develop design allowables specific to their situation.

- 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 will approve the design, which specifies the materials, for a Type Certificate.

2.5 MATERIAL QUALIFICATION PROCESS WHEN USING AN 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. It also assumes that all qualification and design-related testing will be performed or controlled by the end-user. This material qualification process is consistent with the current FAA procedures for an aircraft certification program.

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

- develop a new material for potential market requirements.

- stabilize the prepreg production process through production trials.

- establish and document the 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 potential end-users.

At this point the end-user will

- submit a qualification and design allowables test plan and draft material specification to the FAA.

- perform the qualification and allowables tests, using the end-user's planned production cure process. A minimum of three batches of prepreg material will be used for the manufacture of test panels. Panel fabrication and testing will be witnessed as required.

- upon completion of the testing, calculate proposed material batch acceptance limits and B-basis allowables values. Specification limits and allowables will be calculated using procedures documented in DOT/FAA/AR-00/47 and MIL-HDBK-17.

- submit the test data, material specification, and cure process documentation to the FAA. The FAA 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.

- 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 will approve the materials and design for a Type Certificate.

3. THE SCOPE SECTION OF THE MATERIAL SPECIFICATION.

This section should include a general description of the product and its area of application to guide the prospective user. General temperature use limits and cure conditions should be stated. If the product is to be supplied with 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 for end-user material specifications:

- Form—defines the basic material form; tape, slit tape, towpreg

- Type—defines the resin content of the prepreg

- Grade—defines the areal weight of the fiber in the prepreg

- Class—defines the specific fiber used in the prepreg (fiber type, tow count, size type and content, surface treatment level, manufacturer, facility)

For industry standard specifications, they would specify the form, resin content, areal weight, and specific fiber information for each specific prepreg material covered.

4. THE APPLICABLE DOCUMENTS SECTION OF THE MATERIAL SPECIFICATION.

This section 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 that was developed or approved by industry organizations. Test methods can come from ASTM and SACMA (CFA). 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 kept to a minimum. When used, they should be referenced and included in the PCD.

Examples include:

ASTM C 297-94(1999)	Standard Test Method for Flatwise Tensile Strength of Sandwich Constructions
ASTM C 364-99	Standard Test Method for Edgewise Compressive Strength of Sandwich Constructions
ASTM C 393-00	Standard Test Method for Flexural Properties of Sandwich Constructions
ASTM C 613/C 613M-97	Standard Test Method for Constituent Content of Composite Prepreg by Soxhlet Extraction
ASTM D 792	Specific Gravity (Relative Density) and Density of Plastics by Displacement
ASTM D 2344	Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short-Beam Method
ASTM D 2471-99	Standard Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins
ASTM D 2734	Void Content of Reinforced Plastics
ASTM D 3039	Tensile Properties of Polymeric Matrix Composite Materials
ASTM D 3171-99	Standard Test Method for Constituent Content of Composite Materials
ASTM D 3529/D 3529M-97	Standard Test Method for Matrix Solids Content and Matrix Content of Composite Prepreg
ASTM D 3530/D 3530M-97	Standard Test Method for Volatiles Content of Composite Material Prepreg
ASTM D 3531-99	Standard Test Method for Resin Flow of Carbon Fiber-Epoxy Prepreg
ASTM D 3532-99	Standard Test Method for Gel Time of Carbon Fiber-Epoxy Prepreg
ASTM D 3544-76 (1996)	Standard Guide for Reporting Test Methods and Results on High Modulus Fibers
ASTM D 3800-99	Standard Test Method for Density of High-Modulus Fibers
ASTM D 3878-01	Standard Terminology Composite Materials

ASTM D 4018-99	Standard Test Methods for Properties of Continuous Filament Carbon and Graphite Fiber Tows
ASTM D 4065-95	Standard Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics
ASTM D 4102-82 (1999)	Standard Test Method for Thermal Oxidative Resistance of Carbon Fibers
ASTM D 3479-96	Standard Test Method for Tension-Tension Fatigue of Polymer Matrix Composite Materials
ASTM D 3518-94 (2001)	Standard Test Method for In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a $\pm 45^{o}$ Laminate
ASTM D 5229-92 (1998)e1	Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials
ASTM D 5279-99	Standard Test Method for Measuring the Dynamic Mechanical Properties of Plastics in Torsion
ASTM D 5379	Shear Properties of Composite Materials by V-Notched Beam Method
ASTM D 5418	Standard Test Method for Transition Temperatures of Polymers by Differential Scanning Calorimetry
ASTM D 5467-97	Standard Test Method for Compressive Properties of Unidirectional Polymer Matrix Composites Using a Sandwich Beam
ASTM D 5687-95	Standard Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation
ASTM D 5766-95	Standard Test Method for Open Hole Tensile Strength of Polymer Matrix Composite Laminates
ASTM D 5961-01	Standard Test Method for Bearing Response of Polymer Matrix Composite Laminates
ASTM D 6264-98	Standard Test Method for Measuring the Damage Resistance of a Fiber-Reinforced Polymer-Matrix Composite to a Concentrated Quasi-Static Indentation Force
ASTM D 6484-99e1	Standard Test Method for Open-Hole Compressive Strength of Polymer Matrix Composite Laminates

ASTM D 6641-01e1	Standard Test Method for Determining the Compressive Properties of Polymer Matrix Composite Laminates Using a Combined Loading Compression (CLC) Test Fixture
ASTM D 6742-01	Standard Practice for Filled-Hole Tension and Compression Testing of Polymer Matrix Composite Laminates
ASTM D 5528-01	Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites
ASTM D 6115-97	Standard Test Method for Mode I Fatigue Delamination Growth Onset of Unidirectional Fiber-Reinforced Polymer Matrix Composites
ASTM D 6415-99e1	Standard Test Method for Measuring the Curved Beam Strength of a Fiber-Reinforced Polymer-Matrix Composite
ASTM D 6671-01	Standard Test Method for Mixed Mode I-Mode II Interlaminar Fracture Toughness of Unidirectional Fiber Reinforced Polymer Matrix
ASTM D 6507-00	Standard Practice for Fiber Reinforcement Orientation Codes for Composite Materials
ASTM E 168	General Techniques of Infrared Quantitative Analysis
ASTM E 1252-98	Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis
ASTM E 1309-00	Standard Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases
ASTM E 1356-98	Standard Test Method for Assignment of the Glass Transition Temperature by Differential Scanning Calorimetry of Differential Thermal Analysis
ASTM E 1434-00	Standard Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases
ASTM E 1471-92 (1998)	Standard Guide for Identification of Fibers, Fillers, and Core Materials in Computerized Material Property Databases
ASTM E 1640-99	Standard Test Method for Assignment of the Glass Transition Temperature by Dynamic Mechanical Analysis
ASTM E 2041-99	Method of Estimating Kinetic Parameters by Differential Scanning Calorimetry Using Borchardt and Daniels Method

ASTM E 2070-00	Test Method for Kinetic Parameters by Differential Scanning Calorimetry Using Isothermal Methods
ASTM E 4473-95	Standard Practice for Measuring the Cure Behavior of Thermosetting Resins Using Dynamic Mechanical Procedures
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. THE TECHNICAL REQUIREMENTS SECTION OF THE MATERIAL SPECIFICATION.

5.1 THE DEFINITIONS SECTION.

This section should include definitions for terms or abbreviations that are used. The definitions provide clarity between the supplier and the procurer. Material properties, quality, and defects must be defined such that batches made after the original qualification have the same level of quality and properties. For example, uni-tape defects such as slitter litter, fuzz balls, creases, foreign material, dry spots, gaps, fiber alignment, splices, and edge deviation from a straight line should have a specific definition. Since the ability to 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, SAE, and ASTM should be used.

5.1.1 Batch Definitions.

The following batch definitions are recommended:

Batch (or Lot) (general) – n, A quantity of material produced essentially at the same time and under the same conditions from a well-defined collection of raw materials. The quantity of material must have minimal variation in properties throughout to be considered a unique batch.

Batch (or Lot) (fibers) – n, For fibers, a quantity of material formed during one essentially continuous, uninterrupted production run under the same process conditions using one to three precursor lots. An interruption in the process of up to 72 hours is permitted, provided that the production equipment settings are not modified or another material was not produced on the equipment during the interruption.

Batch (or Lot) (fabric) – n, For fabrics, a quantity of material woven from one to three batches (lots) of fiber.

Batch (or Lot) (resin) – n, For a batch of resin, the definition varies depending on the specific mixing process:

- In a batch mixing process a large vessel is charged with the desired types and quantities of raw ingredients. After mixing is complete, the vessel is discharged. The material made from this single-mix process is defined as a single batch of resin.

- A continuous mixing process for producing resin typically involves incrementally feeding raw ingredients into a mixing device that blends them into a stream of resin. A batch of resin made by this process is defined as a quantity of material formed during one essentially continuous uninterrupted production run under the same process conditions using the same raw ingredients. Since start up and shutdown usually require purging the equipment, a shutdown will signal the end of a specific batch. Material made after start up is defined as a new batch. If a process shutdown does not require purging, an interruption in the process of up to 72 hours is permitted, provided that the production equipment settings are not modified or another material was not produced on the equipment during the interruption.

- In one version of a semi-continuous mixing process, a large vessel is charged with a portion of the raw ingredients (premix). After mixing, the vessel is discharged into several smaller containers, each of which acts as the vessel for subsequent mixing steps. The remaining raw ingredients are added to these smaller vessels and further mixing results in the final resin composition. The premix produced in the large vessel can be considered as a single batch of raw ingredient. The material produced during the final mixing in the small containers can be considered one batch if it is produced from the same raw ingredients batches without an interruption of more than 72 hours without the production of another material in the interval, and until the premix is consumed.

- In another version of a semi-continuous mixing process, small complete mixes of raw ingredients are made without the premix step. A batch of resin then consists of any number of these small mixes if they are made from the same lots of raw ingredients, the production run is not interrupted for more than 72 hours, and there is no other material made in the interval.

- For all mixing processes, blending of raw ingredient lots is permissible if the same blend ratio is found throughout all portions of the resin batch. Traceability must be retained on the ingredient lots that were used. For all mixing processes, a single-resin batch may contain a maximum of three blended lots of each raw chemical ingredient.

- For a resin filming operation, the film batch will correspond to the batch designation of the resin mix used to make the film. An interruption in the filming process of up to 72 hours is permitted, provided that the production equipment settings are not modified or another film was not produced on the equipment during the interruption. Otherwise the

resin films produced on either side of the interruption shall be considered to be separate resin batches.

Batch (or Lot) (specific, prepreg) – n, For prepregs, material made from one to three batches (lots) of fiber and one batch of resin. Prepregging must not be interrupted for more than 72 hours with no other prepregging on the same equipment during the time interval to retain a singular batch designation.

Batch (or Lot) (specific, lamina and laminates) – n, For laminae and laminates, material made from:

- One batch of prepreg
- One batch (lot) of fiber and one batch of resin(s)
- One batch of fabric and one batch of resin(s)

5.1.2 Batch Definitions Discussion.

The above definitions are generally applicable for use with material acceptance processes, including sampling plans for acceptance testing. For material qualification and allowables test programs, stricter definitions of a batch are often specified in order to control the amount of material variability to be evaluated in the test program. For instance, a particular batch of prepreg may be restricted to a single lot of fiber and a single mix of resin.

5.2 THE MATERIAL REQUIREMENTS SECTION.

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

A specific prepreg specification designation should include only a single-resin formulation and a single-specific fiber. A specific designation is defined to be a unique form, type, grade, and class. The specification document can include multiple forms, types, grades, and classes (fiber types).

5.3 THE GENERAL PREPREG REQUIREMENTS SECTION.

This section should include requirements that define the specific raw ingredients and processes for producing the prepreg (many of these requirements may be specified in the PCD, which is referenced by the specification). In the following sections, 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.

5.3.1 The Resin Requirements Section.

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

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 should be defined prior to qualification. Proposed limits of ingredient-weighing accuracy and process times and temperatures should 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 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 should be validated through chemical analysis. Blending of mixed resins is not preferred unless it can be demonstrated that there is no impact on prepreg out-time 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 or specifications. The prepreg material supplier should establish material specifications for all raw materials to be used in the prepreg resin. 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. Testing 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 some cases this information may be considered proprietary and controlled in the PCD. These may include gel time, viscosity, degree of advancement, and analytical signature such as Infrared Spectrophotometry (IR) or High-Pressure Liquid Chromatography (HPLC). In addition, the resin cure kinetics and rheology should be well characterized. It is 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 epoxy resin 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 degree of plasticization which leads to lowering of elastic modulus and glass transition temperature, 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, be made available to potential users of the material, and be made available to the industry committee responsible for the industry specification for the material, if applicable. Table 1 summarizes the resin property data discussed above. None of the tests in table 1 are recommended for batch acceptance testing.

TABLE 1. RECOMMENDED SET OF NEAT RESIN PROPERTIES

Resin Property	Test Method
Density	ASTM D792
Viscosity	Any agreed method
Gel Time	ASTM D2471
IR	ASTM E1252
HPLC (ingredient ratios)	Any agreed method
Cure Kinetics	ASTM E2041, ASTM E2070
Rheology	ASTM E4473

5.3.2 The Fiber Requirements Section.

This section should include requirements that define the mechanical and physical properties of the fiber. The carbon fiber to be used in the prepreg should be purchased to a separate fiber specification that uniquely defines the fiber type, manufacturer, and facility.

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 correspond to a unique prepreg designation (e.g., class) and, in the case of an industry specification, must correspond to a unique designation (note: two or more facilities owned by the same company producing identical fiber can be considered a single item when it has been established by testing). It is not acceptable for a prepreg specification to refer to the carbon fiber by a trade name without specifying the manufacturer, facility that produces the fiber, and the fiber specification that controls it.

The fiber specification must define the average values and ranges for all critical fiber mechanical and physical properties including tensile strength, tensile modulus, and density. The prepreg specification must identify the fiber form, tow count (e.g., 12K flat tow), and twist or no twist. The fiber-sampling plan and test methods for fiber properties and quality must be documented.

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, or content will require equivalency testing or the establishment of a new material designation (see section 2).

The recommended definition of fiber batch is given in section 5.1. Fiber batch blending of up to three lots is allowed as long as there is traceability of each fiber batch, and the lots are randomly distributed across the prepreg. For prepreg materials used to establish the initial material database, each prepreg batch should contain a single and unique fiber batch.

Table 2 summarizes the fiber property data discussed above. These tests should be performed by the fiber supplier on each lot of fiber supplied to the prepreg manufacturer. None of the tests in

table 2 are recommended for prepreg batch acceptance testing by the prepreg supplier or material end-user.

TABLE 2. RECOMMENDED SET OF CARBON FIBER PROPERTIES

Fiber Property	Test Condition	Test Method
Form	N/A	ASTM E1309
Twist	N/A	Any agreed method
Size Content	Ambient	ASTM D4018
Tensile Modulus	RTD	ASTM D4018
Tensile Strength	RTD	ASTM D4018
Density	RTD	ASTM D3800

5.3.3 The Prepregging Process Requirements Section.

The key process parameters for filming and prepregging must be established and documented. In some cases this information may be considered proprietary and controlled in the PCD. Process conditions should have maximum and minimum limits that are monitored, recorded, and reviewed per the SPC procedures.

The recommended definitions of resin and prepreg batches are given in section 5.1. The resin filming process limits should not only control the chemical advancement of the resin but film quality aspects (e.g., fish eyes, mottling, film thickness (or weight) down the length, and film thickness (or weight) across the width). Limits should be defined for film thickness (or weight), including a target thickness (or weight) value and range.

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

The prepreg process limits should 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, drape, thickness, 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 on flat panel laminates. Resin should be subjected to times and temperatures that mimic the minimum and maximum thermal histories that the prepreg is subjected to during its manufacture. These resins should then be subjected to the cure extremes claimed by the manufacturer in terms of heat-up rates, hold steps, and highest and lowest cure temperature. The rheological response of the resin, when subjected to these extremes, should be understood. The degree of cure achieved by the resin and the mechanical properties of the resin, both dry and after fluid and water exposure, can be evaluated via Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA) tests. The tests to evaluate the extremes of the processing window will not require a complete database but sufficient data to demonstrate that the material properties and allowables developed using the nominal cure process are valid (equivalent) for the entire range of process parameters.

5.4 THE UNCURED PREPREG REQUIREMENTS SECTION.

5.4.1 The Uncured Prepreg Properties Section.

This section should include requirements for uncured prepreg physical and chemical properties. Table 3 outlines the minimum recommended 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. Chemical reactivity via DSC can be accomplished by evaluating the onset of exotherm. Total heat liberated cannot be measured accurately due to variability of resin content on the small-scale sample. The degree of advancement can be detected by measurement of the subambient Tg of the uncured resin.

TABLE 3. RECOMMENDED SET OF UNCURED PREPREG PROPERTIES—PREPREG TAPE MATERIALS

Uncured Prepreg Property	Test Condition	Test Method
Fiber Content, areal weight*		SACMA SRM 23
Resin Content, % by weight*		ASTM D 3529 SACMA SRM 23
Insoluble Content		ASTM D 3529
Volatile Content, % by weight*		ASTM D 3530
Flow, % by weight *		ASTM D 3531 SACMA SRM 22
Gel Time, minutes		ASTM D 3532
HPLC (ingredient ratios)*		SACMA SRM 20
IR (Ingredients Chemical Signature)	RTD	ASTM E 1252
Chemical Reactivity and Degree of Advancement via DSC		ASTM E 1356 ASTM D 3418 SACMA SRM 25
Tack	RTD	Any agreed method
Drape	RTD	Any agreed method

*Batch acceptance tests (see section 6.5)

Testing should be conducted on the start and end of rolls and should also be conducted across the full width (at least on the edges and center) of the rolls during production trials and qualification. This is intended to capture down-the-length and across-the-width variability introduced in the filming and prepregging process steps.

5.4.2 Roll Characteristics.

The roll size, weight, width, core type, width and length tolerances, splice allowances, and defect/splice tracer requirements should be defined. They can be specified in the material specification or on a separate purchasing document. A separate purchasing document is expected to be used by an end-user when purchasing material to an industry standard specification.

5.4.3 The Visual Defect Limitations and Dimensions Section.

This section 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 some cases, this information may be considered proprietary and controlled in the PCD. Allowable defect limits can be based on generally accepted industry standards.

Procedures for closing gaps, e.g., through the use of rollers, can be used if documented in the PCD. Edge alignment can be corrected by respooling if procedures are established to control and document material out-time.

Criteria for discontinuous defects, e.g., puckers, fuzz balls, splices, foreign material, incomplete wet out, yarn twists and crossovers, broken yarns, fiber distortion, crushed yarns, and bowed fibers, 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 accepted in the specification.

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 end-user on the bottom of the prepreg when the part is being laid up, the same criteria for allowable defect limits and correction should 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 should be recorded for each roll and attached to the roll. Defects can be removed by splicing per documented procedures and by criteria for maximum number and minimum spacing of splices. The splicing technique must be easily identified by the end-user to avoid incorporation of the splice into a part. The time out of cold storage during defect removal must be recorded and used to adjust the remaining out-time.

5.4.4 The Storage, Handling, and Out-Time Section.

This section should include definitions and limitations for storage life under specified conditions, handling life under ambient conditions, and out-time capability of laid up material. These requirements should be based on specific test data and experience with similar materials. The material supplier can establish and document the storage life as a function of storage temperature. A portion of the material batches produced for the initial material database development should be placed in an appropriate storage facility. After the desired maximum shelf life is reached, the material should be tested and the results compared to the specification requirements. This testing could be performed in conjunction with the equivalency baseline database tests discussed in section 5.6.2. Recommended definitions for storage, handling, staging life, and out-time are shown in figure 1.

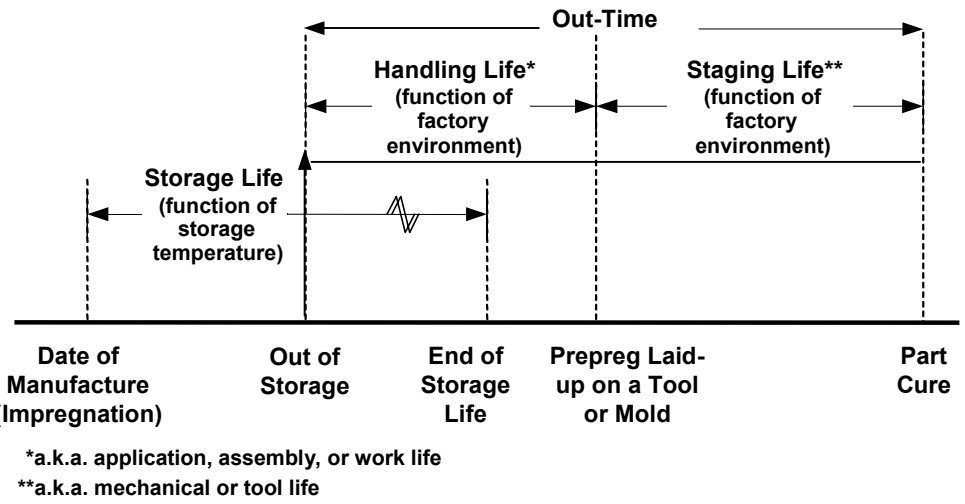

FIGURE 1. RECOMMENDED DEFINITIONS OF STORAGE, HANDLING, STAGING LIFE, AND OUT-TIME

It is recommended that a tracking policy be implemented by the material supplier to document storage/out-time of material from date of manufacture (defined as the date of impregnation) to arrival on dock at the end-user. 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, must 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 end-user. 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-time accumulated during warming, respooling, and repackaging must be subtracted from the total life specified by the material supplier and documented for the end-user.

5.5 THE CURED PREPREG PROPERTY REQUIREMENTS SECTION.

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

5.5.1 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 (for qualification and design allowables) and for batch acceptance testing. The baseline cure cycle should be selected based on the expected end-user applications and requirements for the material (see DOT/FAA/AR-02/110 for recommendations on selecting the cure cycle). Reasonable tolerances on heat-up rates and time-at-temperature should 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 batch acceptance testing by the material supplier and end-user. It is possible that part-manufacturing processes will use a different cure cycle than the baseline cure cycle. The end-user must demonstrate that the part cure cycle produces equivalent properties to the material database in order to use the allowables generated by the database for certification of the part (see DOT/FAA/AR-00/47). If the end-user defines an alternate cure cycle, they should define additional acceptance testing with that cure cycle.

5.5.2 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 from the sides and center. This can be done on a periodic sampling basis.

TABLE 4. RECOMMENDED SET OF CURED LAMINATE PHYSICAL PROPERTIES—PREPREG TAPE MATERIALS

Cured Laminate Physical Property	Test Condition	Test Method
Cured Ply Thickness[1]		Any agreed method
Fiber Volume, % by volume [2]		ASTM D 3171
Resin Content, % by volume [2]		Any agreed method
Void Content, % by volume [2]		ASTM D 2734
Laminate Density	RTD	ASTM D 792
Glass Transition Temperature, Tg [2]	• Dry • Wet (85% RH)	SACMA SRM 18
Equilibrium Moisture Content	85% Relative Humidity	ASTM D 5229
Moisture Absorption	85% Relative Humidity	ASTM D 5229
Thermal Induced Microcracking	Cycles over expected range of usage temperatures; fast heat-up spikes, etc.	Any agreed method

1 - Batch acceptance test (see section 6.5)
2 - Equivalency baseline database test (see section 5.6.2)

5.5.3 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 should be able to detect changes in the fiber, resin, prepregging process, and the response of the prepreg to variations in the 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 on an ongoing basis to further populate the database, especially for use as a baseline in future equivalency evaluations (see section 5.6.2). The results from these tests would be used to monitor 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 represents the minimum tests required to establish a material specification. This test matrix is 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 the 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.

It should be understood that, while material specification acceptance values are not the same as B-basis design allowables, they can be derived from the same test data. The calculation methods are different and they are intended for different objectives. Material specification acceptance values are specifically intended for control of material. The values are calculated using statistical procedures that are a function of a selected value for the probability of rejection of a good batch of material. The acceptance values are a function of the database mean and standard deviation, and the batch sample size. Therefore, as data is added to the database, acceptance values will only change if there is a change in the data mean or variation.

Basis values (allowables) are intended to provide a certain level of statistical confidence for design strength calculations. B-basis values are established such that 90% of the population data falls above the basis value, with 95% confidence. Basis values are a function of database mean and standard deviation, and the number of data points in the database.

5.5.4 Recommended Laminate Tests.

To establish a material specification, the tests in table 5a are recommended as a minimum set for material characterization and qualification 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.

TABLE 5a. RECOMMENDED MINIMUM SET OF CURED LAMINATE MECHANICAL PROPERTIES—PREPREG TAPE MATERIALS

Layup [3]	Test Type and Direction	Property	No. of Batches x No. of Panels x No. of Tests/Batch/Panels			
			Test Temperature/Moisture Condition			
			Lowest Temperature/ Ambient	70°F/ Ambient	Highest Temperature/ Ambient	Highest Temperature/ Wet
[0]n	0 Tension ASTM D 3039	Modulus		3 x 2 x 3 [2]	1 x 2 x 3	
[0]n [3]	0 Compression ASTM D 6641	Modulus		3 x 2 x 3 [2]	1 x 2 x 3	1 x 2 x 3
[90]n [3]	90 Tension ASTM D 3039	Ultimate Strength and Modulus		3 x 2 x 3	1 x 2 x 3	3 x 2 x 3
[90]n [3]	90 Compression SACMA SRM 1	Ultimate Strength and Modulus		3 x 2 x 3	1 x 2 x 3	3 x 2 x 3
[0/90/0/90/0/90/0/90/0]	0/90 Tension ASTM D 3039	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 [1]	3 x 2 x 3	3 x 2 x 3
[90/0]ns [3]	90/0 Compression ASTM D 6641	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 [2]	3 x 2 x 3 [1]	3 x 2 x 3
[+45/-45]ns [4]	In-plane Shear ASTM D 3518	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 [2]	3 x 2 x 3	3 x 2 x 3
[0]n [5]	Short Beam Shear ASTM D 2344	Ultimate Strength		3 x 2 x 3 [1]		

1 - Batch acceptance tests (see section 6.5)
2 - Equivalency baseline database tests (see section 5.6.2)
3 - Select a value of n such that the laminate thickness is approximately 0.100 inch.
4 - Select a value of n such that the laminate thickness is approximately 0.050 inch.
5 - Select a value of n such that the laminate thickness is approximately 0.200 inch.

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 MIL-HDBK-17, Volume 1, Chapters 3 through 7. Moisture conditioning should be conducted per the procedures given in MIL-HDBK-17 and ASTM D 5229. Cross-ply [0/90] tension and [90/0] compression tests are recommended over the traditional unidirectional [0] tests because the cross-ply layups have been demonstrated to be less sensitive to test anomalies. 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 all industry material

specifications to be developed in the near future will require the use of the cross-ply test configurations and CLC test method.

TABLE 5b. RECOMMENDED ADDITIONAL CURED LAMINATE MECHANICAL PROPERTIES FOR GENERAL APPLICATIONS—PREPREG TAPE MATERIALS

Layup[3]	Test Type and Direction	Property	No. of Batches x No. of Panels x No. of Tests/Batch/Panels			
			Test Temperature/Moisture Condition			
			Lowest Temperature/ Ambient	70°F/ Ambient	Highest Temperature/ Ambient	Highest Temperature/ Wet
[45/0/-45/90]ns	Open Hole Tension [2] ASTM D 5766	Ultimate Strength	3 x 2 x 3	3 x 2 x 3 [1]	3 x 2 x 3 [1]	3 x 2 x 3
[45/0/-45/90]ns	Open Hole Compression [2] ASTM D 6484	Ultimate Strength		3 x 2 x 3 [1]	3 x 2 x 3 [1]	3 x 2 x 3

1 - Equivalency baseline database tests (see section 5.6.2)
2 - Open-hole test configuration: 0.25-inch hole diameter, 1.5-inch width
3 - Layups should be selected such that laminate thickness is between 0.100 to 0.150 inch.

Batch acceptance tests are recommended in table 5a (as noted). 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.

The tests listed in table 6a are some optional tests for an expanded material database. These tests are intended to 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.

TABLE 6a. OPTIONAL CURED LAMINATE MECHANICAL PROPERTIES FOR EXPANDED DATABASE—PREPREG TAPE MATERIALS

Layup [1]	Test Type and Direction	Property	Test Temperature/Moisture Condition			
			Lowest Temperature/ Ambient	70°F/ Ambient	Highest Temperature/ Ambient	Highest Temperature/ Wet
[45/0/-45/90]ns	Unnotched Tension ASTM D3039	Ultimate Strength	✓	✓		✓
[45/-45/90/45/-45/45/-45/0/45/-45]$_S$	Unnotched Tension ASTM D3039	Ultimate Strength	✓	✓		✓
[45/90/-45/0/0/45/0/0/-45/0]ns	Unnotched Tension ASTM D3039	Ultimate Strength	✓	✓		✓
[45/0/-45/90]ns	Unnotched Compression	Ultimate Strength		✓	✓	✓
[45/90/-45/0/0/45/0/0/-45/0]ns	Unnotched Compression	Ultimate Strength		✓		✓
[45/-45/90/45/-45/45/-45/0/45/-45]$_S$	Unnotched Compression	Ultimate Strength		✓		✓
[45/90/-45/0/0/45/0/0/-45/0]ns [2]	Open Hole Tension ASTM D5766	Ultimate Strength	✓	✓		✓
[45/-45/90/45/-45/45/-45/0/45/-45]$_S$ [2]	Open Hole Tension ASTM D5766	Ultimate Strength	✓	✓		✓
[45/90/-45/0/0/45/0/0/-45/0]ns [2]	Open Hole Compression ASTM D6484	Ultimate Strength		✓		✓
[45/-45/90/45/-45/45/-45/0/45/-45]$_S$ [2]	Open hole Compression ASTM D6484	Ultimate Strength		✓		✓
[45/0/-45/90]ns [3]	Filled Hole Tension ASTM 6742	Ultimate Strength	✓	✓		
[45/90/-45/0/0/45/0/0/-45/0]ns [3]	Filled Hole Tension ASTM 6742	Ultimate Strength	✓	✓		
[45/-45/90/45/-45/45/-45/0/45/-45]$_S$ [3]	Filled Hole Tension ASTM 6742	Ultimate Strength	✓	✓		
[45/0/-45/90]ns [3]	Filled Hole Compression ASTM 6742	Ultimate Strength		✓		✓
[45/90/-45/0/0/45/0/0/-45/0]ns [3]	Filled Hole Compression ASTM 6742	Ultimate Strength		✓		✓
[45/-45/90/45/-45/45/-45/0/45/-45]$_S$ [3]	Filled Hole Compression ASTM 6742	Ultimate Strength		✓		✓

TABLE 6a. OPTIONAL CURED LAMINATE MECHANICAL PROPERTIES FOR EXPANDED DATABASE—PREPREG TAPE MATERIALS (Continued)

Layup [1]	Test Type and Direction	Property	Test Temperature/Moisture Condition			
			Lowest Temperature/ Ambient	70°F/ Ambient	Highest Temperature/ Ambient	Highest Temperature/ Wet
[45/0/-45/90]ns [4]	Single Shear Bearing ASTM 5961	Ultimate Strength		✓		✓
[45/90/-45/0/0/45/0/0/-45/0]ns [4]	Single Shear Bearing ASTM 5961	Ultimate Strength		✓		✓
$[45/-45/90/45/-45/45/-45/0/45/-45]_s$ [4]	Single Shear Bearing ASTM 5961	Ultimate Strength		✓		✓
[0]n	Compression Interlaminar Shear	Ultimate Strength	✓	✓		✓

1 - Layups should be selected such that laminate thickness is between 0.100 to 0.150 inch.
2 - Open-hole test configuration: 0.25-inch hole diameter, 1.5-inch width
3 - Filled hole test configuration: 0.25-inch hole diameter, 100° tension head countersunk fastener, 1.5-inch width
4 - Single shear bearing configuration: 0.25-inch hole diameter, 1.5-inch width, one protruding head fastener, and stabilization fixture

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

The 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:

- Methyl Ethyl Ketone Washing Fluid. ASTM D740
- Polypropylene Glycol Deicer (Type I) MIL-A-8243
- Isopropyl Alcohol Deicing Agent (TT-I-735)

TABLE 6b. OPTIONAL CURED SANDWICH PANEL MECHANICAL PROPERTIES FOR EXPANDED DATABASE—PREPREG TAPE MATERIALS

Layup [1]	Test Type and Direction	Property	Test Temperature/Moisture Condition			
			Lowest Temperature/ Ambient	70°F/ Ambient	Highest Temperature/ Ambient	Highest Temperature/ Wet
[0/90/90/0/core/0/90/90/0]	Sandwich Long Beam Flexure	Ultimate Strength		✓		✓
[0/45/90/-45/core/-45/90/45/0]	Sandwich Long Beam Flexure	Ultimate Strength		✓		✓
[0/45/90/-45/core/-45/90/45/0]	Sandwich Long Beam Flexure, with Open Hole	Ultimate Strength		✓		✓
[0/45/90/-45/core/-45/90/45/0]	Sandwich Long Beam Flexure, with 30 in-lb impact	Ultimate Strength		✓		
[0/45/90/-45/core/-45/90/45/0]	Sandwich Long Beam Flexure, with 120 in-lb impact	Ultimate Strength		✓		

1 - If the material is designed to be self-adhesive to the core, then these tests should be conducted on cocured panels fabricated without adhesive. If the material requires an adhesive layer for bonding to the core, then the tests can be conducted on either (or both) cocured panels or precured skins secondarily bonded to the core, depending on the anticipated design and fabrication methods to be used with the material.

It is recommended that the test laminates be exposed to the above fluids at room temperature conditions, unless the material is expected to be used in an application where fluid exposure occurs for significant time periods at a different temperature. For example, materials intended for use in integral fuel tanks should be exposed to fuel over the expected range of service temperatures for the fuel tank (typically cold to hot conditions). Tests for extended contact fluids should be conditioned by immersion for 500 ±50 hours; tests for short duration contact fluids should be conditioned by immersion for 48 ±4 hours.

The test method to evaluate solvent affect must 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 should be conducted at the temperatures expected during service.

It is recommended that fatigue testing of open-hole specimens be conducted to confirm that the parts will be durable over the expected service life. Postimpact residual strength evaluation for damage tolerance is recommended for primary structure applications. Fatigue testing of impact-damaged specimens may also be required for certification of certain primary structures; however, the detailed recommendations for these tests are beyond the scope of this document. See FAA AC 21-107A for further guidance on these issues.

TABLE 7. RECOMMENDED TESTS FOR DURABILITY AND SERVICE LIFE CONFIRMATION—PREPREG TAPE MATERIALS

Layup	Test Type and Direction	Property	Test Temperature/Moisture Condition			
			Lowest Temperature/ Ambient	70°F/ Ambient	Highest Temperature/ Ambient	Highest Temperature/ Wet
[+45/-45]ns [1] Exposure to Solvent A (repeat for each potential exposure fluid)	In-plane Shear ASTM D3518	Ultimate Strength and Modulus		✓		✓
[0]n [1]	Mode I Fracture Toughness ASTM D6115	G1c	✓	✓		✓
[0]n [1]	Mode II Fracture Toughness	G2c	✓	✓		✓
[45/0/-45/90]ns [3]	Open Hole Fatigue, R=-1 (Tension/ Compr.)	Fatigue Life [4]		✓		✓
[45/90/-45/0/0/45/0/0/-45/0]ns [3]	Open Hole Fatigue, R=-1 (Tension/ Compr.)	Fatigue Life [4]		✓		✓
[45/-45/90/45/-45/45/-45/0/45/-45]$_S$ [3]	Open Hole Fatigue, R=-1 (Tension/ Compr.)	Fatigue Life [4]		✓		✓
[45/0/-45/90]ns [2]	Compression After Impact, 270 in-lbs impact	Ultimate Strength		✓		
[45/0/-45/90]ns [2]	Compression After Impact, 540 in-lbs impact	Ultimate Strength		✓		
[45/0/-45/90]ns [2]	Compression After Impact, 1080 in-lbs impact	Ultimate Strength		✓		

1 - Layups should be selected such that laminate thickness is approximately 0.100 inch.
2 - Layups should be selected such that laminate thickness is between 0.140 to 0.200 inch.
3 - Layups should be selected such that laminate thickness is between 0.100 to 0.150 inch. Open-hole test configuration: 0.25-inch hole diameter, 1.5-inch width
4 - Runout for fatigue life tests should be at least $1x10^6$ cycles, unless the material is intended for use in severe fatigue environments, in which case the number of cycles for runout should be increased.

It is also recommended that any other unique environmental conditions, such as ultraviolet (UV) exposure and other weathering effects that may degrade material properties, be considered. These environmental conditions should be applied for the expected service conditions and life for the material and structural application.

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 FAA.

All of the tests shown in tables 6 and 7 may not be applicable depending on the physical limitations of the material (for instance, impact energy levels may have to be adjusted for different laminate thicknesses and material characteristics).

5.6 THE MATERIAL CHARACTERIZATION SECTION OF THE MATERIAL SPECIFICATION.

5.6.1 The Initial Material Qualification.

This section should include procedures and requirements for initially characterizing the material to establish the specification requirements.

5.6.1.1 Industry Material Specification.

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 will 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. Appropriate reviews and inspections should be agreed upon to ensure FAA acceptance of the qualification.

5.6.1.2 The 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.

5.6.1.3 Minimum Level of Testing.

It is recommended that the initial material database include the minimum required properties listed in sections 5.5.2 and 5.5.3. At the discretion of the organization(s) developing the database, the test program may include the additional recommended tests listed in section 5.5.3 and any additional tests desired by the prospective end-users of the material. It is strongly recommended that fatigue be among the tested properties.

It is strongly recommended that a minimum of three different material batches consisting of a minimum of two different fiber batches and three different resin batches be used for the initial database (see batch definitions, section 5).

Per the AGATE approach, it is also recommended that laminates for mechanical property data for each batch be processed using two independent cure cycles. The data from the two processing cycles can be considered separate batches when calculating design allowables from the data.

The statistical procedures given in DOT/FAA/AR-00/47 and MIL-HDBK-17 can be used to calculate the material property equivalency requirements and material batch acceptance limits. When using these procedures, the equivalency requirements should be calculated using an alpha = 0.05, while the batch acceptance limits should be calculated using an alpha = 0.01 (note: these recommended values for alpha are currently under investigation by the MIL-HDBK-17 Statistics Working Group, which may issue revised recommendations in the future). 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 are recommended to 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 cured ply thickness, resin content, areal weight, Tg, etc.
- maximum average values for volatile content, void content, etc.

5.6.2 Equivalency Baseline Enhancement.

Since the initial material qualification testing is performed on only three batches of prepreg (containing only three batches of resin and two batches of 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 requirements to test all structural and other properties of interest on each batch, with the test frequency for these tests reduced once the properties are verified to be stable.

The reasons for recommending this additional testing are:

- The additional data will provide a more robust database (closer estimate of the population means and variabilities) 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 ongoing validation of structural properties, thereby minimizing the chance of surprise changes in material properties.

- A larger database will result, thereby providing the potential for higher allowables.

It is recognized that updating statistical basis values may require significant engineering expense to revisit strength calculations. It should be understood that basis values are not a constant value for samples drawn from a population, but that they have a distribution of values. For instance, two samples drawn from the same population will produce two different basis values; both values are valid in the sense that they represent an estimate of the statistical population distribution. For this reason, it is not practical to continually recalculate basis values as new data points are obtained.

The following testing protocol is recommended:

1. After qualification, the equivalency baseline database (EBD) tests listed in section 5.5 should be performed for each prepreg production batch. After the 12th batch of production material is tested, the material equivalency and batch acceptance requirements should be recalculated, and the specification revised with the updated values.

2. If there are no significant changes in the specification requirements from the calculations in step 1, and an SPC analysis of all of the batch data shows that the material is in control, then the EBD testing frequency can be reduced to once every 30 batches or once a year, which ever is more frequent. Normally, the material supplier should not have to obtain formal end-user or FAA approval for the reduction in EBD testing frequency; the batch data will be reviewed as part of the specification update described in step 1.

3. Upon accumulation of each additional ten sets of test results (either from the EBD testing or from intervening material equivalency testing), the material equivalency and batch acceptance requirements should 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.

This testing must be compared to the basis values to assure they are still valid for the new batch and equivalent values.

5.6.3 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. For more discussion of scale-up issues of composite processing see DOT/FAA/AR-02/110. 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; otherwise the tests can be left to the individual end-users of the material.

6. THE QUALITY ASSURANCE SECTION OF THE MATERIAL SPECIFICATION.

This section 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 end-user (purchaser). Key characteristics such as resin content and carbon fiber areal weight are recommended for SPC control. These key characteristics should 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 that 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 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. This determination must always be tempered by the criticality of the part and the acceptable risk level for the operation. 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.

6.1 CHANGES TO QUALIFIED MATERIALS.

This section 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 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 property distributions are not considered equivalent.

DOT/FAA/AR-00/47 provides procedures for evaluating material equivalency. The procedures for material equivalency described in that document are only applicable to specific types of changes and subject to limitations. For details of the changes and conditions, see the report.

The following sections are intended to expand upon the material equivalency guidelines given in DOT/FAA/AR-00/47 by encompassing a greater range of material changes. MIL-HDBK-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 that 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 materials 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 21.93. The approval of minor and major changes are covered in 14 CFR 21.95 and 21.97, respectively. The following sections describe five levels of material changes and the testing and notification requirements associated with these levels.

6.1.1 Level 0 Changes.

These are changes that do not affect 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 of alternate storage facility locations using identical storage conditions. No notification to the end-users is necessary for these changes.

6.1.2 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 recommended examples of level 1 changes are

- Change in release (backing) paper or other process aid
- Alternate vendor for chemically and physically identical raw materials

- Changes to packaging methods and materials

Physical aspects of constituent resin chemicals, such as particulate size and shape, can have a significant effect on the properties of the mixed resin even if the constituents are chemically identical.

Current end-users shall be notified of these changes. Due to potential producibility issues, end-users should approve changes to backing and release papers prior to incorporation. A new revision of the applicable material specification is recommended.

6.1.3 Level 2 Changes.

Due to the type of change involved, this level is considered major by the FAA. 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 that establishes the requirements listed in the material and process specifications will not change. Some recommended 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 and physically similar raw materials
- Changes to test methods that reduce variability
- Modifications to process equipment or processes 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.

A new revision letter for the applicable material specification should be used when this level or higher change is incorporated. Current end-users shall be notified of these changes and approval of the end-users obtained prior to incorporation. End-user approval is only required for those users receiving material to the new revision of the material specification.

6.1.4 Level 3 Changes.

These changes are major changes that are subjected to a full equivalency test program, such as defined in DOT/FAA/AR-00/47. Level 3 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. The supplier should develop and deliver to the end-users a test plan, a description of the change, and the original material and process specifications. Some end-users may require additional tests beyond those given in DOT/FAA/AR-00/47 to address specific critical design issues. Some recommended examples of level 3 changes are

- Change in fiber manufacturing process
- Change in fiber size type, size level, finish, or coupling agents
- Change in resin chemical characteristics (e.g., alternate resin ingredient)
- Change in viscosity of major resin components
- Change in manufacturing site for fiber or resin
- Change in resin mixing, filming and prepregging equipment, process and key process parameters that change key characteristics, or key process parameters
- Change in cure cycle (e.g., temperature, dwell time, and pressure)
- Change in tack
- Change to/from autoclave from/to vacuum pressure cure
- Change in resin content (small)
- Change in nominal number of fibers per tow (small)

Testing to validate level 3 changes should involve a minimum of three batches of prepreg containing three batches of resin and two batches of fiber. The material batch acceptance tests and the EBD tests listed in sections 5.5.2 and 5.5.3 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 requirement values 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 should be used when the change is incorporated. Current end-users shall be notified of these changes, and approval by the end-users obtained prior to incorporation. End-user approval is only required for those users receiving material to the new revision of the process specification.

6.1.5 Level 4 Changes.

Level 4 is a major change, where equivalency testing will not suffice for links to a previous material characterization. Level 4 changes require a new product identification (new specification designation) and a new qualification test program. Level 3 or lower material changes that fail to demonstrate equivalency will typically be considered level 4 changes. Some changes will be considered level 4 changes regardless of the results of equivalency results, due their significant potential effect on material properties or on part fabrication processing. Some recommended examples of level 4 changes are

- Change in resin composition
- Change in resin content (large)
- Change in nominal number of fibers per tow (e.g., 3000 fibers per tow to 6000)
- Change in fiber areal weight (e.g., 145 to 190 gm/m^2) that changes cured ply thickness
- 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 eight harness satin)

Because level 4 changes are considered a new material, existing end-users will not be affected 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 and rotorcraft application.

6.2 SUPPLIER SITE QUALIFICATION.

The prepreg manufacturing site should demonstrate to end-users and certification agencies the capability to conduct raw material testing, final product testing, record maintenance, calibrations, and statistical process control. Training programs and records should 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 should be demonstrated. Major equipment maintenance and modification records should be available. An appropriate organizational structure should exist to ensure that each major function (i.e., operations and quality assurance) can perform their functions.

6.3 STATISTICAL PROCESS CONTROL.

The quality assurance department should maintain the procedures and requirements for SPC based on key characteristics (KC) and key process parameters (KPP). The KCs are a subset of those properties detailed in the uncured and cured prepreg material requirement tables. KCs should be selected such that they ensure all properties of the material are within acceptable statistical limits. These are usually the set of requirements used for acceptance testing. KPPs are

those process parameters that have a significant influence on the KCs. KPPs must be determined prior to qualification and be documented in the PCD. Average values, ranges, limits, and sampling frequency should be established and documented in the PCD.

The procedures used by the quality assurance department to conduct SPC analysis of the KCs and KPPs should be documented in the PCD. The PCD should also include the procedures used to establish and calculate the control limits. It is expected that control charts will be maintained on the KCs and KPPs and will be available for inspection by end-user and FAA personnel. It is strongly recommended that there be an effective program to collect, plot, analyze, and act on KC and KPP data. It is expected that action will be taken when the criteria for nonrandom data trends are met. Action should 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 should be identified and eliminated, re-establishing statistical control. The supplier's quality assurance department must document all corrective actions affecting the process and monitor if the corrective action has been effective.

6.4 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 end-user(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 must 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 reduction of end-user testing or less frequent supplier testing. A prerequisite for reduced testing is adherence to monitoring and action based on control charts.

6.5 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).

6.5.1 Supplier Certification Testing.

The material supplier must perform material certification (acceptance) testing as designated in sections 5.5.2 and 5.5.3. The material specification should define the number of rolls of each prepreg batch that should be tested by the supplier. It is recommended that

- resin content and fiber areal weight testing be performed on every roll in the batch,
- other chemical and physical property tests be performed on at least the first and last rolls in the batch, and
- mechanical testing be performed on at least one roll for every 500 lbs. of prepreg tape in the batch.

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 unless superceded by other requirements. The supplier quality department will review the certification test results prior to shipment to an end-user. Materials that fail the acceptance criteria can undergo a material review board process.

Retest or replacement of test data is allowed only if

- an abnormality is observed or can be reasonably deduced to have occurred during testing,
- data is a statistical outlier, and
- the test has been conducted on materials that have not been prepared or conditioned properly (e.g., machining errors on laminates).

  Note: any testing error should be identified and corrected prior to retest.

If a retest is required, a complete set of replicates for the property should be tested. If the retest results fail the acceptance criteria, the material batch covered by the failed test should be rejected and dispositioned through a material review board (including engineering personnel) process. All end-users of a material batch for which retests were performed must be notified at the time of batch shipment.

6.5.2 End-User (Purchaser) Testing.

The end-user (purchaser) quality department shall perform the specified acceptance tests in sections 5.4 and 5.5 on each prepreg batch. The material specification should define the number of rolls of each prepreg batch that should be tested by the end-user. It is recommended that testing be performed on at least 1 out of every 20 rolls in the 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 end-user should hold to the same record keeping requirements and retest criteria as the material supplier.

An end-user must conduct the acceptance testing whether 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. If material is supplied by a distributor, it is recommended that purchaser testing be maintained as a safeguard against uncontrolled material. It is expected that the FAA will evaluate requests to reduce or eliminate end-user testing on a case-by-case basis (specific FAA policy for reduced

testing approvals will be developed in the future). If end-user 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.

6.5.3 Storage Relifing.

Material storage life is determined by testing conducted on material stored for the maximum desired time (see section 5.4.4). Acceptance testing of material only validates the material for storage up to the time characterized during initial qualification. To relife material that has been in storage beyond its previously validated storage life, it is recommended that a relifing procedure be included in the specification. This will provide a simple standard procedure to validate the use of material that has exceeded its original storage life:

The following is a typical relifing sequence used in specifications:

1. A sample of the material is removed for relife testing.

2. The sample is tested for key characteristics that are affected by storage (aging affects).

3. If the material passes the relife tests, then the material may be used for an additional amount of time. If the material fails, it is scrapped and new material obtained.

4. The storage life is extended as determined from the knowledge of the material volatility and reactivity. A typical first extension is one-half the original storage life. Extension is normally done a maximum of two times with the second extension being for half of the first extension. This would allow an additional life of three-quarters of the original storage life.

   Note: The time extension is material dependent and can only be set after characterization of the specific material's aging behavior.

As an alternate, the determination of extension of storage life may be handled as a material review board (including engineering personnel) action. Again, characterization of the material must be done to give the review board the information necessary to properly extend the shelf life.

A supplier or end-user may choose to run intermediate surveillance tests on the material. This will provide information on the time degradation and age dependency of the material properties. This will reduce the risk of unacceptable material being used for production parts, causing them to be scrapped.

6.6 MATERIAL TEST METHODS.

Recommended test methods for each property are given in Chapters 3 through 7 of Volume 1 of MIL-HDBK-17. In general, ASTM Standard Test Methods are recommended. However in most cases, additional test specimen configuration requirements and test procedures will have to be defined to provide sufficient detail to avoid undesired variations. Deviations from industry

standard methods must be clearly detailed in the specification. In the event that end-user testing is required by the specification, it is recommended that the material supplier and end-user conduct round-robin test evaluations to reduce test result differences.

It is recommended that all testing be conducted by a laboratory certified to conduct the tests to the specified methods; this certification applies to supplier, end-user, and independent test labs. A certified laboratory follows established policies and procedures such as training of test technicians, written procedures for performing tests, documenting the dimensional accuracy of test fixtures, and tracing calibration to National Institute of Standards and Technology (NIST) standards. The specification should define the requirements and procedures for certifying a test lab. It is recommended that for an industry specification, a national laboratory certification be required for facilities used.

6.7 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 material specification, a referenced process specification, or a test panel process instruction document (for further information on process control see DOT/FAA/AR-02/110). Acceptable nominal values and ranges for temperature ramp rates and holds should be defined.

Panels for material qualification, allowables and design values testing should be nondestructively inspected (NDI) to document the quality state of the panels. Test panels for batch acceptance testing do not require NDI, but if the panels are not inspected then the testing organization assumes the risk of testing unacceptable panels.

6.8 MATERIAL DISTRIBUTORS.

Material distributors, either a facility of the manufacturer or an independent facility, must abide by all requirements of the material specification and the applicable portions of the process specification. The end-user should approve a distributor under their supplier surveillance system as described in their quality control manual. It is recommended that the material manufacturer will also have a role in authorizing distributors for proper control of the material. Material batches should remain traceable to the manufacturer's original batch and test reports. The distributor should provide copies of the original material certification and test reports to the user. The manufacturer's material, batch, and lot identification should be maintained.

A distributor should practice the same documentation of storage life and conditions as the material supplier. The distributor should be able to provide objective evidence of the material storage conditions. All shelf life should be determined from the date of material manufacture (impregnation). Any extension of the shelf life allowed by the material specification should be performed by a source approved by the original manufacturer.

If the product is repackaged, the materials used to repackage should be the same as approved for use by the material manufacturer. When the material is repackaged, it should first be allowed to reach room temperature in the unopened package such that moisture does not condense on the prepreg. The repackaged material should be inspected for visual defects and documented. All out-time accumulated during warming, respooling, and repackaging must be subtracted from that specified by the manufacturer and documented for end-users, who assume responsibility after acceptance.

7. THE PACKAGING AND SHIPPING SECTION OF THE MATERIAL SPECIFICATION.

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-time when received by the end-user.

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

7.1 MATERIAL IDENTIFICATION.

The batch number and roll number should 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. The outside label should also clearly define the required material storage conditions.

7.2 INTERLEAF OR RELEASE LAYER.

The prepreg release film or paper should be easily removable, noncontaminating, and controlled as specified by the specification. Changes to a different release paper must demonstrate that no contaminants are transferred from the paper to the product, and that there is no impact on the end-user's producibility requirements. In general, it is desirable that release paper be detectable using NDI techniques.

7.3 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.

7.4 SHIPPING.

The appropriate shipping and storage temperatures must be established for the material. 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.

8. THE ACKNOWLEDGEMENT SECTION OF THE MATERIAL SPECIFICATION.

This section of the specifications should contain the standard phrase:

> "A vendor shall mention this specification number and the applicable detail specification number and their revision letters, if any, in all quotations and when acknowledging purchase orders."

9. THE REJECTION SECTION OF THE MATERIAL SPECIFICATION.

This section should contain the standard phrase:

> "Product not conforming to this specification and the applicable detail specification, or to modifications authorized by purchaser, will be subject to rejection. Rejected batches, by a purchaser, should not be rerouted to other purchasers."

10. THE NOTES SECTION OF THE MATERIAL SPECIFICATION.

This section is reserved for explanatory and other notes.

11. GLOSSARY.

This glossary is a compilation of terms with their definitions used within this report and of general interest. Definitions for this glossary were obtained from a variety of sources, which are noted at the end of the definition. Refer to MIL-HDBK-17 for a more complete listing of terms and their definitions.

Autoclave, n—a closed vessel for conducting a chemical reaction or other operation under pressure and heat (Handbook of Composites).

B Stage, n—an intermediate stage in the reaction of a thermosetting resin in which the material softens when heated and swells when in contact with certain liquids but does not entirely fuse or dissolve. Materials are usually procured to this stage to facilitate handling and processing prior to final cure. See also C Stage (MIL-HDBK-17).

bag, v—the process of enclosing the ply layers within a flexible container. See also vacuum bag (ASTM D 5687).

baseplate, n—a flat plate on which a laminate is laid up. See also mold (ASTM D 5687).

bleeder, n—cloth that allows matrix to flow into it for the purpose of removing excess matrix from the laminate. Net resin prepreg systems do not require the use of bleeder materials (ASTM D 5687).

braided fabric, n—a cloth constructed by a braiding process (ASTM D 3878).

breather, n—cloth which allows even gas flow over the layup surface. The breather also helps minimize bag punctures by protecting the bag from sharp points (ASTM D 5687).

> Discussion: Typically within the bagging layup sequence, the breather material is a mixture of materials. The layer closest to the laminate is a lightweight glass fabric, such as Style 120, in order to minimize mark off on the laminate. The remaining layers are materials selected for their ability to transport gasses under pressure and elevated temperature. Typical materials are heavy weight glasses such as Style 1000 or synthetic nonwoven materials.

breather string, n—a glass string connected from the laminate to a breather in the bagging lay-up. It provides a path for gasses to be transferred from the laminate while minimizing matrix flow (ASTM D 5687).

broadgoods, n—prepreg material (fabric or unidirectional) where the width is greater than 24 inches. See also tape.

C Stage, n—the final stage of the curing reaction of a thermosetting resin in which the material has become practically infusible and insoluble. See also B Stage (MIL-HDBK-17).

caul plate, n—a flat plate used to provide a flat surface to the top of the laminate during laminate consolidation or cure (ASTM D 3878).

CFR—Code of Federal Regulations.

cloth, n—a piece of textile fabric containing woven reinforcement without a load transferring matrix (ASTM D 5687).

composite material, n—a substance consisting of two or more materials, insoluble in one another, which are combined to form a useful engineering material possessing certain properties not possessed by the constituents. Composites are subdivided into classes on the basis of the form of the structural constituents; Laminar: Composed of layer or laminar constituents; Particular: The dispersed phase consists of fibers; Flake: The dispersed phase consists of flat flakes; Skeletal: Composed of a continuous skeletal matrix filled by a second material (ASTM D 3878 and Handbook of Composites).

consolidation, v—the process of forming individual plies into one solid composite laminate. For polymeric based composite materials, consolidation is the compaction of the plies under pressure at elevated temperature until the polymer matrix material is cured.

cure, v—to change the physical properties of a polymer by chemical reaction, which may be by condensation, polymerization, or vulcanization; usually accomplished by the action of heat and catalyst, alone or in combination, with or without pressure (ASTM D 907).

cured ply thickness (CPT), n—the theoretical thickness of an individual ply, which is a function of the fiber areal weight, resin content, fiber density, and resin density.

Discussion: cured per ply thickness is determined from the fiber areal weight, fiber volume, and fiber density:

$$CPT = \frac{FAW}{25400 \rho_f FV}$$

Where:

CPT	is theoretical cured ply thickness (inches)
FAW	is fiber areal weight (g/m^2)
25400	is a units conversion factor
ρ_f	is the fiber density (g/cc)
FV	is fiber volume (fraction, e.g., 0.61)

Or cured per ply thickness can also be determined from the fiber areal weight, resin content, fiber density, and resin density:

$$CPT = \frac{FAW}{25400}\left[\frac{1}{\rho_f} + \frac{RC}{\rho_r(1-RC)}\right]$$

Where:

CPT	is theoretical cured ply thickness (inches)
FAW	is fiber areal weight (g/m^2)
25400	is a units conversion factor
ρ_f	is the fiber density (g/cc)
ρ_r	is the resin density (g/cc)
RC	is resin weight content (fraction, e.g., 0.33)

The actual cured ply thickness is determined by measuring the laminate thickness and dividing it by the number of plies (see SACMA SRM 10).

Fiber volume and resin content are related by the fiber and resin densities:

$$FV = \frac{1-RC}{\rho_f}\left[\frac{1}{1-RC/\rho_f + RC/\rho_r}\right]$$

Where:

FV	is fiber volume (fraction, e.g., 0.61)
ρ_f	is the fiber density (g/cc)
ρ_r	is the resin density (g/cc)
RC	is resin weight content (fraction, e.g., 0.33)

dam, n—a solid material (such as silicone rubber, steel, or aluminum) used in the lay-up to contain the matrix material within defined boundaries during laminate consolidation (ASTM D 5687).

DAR—Designated Airworthiness Representative. FAA designees authorized to conduct conformity inspections on behalf of the FAA.

debulk, v—process of decreasing voids between lamina before laminate consolidation through use of vacuum or by mechanical means. Lamina can be debulked at ambient or elevated temperature (ASTM D 5687).

degree of cure (α), n—in thermoset polymers, the quantity of heat of reaction of the unreacted resin remaining after a reaction (cure cycle) compared to the total available quantity of heat of reaction expended by the complete reaction (cure) of a reacted resin.

Discussion: The degree of cure of a laminate can be obtained from differential scanning calorimetry (DSC) data. In order to obtain the degree of cure of a laminate, the baseline or total heat of reaction released by the complete curing of the resin (or prepreg) must first be obtained. This total heat of reaction is determined from the DSC curve. It is important to obtain the total heat of reaction from a sample that is of the same resin content as the laminate in question. This is typically accomplished by testing a sample of the prepreg used to fabricate the laminate. The laminate in question is then tested to determine the partial heat of reaction. The DSC heating rate used to determine the baseline heat of reaction and partial heat of reaction must be the same. Typically a heating rate of 10°C per minute is used. The degree of cure is calculated as follows:

$$\alpha = 100 - \left(\frac{\Delta H_P}{\Delta H_T} \times 100 \right)$$

Where:

α is the percent degree of cure (ranges from 0% to 100% with 100% being fully cured)

ΔH_P is the heat of reaction released by the partially cured sample (laminate in question) expressed in Joules

ΔH_T is the total heat of reaction released by the uncured resin expressed in Joules (baseline)

Resin formulations commonly used in the aerospace industry rarely reach a degree of cure of 100%. Values of 95% to 98% are common. It should be noted that determining degree of cure by DSC is not considered the most repeatable test and is best limited to research investigations and not used as a production test. Depending on the circumstances, measurement of the glass transition temperature may be the best method to determine if a laminate is fully cured.

DER—Designated Engineering Representative. FAA designees authorized to approve engineering data.

differential scanning calorimetry (DSC), n—a technique in which the temperature difference between the substance and a reference material is measured as a function of temperature while the substance and reference material are subjected to a controlled-temperature program (ASTM E 473).

DMIR—Designated Manufacturing Inspection Representative. FAA designees authorized to conduct conformity inspections on behalf of the FAA.

end, n—in fabric, an individual warp yarn (single or ply) or cord (ASTM D 123).

ETD—Elevated Temperature Dry.

ETW—Elevated Temperature Wet.

FAA—Federal Aviation Administration.

fabric, n—in textiles, a planar structure consisting of yarns or fibers (ASTM D 123).

FEP, n—fluorinated ethylenepropylene.

> Discussion: fluorinated ethylenepropylene is a fluorocarbon polymer commonly known by its DuPont trade name Teflon® FEP.

fiber, n—in textiles, a generic term for any one of the various types of matter that form the basic elements of a textile and that is characterized by having a length at least 100 times its diameter (ASTM D 123).

fiber areal weight (FAW), n—the weight per area of the fiber reinforcement within a composite, expressed as grams per square meter or ounces per square yard. See also prepreg areal weight.

fiber content, n—the amount of fiber present in a composite expressed either as a percent by weight or percent by volume. This is sometimes stated as a fraction, that is, fiber volume fraction (ASTM D 3878).

fiber volume fraction (FV or V_f), n—see fiber content (ASTM D 3878).

filament, n—a fibrous form of matter with an aspect ratio >10 and an effective diameter <1 mm (ASTM D 3878).

fill, n—in a woven fabric, (1) the yarn running from selvage to selvage at right angles to the warp and (2) fiber inserted by the shuttle during weaving also designated as filling (ASTM D 3878, MIL-HDBK-17, and ASTM D 5687).

flip/flop, v—the process of alternating plies through an angle orientation of 180° during laminate lay-up. This practice is commonly used if the material of the same width as the laminate has a

recurring flaw. The process changes the location of the flaw so that it does not unduly affect the laminate structure (ASTM D 5687).

glass transition, n—the reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one (MIL-HDBK-17).

> Discussion: The glass transition generally occurs over a relatively narrow temperature region and is similar to the solidification of a liquid to a glassy state; it is not a phase transition. Not only do hardness and brittleness undergo rapid changes in this temperature region but other properties, such as thermal expansibility and specific heat, also change rapidly. This phenomenon has been called second order transition, rubber transition, and rubbery transition. The word transformation has also been used instead of transition. Where more than one amorphous transition occurs in a polymer, the one associated with segmental motions of the polymer backbone chain or accompanied by the largest change in properties is usually considered to be the glass transition (ASTM D 883).

glass transition temperature (Tg), n—the approximate midpoint of the temperature range over which the glass transition takes place (MIL-HDBK-17).

> Discussion: The glass transition temperature can be determined readily only by observing the temperature at which a significant change takes place in a specific electrical, mechanical, or other physical property. Moreover, the observed temperature can vary significantly depending on the specific property chosen for observation and on details of the experimental technique (for example, rate of heating and frequency). Three common methods for determining Tg are Thermal Mechanical Analysis, Differential Scanning Calorimetry, or Dynamic Mechanical Analysis.

knit, v—a textile process that interlocks, in a specific pattern loop, by means of needles or wires (ASTM D 3878).

knitted fabric, n—a cloth constructed by a knitting process (ASTM D 3878).

lamina, n—a subunit of a laminate consisting of one or more adjacent plies of the same material with identical orientation (ASTM D 3878).

laminate, n—any fiber or fabric-reinforced composite consisting of lamina (plies) with one or more orientations with respect to some reference direction (ASTM D 3878).

lamination, v—see consolidation.

laminate coordinate axes, n—a set of coordinate axes, usually right-handed Cartesian, used as a reference in describing the directional properties and geometrical structure of the laminate. Usually the x-axis and the y-axes lie in the plane of the laminate, and the x-axis is the reference axis from which ply angle is measured (ASTM D 3878).

laminate principal axis, n—the laminate coordinate axis that coincides with the direction of maximum in plane Young's modulus (ASTM D 3878).

lay-up, n—(1) the stack of plies in specified sequence and orientation before cure or consolidation; (2) the complete stack of plies, bagging material, and so on before cure or consolidation; and (3) a description of the component materials, geometry, and so on of a laminate (ASTM D 3878).

lay up, v—to stack plies of material in specified sequence and orientation (ASTM D 3878).

lay-up code, n—a designation system for abbreviating the stacking sequence of laminated composites (ASTM D 3878).

mandrel, n—a form, fixture, or male mold used as the base for production of a part in processes such as lay-up or filament winding (ASTM D 3878).

material form, n—the contour, arrangement, and structure of an unconsolidated composite material, especially with regard to the geometry and nature of the reinforcement. Factors considered part of the material form include, but are not limited to, reinforcement length (for discontinuous reinforcements), tow size or count, fabric areal weight, fabric style, reinforcement content, and ply thickness (ASTM D 3878).

matrix, n—the continuous constituent of a composite material, which surrounds or engulfs embedded filler or reinforcement (ASTM D 3878).

matrix content, n—the amount of matrix present in a composite expressed either as a percent by weight or percent by volume. Standard practice is to specify matrix content as weight percent (ASTM D 3878).

MIDO—Manufacturing Inspection District Office for the FAA.

MOL—Material Operational Limit.

mold, n—the support structure that holds the laminate or lay-up during laminate consolidation process (ASTM D 5687).

MRB—Material Review Board.

NIST—National Institute of Standards and Technology.

nondestructive inspection (NDI), v— to identify and measure abnormal conditions within a laminate without degrading or impairing the utility of the laminate.

nonperforated FEP, n—a nonporous fluorinated ethylenepropylene film used as a release film in the bagging lay-up.

> Discussion: fluorinated ethylenepropylene is a fluorocarbon polymer commonly known by its DuPont trade name Teflon® FEP.

nonperforated TFE, n—a nonporous tetrafluoroethylene film used as a release film in the bagging lay-up (ASTM D 5687).

Discussion: tetrafluoroethylene is a fluorocarbon polymer commonly known by its DuPont trade name Teflon® TFE.

nonporous TFE-coated cloth, n—a cloth coated with tetrafluoroethylene used as a release material in the bagging process (ASTM D 5687).

nonwoven fabric, n—a cloth constructed by bonding or interlocking, or both (but not interlacing), fiber by any combination of mechanical, chemical, thermal, or solvent means (ASTM D 3878).

panel, n—a uniformly contoured composite laminate, typically flat (ASTM D 5687).

peel ply, n—a cloth with release capabilities, usually used in conjunction with laminates requiring secondary bonding (ASTM D 5687).

perforated FEP, n—a porous fluorinated ethylenepropylene film used in the bagging process that allows gasses or excess matrix materials to escape (flow) from a laminate during consolidation while protecting the laminate from physical bonding to other items such as caul plates.

perforated TFE, n—a porous tetrafluoroethylene film used in the bagging process that allows gasses or excess matrix materials to escape (flow) from a laminate during consolidation while protecting the laminate from physical bonding to other items such as caul plates (ASTM D 5687).

plied yarn, n—a yarn formed by twisting together two or more single yarns in one operation (ASTM D 3878).

ply, n—in laminar composites, the constituent single layer as used in fabricating or occurring within a composite structure (ASTM D 3878).

ply coordinate axes, n—a set of Cartesian coordinates, two of which lie within the plane of the ply, one axis of which is parallel to the principal fiber direction and the other axis perpendicular to the principal fiber direction (the third axis is through the ply's thickness) (ASTM D 3878).

ply count, n—in laminated composite materials, the number of plies or lamina used to construct the composite (ASTM D 3878).

ply orientation, n—the acute angle (theta) including 90° between a reference direction and the ply principal axis. The ply orientation is positive if measured counterclockwise from the reference direction and negative if measured clockwise (ASTM D 3878).

ply principal axis, n—the ply coordinate axis that coincides with the direction of maximum in plane Young's modulus. For balance weave fabric, either warp or fill direction may be chosen (ASTM D 3878).

polymer, n—an organic material composed of molecules characterized by the repetition of one or more types of monomeric units (MIL-HDBK-17).

polymerization, n—a chemical reaction in which the molecules of a monomer(s) are linked together in repeating units to form larger molecules (ASTM D 907).

porosity, n—a condition of trapped pockets of air, gas, or vacuum within a solid material, usually expressed as a percentage of the total nonsolid volume to the total volume (solid plus nonsolid) of a unit quantity of material (MIL-HDBK-17).

porous TFE-coated cloth, n—a porous cloth coated with tetrafluoroethylene used in the bagging process that allows gasses or excess matrix materials to escape (flow) from a laminate during consolidation. It differs from perforated TFE in that it gives a textured surface to the laminate (ASTM D 5687).

prepreg, n—a ready to mold or cure fibrous reinforcement impregnated with a polymeric matrix. Its form may be sheet, tape, or tow. For thermosetting matrices, it has been partially cured to a controlled viscosity called B stage (ASTM D 3878 and MIL-HDBK-17).

prepreg areal weight (PAW), n—the weight per area of the prepreg composite material, expressed as pounds per square foot or the inverse square feet per pound. Used as a conversion factor to convert prepreg area to prepreg weight. See also fiber areal weight.

Discussion: Prepreg areal weight is a function of resin content and fiber areal weight:

$$PAW = \frac{FAW}{1 - RC}$$

Where:

PAW is prepreg areal weight (g/m^2)
FAW is fiber areal weight (g/m^2)
RC is resin weight content (fraction, e.g., 0.33)

To convert g/m^2 to lb/ft^2 multiply by 204.81×10^{-6}.

reinforcement, n—in a composite material, the discrete constituent of a composite material, either fiber or particle, which is contained within the continuous matrix (ASTM D 3878).

resin, n—a solid or pseudosolid organic material, often of high molecular weight, which exhibits a tendency to flow when subjected to stress, usually has a high softening or melting range, and usually fractures conchoidally (ASTM D 3878).

resin content (RC), n—see matrix content (ASTM D 3878).

sealant, n—a high-temperature material used to seal the edges of a vacuum bag to a base plate during consolidation (ASTM D 5687).

selvage, n—the woven edge portion of a fabric parallel to the warp (ASTM D 3878).

single yarn, n—an end in which each filament follows the same twist (ASTM D 3878).

stacking sequence, n—the arrangement of ply orientations and material components in a laminate specified with respect to some reference direction (ASTM D 3878).

staggered, adj—the description of ply placement where the joints are not positioned in the same in-plane location through some specified thickness of the laminate (ASTM D 5687).

tape, n—prepreg material (typically unidirectional material) equal to or less than 24 inches in width. Also see broadgoods.

textile, n—a general term applied to fibers and organized assemblies of fibers with sufficient integrity to retain the organization (ASTM D 3878).

thermoplastic, n—a plastic that repeatedly can be softened by heating and hardened by cooling through a temperature range characteristic of the plastic, and that in the softened state can be shaped by flow into articles by molding or extrusion for example (ASTM D 883).

thermoset, n—a class of polymers that, when cured using heat, chemical, or other means, changes into a substantially infusible and insoluble material (ASTM D 3878).

tow, n—in fibrous composites, a continuous, ordered assembly of essentially parallel, collimated filaments, normally without twist and of continuous filaments (ASTM D 3878).

traveler, n—a coupon with the same nominal thickness and, preferably, width as the test specimen, made of the same material, and processed similarly to the specimen except usually without tabs or gages. The traveler is used to measure mass changes during environmental conditioning when it is impractical to measure these changes on the actual specimen (ASTM D 5687).

traveler panel, n—(aka witness panel) a panel that is subjected to the same conditions as a part or group of parts to allow destructive testing to verify processing.

unidirectional, n—any fiber-reinforced composite with all the fibers aligned in a single direction. Both prepreg material and consolidated laminates can be described as being unidirectional.

vacuum bag, n—a low gas permeable material used to enclose and seal the lay-up during a consolidation or debulking cycle (ASTM D 5687).

vacuum couple, n—the mechanical connection that seals the vacuum source to the lay-up during a consolidation or debulking cycle (ASTM D 5687).

vitrification, n—the point during polymerization where the Tg of the polymer rises above the temperature of cure.

void, n—any pocket of enclosed gas or air within a composite (ASTM D 3878).

void content, n—the volume percentage of voids in a composite (ASTM D 3878).

warp, n—(1) the yarn running lengthwise in a woven fabric; (2) a group of yarns in long lengths and approximately parallel, put on beams or warp reels for further textile processing including weaving, knitting, twisting, dyeing, and so forth (ASTM D 3878).

warp surface, n—the ply surface that shows the larger area of warp tows with respect to fill tows (ASTM D 3878).

warp surface nesting, v—process of laying up fabric plies in an alternating pattern where the warp surface is placed up and then for the next ply the warp surface is placed down, thus nesting the plies.

weave, v—interlaces, in a specific pattern, strands or yarns orientated in two or more directions in a planar textile process (ASTM D 3878).

woven fabric, n —a cloth constructed by a weaving process (ASTM D 3878).

yarn, n—in fibrous composites, a continuous, ordered assembly of essentially parallel, collimated filaments, normally with twist, and either discontinuous or continuous filaments (ASTM D 3878).

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., improved Infrared Spectrophotometry and High-Performance Liquid Chromatography methods.

- Improved prepreg rheology test methodology. Current prepreg flow measurements are too subjective and have high variability.

- Development and implementation of in-line measurements for resin content and fiber areal weight for use during prepregging.

- Automated visual systems for detection of in-line 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.

- Improved methods for identification of sources of variability in composite materials and laminates (such as through the use of nested experimental designs)

- Development of advanced prepreg chemical/physical characterization techniques and analysis methods for detecting significant changes in prepreg materials.

- Improved procedures for evaluating and approving changes to qualified materials (systems approach).

- Identification and development of monitorable key characteristics of the raw material which directly correlate to physical and mechanical properties.