

**Guidelines and Recommendations for
the Development of a Material
Specification for
Carbon Fiber Reinforced Liquid Resin
Molded Materials**

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FAA Workshop on Key Characteristics for Advanced
Material Control

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Document Development

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- Time Line

- First Draft: September 2003
- FAA Workshop: September 2003
- Rework Draft with Workshop Input: September - October 2003
- Final Draft to FAA: November 2003
- Release by FAA: TBD

Introduction

- Incorporated the philosophy developed for the uni-directional tape and fabric documents.
- Fabric selected as preform product.
- Familiar industry standards for manufacture, design, and use.
- Addresses common themes such as resin mixing, combining resin with preform, and testing.

Document Outline

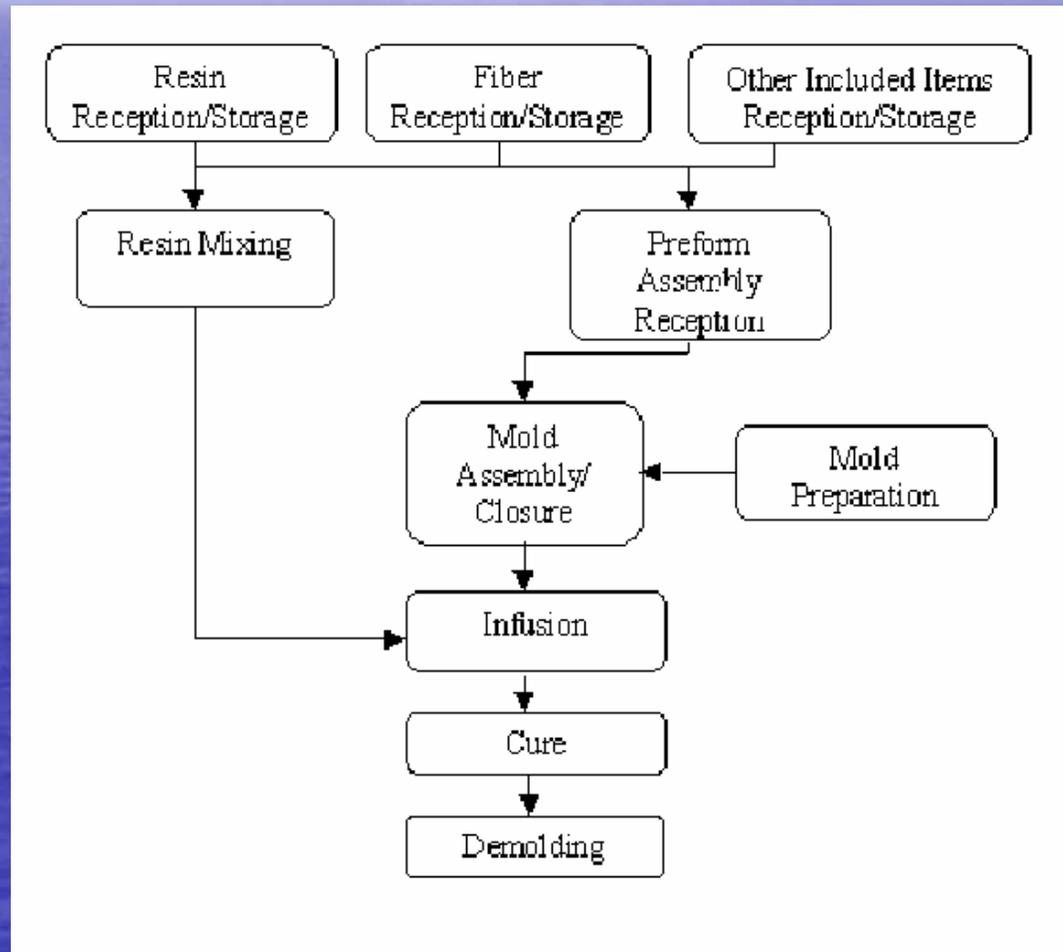
Executive Summary

1. Introduction.
 - 1.1 Objective.
 - 1.2 Background.
 - 1.3 Related Documents
 - 1.4 Certification Process.
2. Liquid Resin Molding Process and Materials
 - 2.1 Liquid Molding Process
 - 2.2 Liquid Molding Materials
3. Development of Material Controls.
 - 3.1 Industry Material Specification.
 - 3.2 Part Producer Material Specification.
 - 3.3 Part Producer Responsibilities for Material Use in Structural Design
 - 3.4 Material Qualification Process When Using an Industry Material Specification.
 - 3.5 Material Qualification Process When Using an Part producer Material Specification

Document Outline

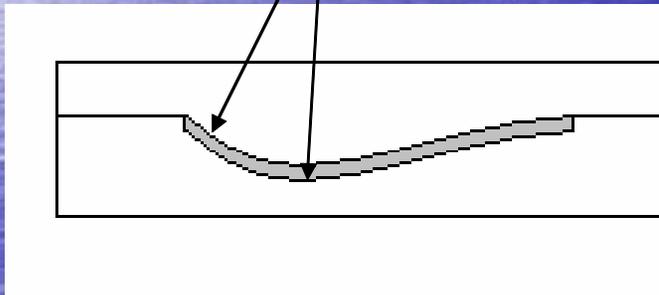
4. Qualification Requirements
 - 4.1 Initial Material Qualification
 - 4.2 Minimum Level Of Testing
 - 4.3 Equivalency Baseline Enhancement
 - 4.4 Additional Characterization Testing For Specific Design Applications
 - 4.5 Supplier Site Qualification
 - 4.6 Changes To Qualified Materials
5. Recommended LRM Specification Structure
 - 5.1 Recommended Specification Structure
 - 5.2 Sectional Arrangement of Specification
6. Guidelines for Sections of a Resin Material Procurement Specification
7. Guidelines for Sections of a Fabric Material Procurement Specification
8. Guidelines for Sections of a Material Acceptance Specification for Composite Parts

Generic Liquid Resin Molding Process Flow



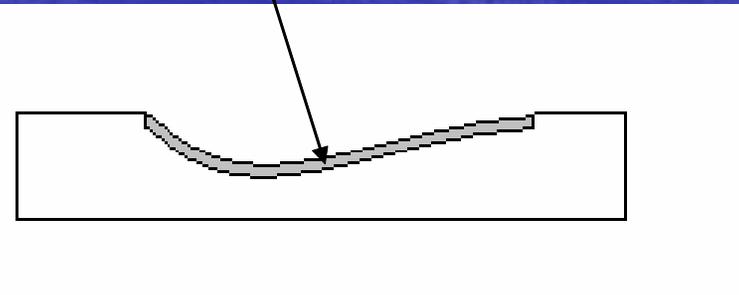
Liquid Resin Molding Process Tooling Options

All surfaces controlled by rigid tooling



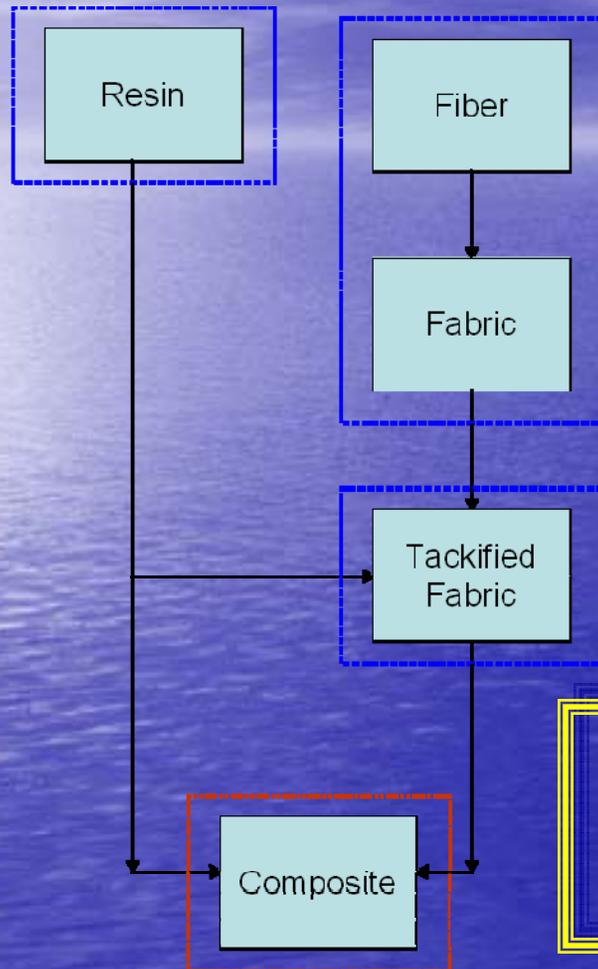
Closed-cavity Mold

Only a portion of the part controlled by rigid tooling



Open-cavity Mold

LRM Material Flow

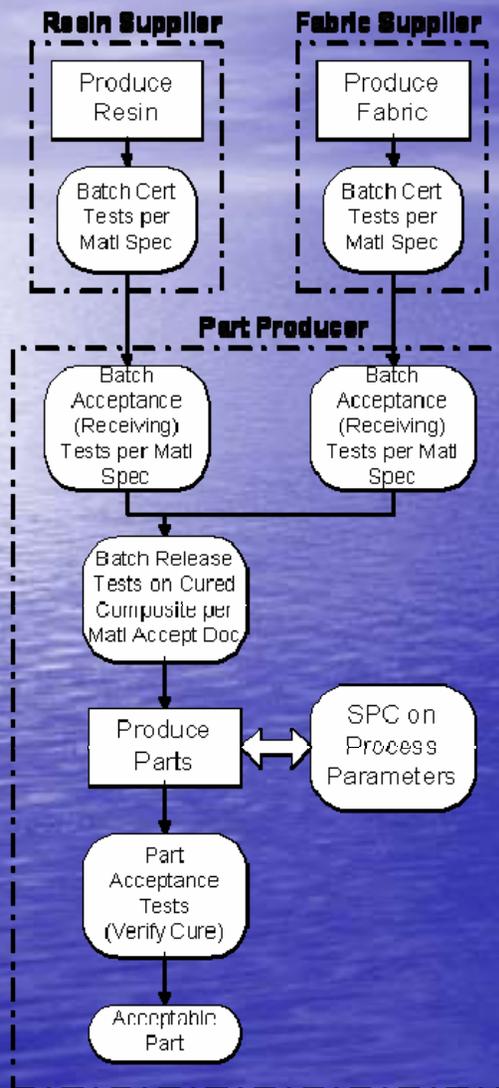


- Reactive Resin Material
- Non-Reactive Reinforcement Material
- Assembled Material (Preform)
- Consumable & Ancillary Materials

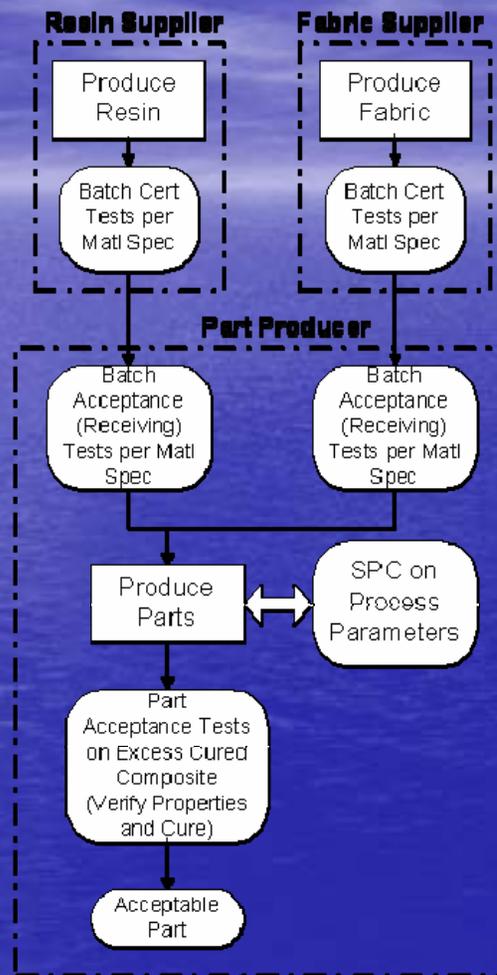
Possible to combine different resin batches with different fabric batches. Do we need to Test all possible combinations?

 Material Procurement Specification
 Material Acceptance Specification

TEST APPROACH A



TEST APPROACH B



Test Approach Based On Risk.

Material Control Approach

- One approach for material control of liquid resin molded parts is the use of material procurement specifications for the constituent materials (resin and reinforcement) in combination with an a material acceptance specification for the cured parts.
- The constituent materials are controlled through material procurement specifications.
- The combined system is then controlled at the cured composite assembly (resin combined with reinforcement) through an acceptance specification by the purchaser part producer.
- This same document would can define the process for qualifying the material system.

Let the Material Drive the Specification

Traditional Approach

- Users develop a specification and material suppliers strive to provide materials that meet the requirements. The same material is be qualified to many different specifications.
- Limited number of batches qualifies a material to the user specification. The qualification tests are rarely run again.
- This approach leads to:
 - A 'test to pass' minimum specification values mentality.
 - A lack of knowing what the true material variability is.
 - Barn door manufacturing process limits to ensure the supplier had plenty of room to make material that passes.
 - Preaching, but not practicing, SPC.
 - A failure to continually add to the qualification database and keep test methods current.
 - Scary re-qualifications.

Let the Material Drive the Specification

Shared Database Approach

- Material supplier can also establish the database for an industry material and define the specification.
- Establish preliminary targets and ranges for material characteristics and properties in a limited 'qualification':
 - Acceptance testing for certification and purchase
 - Minimum testing for database.
 - Additional testing to show material is acceptable for intended use.
- Database should be augmented on an ongoing basis:
 - Establish true material variability.
 - Allow for SPC monitoring and control.
 - No shocks or surprises when a change happens.
 - Spec acceptance limits are unique for EACH material and based on properties of that material – NO requirements based on the minimum of a bunch of materials!

Encourage Use of Industry Standards for Definitions of Terms, Test Methods, and Change Level

- Test methods can come from SACMA and ASTM.
- Definitions can come from MIL-HDBK-17.
- Methods for calculating specification limits are found in DOT/FAA AR/00-47.
- Allowables can be calculated from procedures found in MIL-HDBK-17.

Development of Material Controls: Industry Material Specification/Qualification

- Supplier will:
 - Develop new material or make an industry specification for an old material.
 - Stabilize the production process.
 - Establish the cure cycle to be used for the qualification database.
 - Perform minimum qualification tests per FAA approval and witnessing.
 - Develop statistical material control limits and allowable values.
 - Submit data to an industry committee for approval.
- Part Fabricator / End-user will:
 - Demonstrate equivalency to industry-approved data base and use material allowables.
 - End-user will perform design verification and certification tests.

Development of Material Controls: Part Fabricator / End-User Material Specification/Qualification

- Supplier will:
 - Develop new material or supply an old material.
 - Stabilize the production process.
 - Establish the cure cycle to be used for the qualification database.
 - Supply to an End-user
- The Part Fabricator / End-user will:
 - Perform minimum qualification tests per FAA approval and witnessing.
 - Develop statistical material control limits and allowable values.
 - Perform design verification and certification tests.

Development of Material Controls: Part Fabricator / End-User Responsibilities

- Qualify the material for use in a specific application.
- Validate design details.
- If an Industry Material Specification is selected, conduct tests to show statistical equivalency to original property database.
- Conduct tests to validate any deviations from baseline cure cycle.
- Conduct acceptance testing to receive material to industry or end-user material specification

Technical Requirements: Batch Definitions

- Fiber - made in one continuous run under the same process conditions using one to three precursor batches with up to a 72 hour interruption.
- Fabric - woven from up to three batches of fiber without an interruption greater than 72 hours.
- Resin - depends on mixing process but typically means that the same batches of raw materials are used and the actual mix process is not interrupted. Some blending of individual raw material batches is permitted as long as the overall resin has the same mix ratio of the specific ingredients.

Establishing and Following a Process Control Document is Critical to The New Philosophy

- The PCD should document all aspects of the material fabrication.
 - Raw materials.
 - Key process parameters.
 - SPC procedures and requirements.
- Process parameter targets and ranges should be determined by engineering trials and should be re-evaluated on an ongoing basis like the material property database.
- The PCD is maintained by the material supplier and should be made available for review by the end-users and certification agencies.

Recommend Minimum Testing Requirements so the PCD is Data Driven

- It is expected that behind the process limits and in process tests is a body of data and an understanding of the material and the effects of the various processing steps upon the material.
- Selection of appropriate test methods and generation of data will result in a database that can ensure the sale of consistent material and a reservoir of knowledge to aid problem solving and identify material changes.
- Recommended that tolerances be stacked and the resulting material evaluated to demonstrate that its cure characteristics and mechanical properties are still within statistical limits.
- Effects of stacked tolerances on handling characteristics such as tack and drape, and cured composite quality, should be evaluated.

Technical Requirements: Neat Resin Properties

- Recommended that the supplier develop a database of resin properties.
- Although resin properties are not used in design, the database will enable processes to be developed with an fundamental knowledge and understanding of the resin composition and reactivity.
- Test methods include, but are not limited to:
 - Density
 - Viscosity
 - Gel time
 - Infrared spectroscopy
 - High performance liquid chromatography
 - Rheology
 - Reaction kinetics
- Tests will have to be modified to deal with solvated resins as solvents have a profound effect on the rheology of resin.

Technical Requirements: Carbon Fiber and Fabric Properties

- Carbon fiber must be purchased to a specification tied to either the prepreg or fabric specification.
- Not only should mechanical properties such as tensile strength, modulus and elongation be part of the batch acceptance testing, but the size type and level must be specified as well as tow bundle count and twist.
- Fiber specification should define the average values and ranges for all critical mechanical and physical
- Fabric specification should establish the critical fiber properties, fabric areal weight and fabric style.
- Physical characteristics such as width, tracer type and spacing, alignment, openness, yarn count per inch should be defined.

Technical Requirements: Cured Composite Material Requirements

- Document is not a typical material procurement document, rather it defines the qualified constituent materials, and the cured material property requirements that the part producer themselves are required to demonstrate adherence to for each production part
- Test panels are to be fabricated using the same processes used to develop qualification and design data.

Technical Requirements: Recommended Mechanical Property Test Matrices

- Minimum set
 - Unidirectional and cross-ply strength and modulus
 - Batch acceptance tests
 - RT [90] (fill) tension
 - Hot [90] (fill) compression
 - RT SB shear
- Additional for general applications
 - Open hole tension, compression strength
- Additional for expanded database
 - Market/application driven
- Essentially the same as recommended for tape prepreg materials

Technical Requirements: Minimum Set of Mechanical Properties

Recommended Layups:

Lay-up Name	Ply Lay-up Sequence (starting from toolside)	Recommended Thickness Range for Selecting 'n' (inches)
1. Warp Tension	[0*]n	0.060 to 0.100
2. Warp Compression	[0*]n	0.100 to 0.150
3. Fill Tension	[90*]n	0.060 to 0.100
4. Fill Compression	[90*]n	0.100 to 0.150
5. ±45 Shear	[45/-45/45/-45 // -45*/45*/-45*/45*]	
6. Warp Interlaminar	[0*]n	0.240 to 0.260
7. Quasi Laminate	[(45/0/-45/0)n // (0*/-45*/0*/45*)n]	0.100 to 0.150
8. Soft Laminate	[(45/-45/0/45/-45)n // (-45*/45*/0*/-45*/45*)]	0.100 to 0.150
9. Hard Laminate	[(0/0/45/0/0)n // (0*/0*/45*/0*/0*)]	0.100 to 0.150
10. Warp Sandwich	[0*/0*/ core /0/0] (warp face next to core)	
11. Fill Sandwich	[90*/90*/ core /90/90] (warp face next to core)	
12. Quasi Sandwich	[45*/0*/ core /0/45] (warp face next to core)	
13. Warp Toughness	[0*]n	0.120 to 0.200
14. Quasi CAI	[(45/0/-45/0)n // (0*/-45*/0*/45*)n]	0.140 to 0.200

Technical Requirements: Minimum Set of Mechanical Properties

Recommended Test Matrix:

Layup (see Table 5A)	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
1. Warp Tension	0 (Warp) Tension ASTM D 3039	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 ²	1 x 2 x 3	
2. Warp Compression	0 (Warp) Compression ASTM D 6641	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 ²	1 x 2 x 3	1 x 2 x 3
3. Fill Tension	90 (Fill) Tension ASTM D 3039	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 ¹	1 x 2 x 3 ²	3 x 2 x 3
4. Fill Compression	90 (Fill) Compression ASTM D 6641	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 ²	1 x 2 x 3 ¹	3 x 2 x 3
5. ±45 Shear	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
6. Warp Interlaminar	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)

Technical Requirements: Recommended Expanded Database Tests

- Quasi, hard, soft layups:
 - Unnotched tension and compression
 - Open hole tension and compression
 - Filled hole tension and compression
 - Bearing
- Interlaminar shear
- Sandwich flexure (unnotched, open hole, impacted)
- In-plane shear (solvent sensitivities)
- Fracture toughness
- Laminate compression after impact
- Open hole fatigue

Technical Requirements: Recommended Expanded Database Tests

- Generally critical environmental conditions shown with a ✓
- Portion of matrix shown below:

Layup (see Table 5A)	Test Type and Direction	Property	Test Temperature/Moisture Condition			
			Lowest Temperature / Ambient	70°F/ Ambient	Highest Temperature / Ambient	Highest Temperature / Wet
7. Quasi Laminate	Unnotched Tension ASTM D3039	Ultimate Strength	✓	✓		✓
8. Soft Laminate	Unnotched Tension ASTM D3039	Ultimate Strength	✓	✓		✓
9. Hard Laminate	Unnotched Tension ASTM D3039	Ultimate Strength	✓	✓		✓
7. Quasi Laminate	Unnotched Compression	Ultimate Strength		✓	✓	✓
8. Soft Laminate	Unnotched Compression	Ultimate Strength		✓		✓

Technical Requirements: Qualification

- Initial material characterization (Qualification)
 - Minimum of 3 prepreg batches (3 resin, 2 fiber batches)
 - Use AGATE developed procedures for establishing acceptance limits and equivalency requirements
 - Max average, min average and min individuals for strength properties.
 - Max and min average values for stiffness properties
 - Max and min average values for cured ply thickness, resin content, areal weight, Tg, etc.
 - Max average values for volatile content, void content, etc.
 - Acceptance Tests:
 - Fiber, resin, volatile contents; flow; HPLC; cured thickness
 - RT [90] (fill) tension, Hot [90] (fill) compression, RT SB shear

Technical Requirements: Expanded Batch Testing

- Recommends expanded batch testing:
 - Provide robust equivalency database
 - Ongoing validation of structural properties
 - Reduce chances for “surprises” during re-qualifications, equivalency demonstrations
 - Early detection of drift in properties so corrective action can be taken
 - Potential for higher allowables
 - Perform tests for 1st 12 production batches
 - Recalculate spec acceptance/equivalency requirements
 - If material in control, reduce to once every 30 batches
 - After 10 sets of data, recalculate spec requirements

Technical Requirements: Expanded Batch Testing

- Expanded batch testing
(recommended tests for equivalency baseline database):
 - Tg
 - RT [0] (warp) tension, compression strength, modulus
 - Hot [90] (fill) tension strength, modulus
 - RT [90] (fill) compression strength, modulus
 - RT In-plane shear
 - RT, Hot Open hole tension, compression

Quality Assurance: Changes to Qualified Materials

- Expansion of AGATE equivalency procedures
- Defines 5 levels of change
 - Based on FAA draft composite material TSO
 - Recommends types of changes that apply to each level
- Degree of FAA approval of each level of change is not covered in detail in these guidelines documents
 - Major changes effect material allowables or acceptance limits per 14 CFR 21.93
 - Approval of minor and major changes covered in 14 CFR 21.95 and 21.97

Quality Assurance: A Clear Protocol for Dealing With Change

- Level 0 (Minor Change)
 - Change in ingredient name, company name, correction of typos
 - No notification of end users is required
- Level 1 (Minor Change)
 - Changes to packaging materials
 - Alternate vendor for chemically and physically identical raw materials
 - Current end users are notified, but approval by end user not required
- Level 2 (Major Change)
 - Change in resin ingredient supplier, change in resin ingredient precursor, modifications of process equipment, addition of new similar equipment.
 - New source for chemically and physically similar raw materials
 - Notify and obtain approval of current end users

Quality Assurance: A Clear Protocol for Dealing With Change

- Level 3 (Major Change)
 - Change in fiber manufacturing process, fiber size or finish
 - Change in resin chemistry, viscosity of resin components
 - Change in resin mixing, filming and prepregging equipment, change in resin or fiber manufacturing site.
 - Change in cure cycle
 - Full equivalency testing and end user approval
- Level 4 (Major Change)
 - Change in fiber type, areal weight, tow count, manufacturer
 - Change in fabric weave
 - Change in resin formulation, large change in resin content
 - New product specification

Quality Assurance: SPC Methods are Key to Reducing Variability

- Recommends SPC for key characteristics and key process parameters
 - KCs: batch acceptance tests
 - KPPs: resin mixing and fabric weaving parameters that have a significant influence on the KCs
 - Determine prior to qualification
 - Document in PCD
 - Control limits set based on natural variability of product and processes.
 - Data should be collected, plotted, analyzed and acted upon
 - Action should be taken based on standard criteria for non-random variability and before data shows product is outside the control limits.

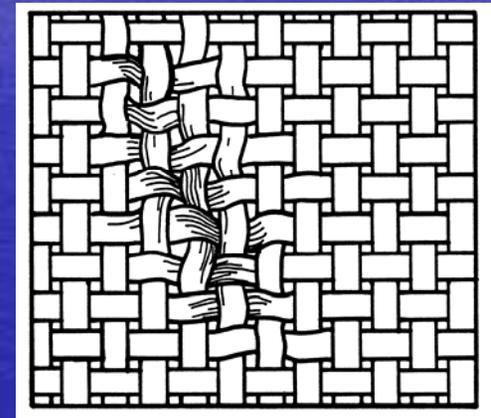
Quality Assurance: Reduced Testing

- Requires use of SPC to monitor:
 - Resin Mixing & Fabric Weaving process (KPPs)
 - Material properties (KCs)
- Requires high levels of SPC control and capability
 - Demonstrated over minimum of several years of prepreg production
- Requires approval of FAA and end-users
- Must be documented in the PCD

Miscellaneous Topics

- Product Certification
- Test Methods
- Storage Life Revalidation
- Material Test Methods
- Material Distributors
- Glossary
 - General terms (from tape document)
 - Fabric defect definitions and figures

Crease or wrinkle:



Workshop Goals for LRM Materials Document

- Comments, criticisms, and contributions from you.
 - The goal is to reduce cost to both suppliers and users while maintaining safety.
 - Focus discussions on LRM material specific issues.
- All input will be reviewed, discussed, and incorporated into the final document.
- Thanks in advance for your help.