Composite Structure Engineering Safety Awareness Course

Module 3:
Stabilizing Materials and Processes

Steve Ward, SWComposites

September 14-16, 2010

Stabilizing Composite Materials and Processes

AGENDA

- Why do we need stable materials?
- What is a stable material
- Property control
- “Higher” properties
- Multiple material sources
- What is (is not) material qualification
- What is needed before material qualification

References

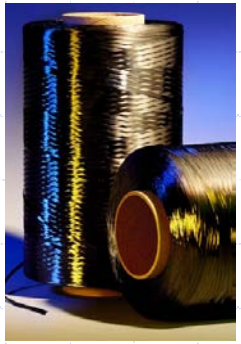
- FAA AC20-107B, Composite Aircraft Structure
- FAA AC21-26 Quality Control for the Manufacture of Composite Structures
- FAA AC23-20, Acceptance Guidance on Material Procurement and Process Specifications for Polymer Matrix Composite Systems
- FAA AC29 MG 8, Substantiation of Composite Rotorcraft Structure

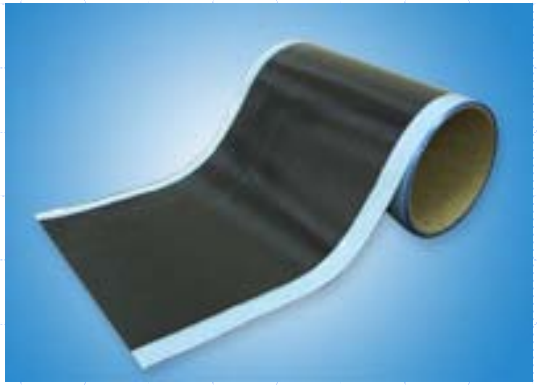
- CMH-17 Composite Materials Handbook, Rev G

- DOT/FAA/AR-03/19, Material Qualification and Equivalency for Polymer Matrix Composite Material Systems: Updated Procedure
- DOT/FAA/AR-03/21, Characterization of In-Plane, Shear-Loaded Adhesive Lap Joints: Experiments and Analysis
- DOT/FAA/AR-07/3, Guidelines and Recommended Criteria for the Development of a Material Specification for Carbon Fiber/Epoxy Unidirectional Prepregs Update
- DOT/FAA/AR-02/110, Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber-Reinforced Polymer Composites
- DOT/FAA/AR-06/10, Guidelines and Recommended Criteria for the Development of a Material Specification for Carbon Fiber/Epoxy Fabric Prepregs
- DOT/FAA/AR-06/25, Preliminary Guidelines and Recommendations for the Development of Material and Process Specifications for Carbon Fiber-Reinforced Liquid Resin Molded Materials

  - See also other references listed in AC20-107B, appendix 1

What is a “material”?





- Raw materials
  - Prepreg (resin + fiber purchased together)
  - Resin, fibers purchased separate for liquid molding processes (RTM, VARTM, RFI, etc.)
  - Controlled by material specifications and PCDs
- Fabricated parts
  - Controlled by process specifications
  - Liquid molding
    - Fabricator has to control properties of the combined fiber and resin “material”

Composite Materials

- Final material is actually created during part fabrication.
- Material Specifications control raw material
- Design properties are dependent on the fabrication process used to produce the part

- Process Specifications control fabrication process
- Design allowables are tied to both the materials and process used to fabricate parts
- Design allowables often depend on the analytical methods used in design

Why do we need stable materials?

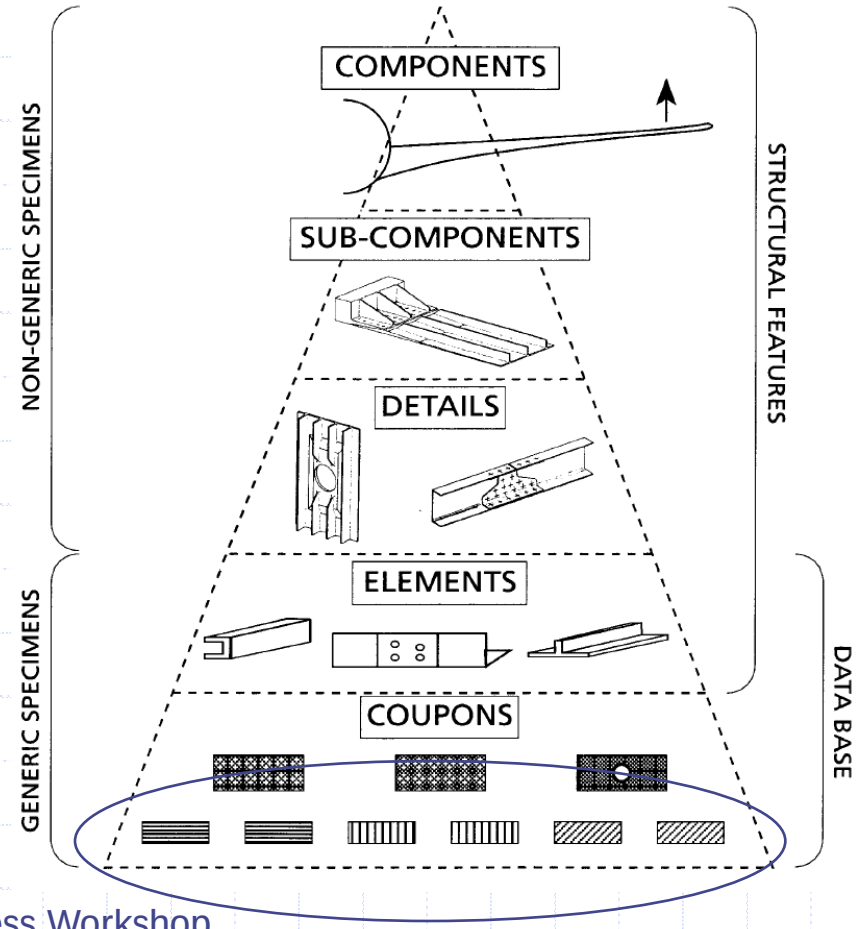
- Materials used for parts ... must ... conform to approved specifications ... that ensure their having the strength and other properties assumed in the design data (FAR § 2X.603)
- The methods of fabrication used must produce a consistently sound structure (FAR § 2X.605)

Note: In order to derive design values which satisfy the next bullet a certain "maturity" in the controlling material and processing specifications must be achieved. Values derived prior to controlling the process to provide a "stable and repeatable" product may not reflect the actual capacities of the product.

- Material strength properties must ... minimize the probability of structural failures due to material variability (FAR § 2X.613)

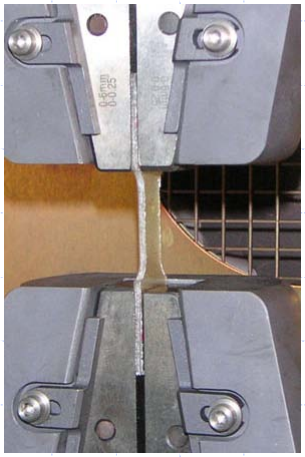
Why do we need stable materials?

- Uncontrolled and/or excessive variability at the material level
(typically evaluated and controlled at the base coupon level)
can cause the entire certification pyramid to collapse
(design values and certification tests do not represent actual parts)



Why do we need stable materials?

- We are using this:



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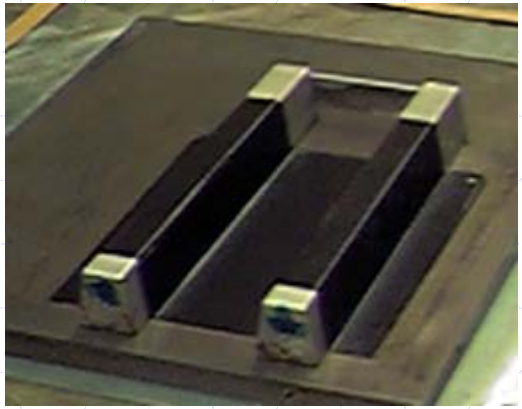

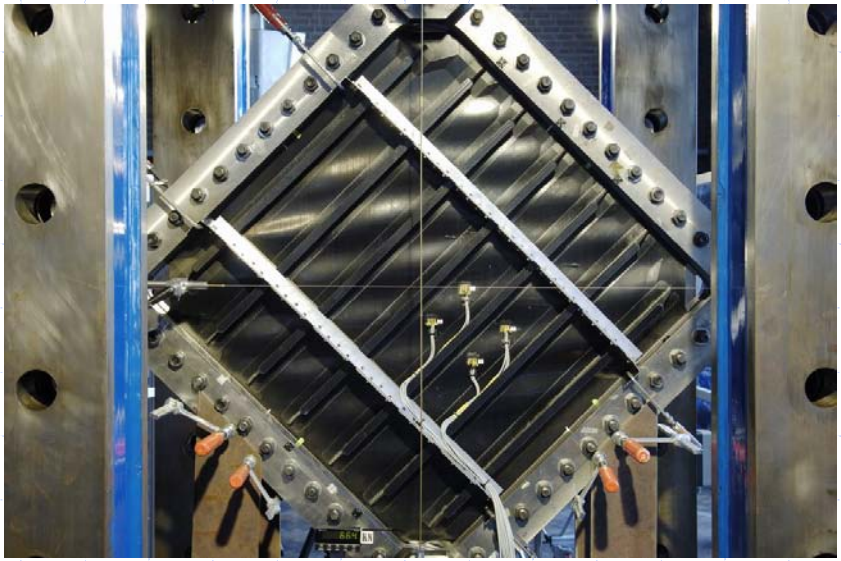

Why do we need stable materials?

- To protect this:

Why do we need stable materials?

Safety ↑
Cost ↓

- FAA certification requirement
  - Qualification data and allowables represent actual material properties
  - Structural building block tests are representative
- Ensure fabrication trials are representative
- Cost avoidance, schedule risk reduction
  - Additional tests
  - Rejected materials
  - Part fabrication problems / rejected parts

Stabilizing Composite Materials and Processes

AGENDA

- Why do we need stable materials
- What is a stable material
- Property control
- “Higher” properties
- Multiple material sources
- What is (is not) material qualification
- What is needed before material qualification

What is a stable material?
Raw materials (prepreg, etc)

- Low variation over time:
  - Chemistry, RC, FAW, PPT, Tack, Drape
  - Mechanical properties, etc

- Production scale equipment
- Locked in PCD
- SPC controls
  - All of above for fiber manufacture, resin mixing, prepregging

What is a stable material?
Fabricated parts

- Low variation over time
- Stable and controlled processes
  - Layup
  - Tooling
  - Cure process
- Liquid molding
  - Requires some type of “material batch control” of fiber/resin combination to be done by part fabricator
    - Batch panels, witness panels, prolongs, etc

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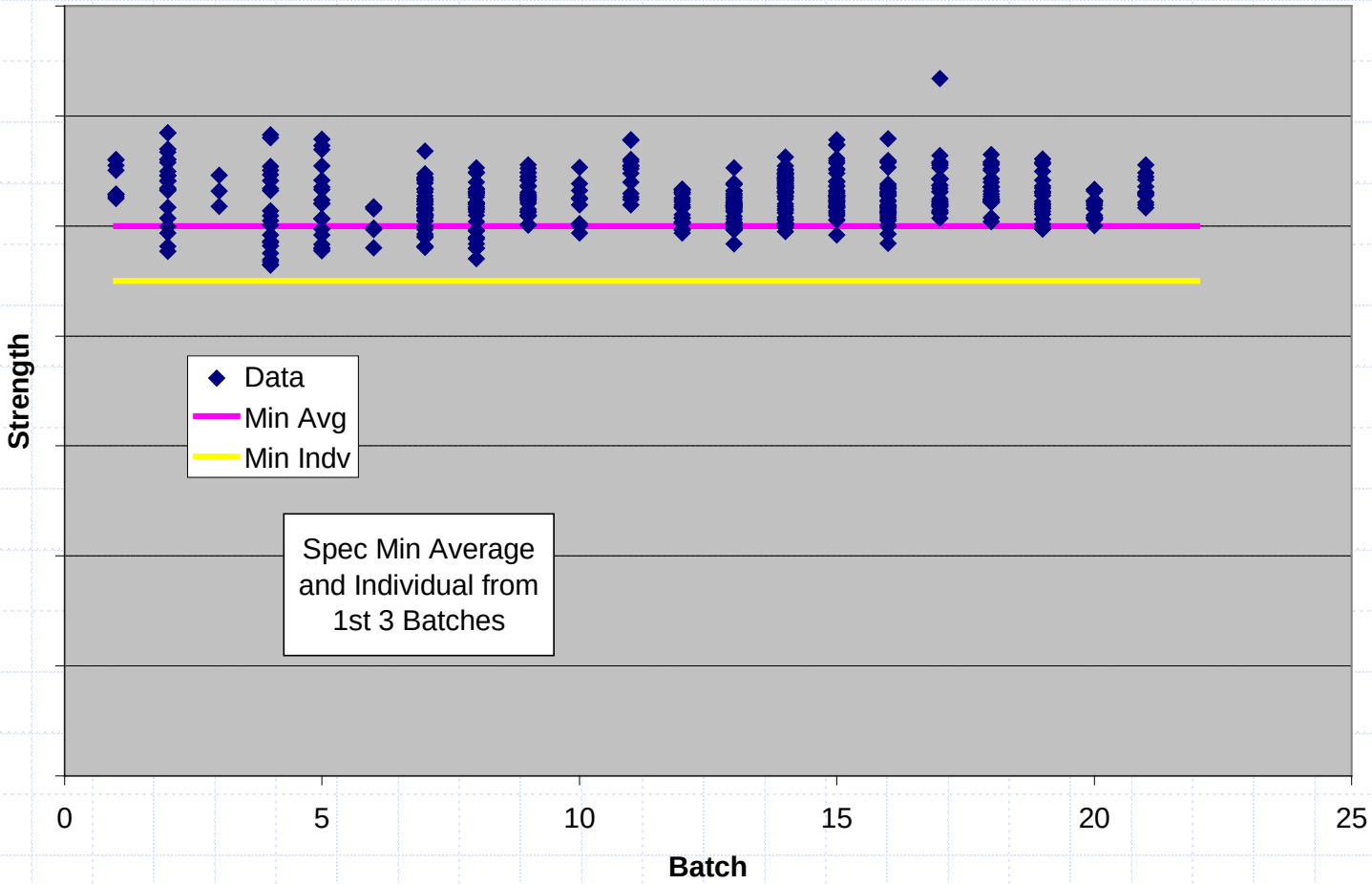
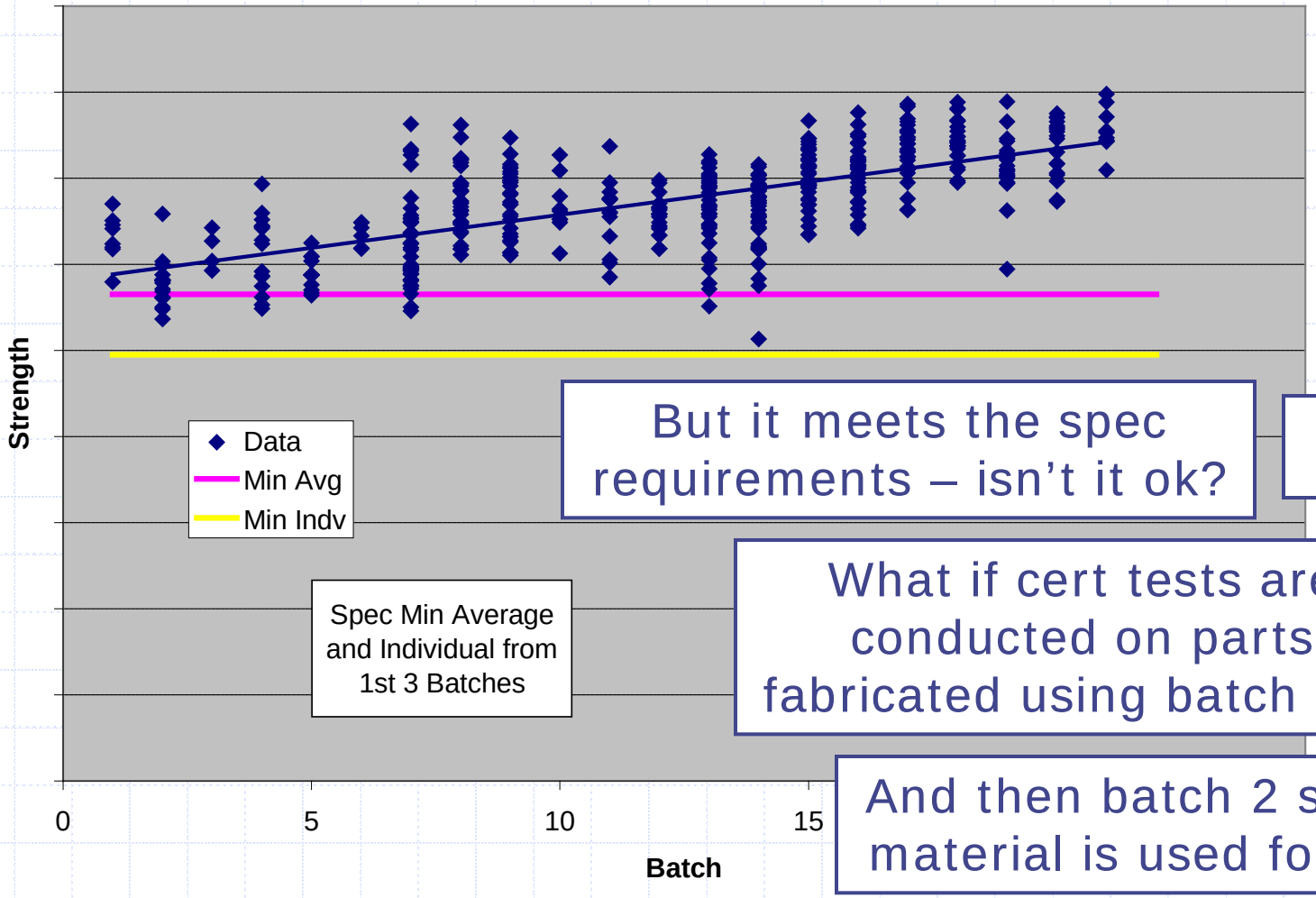
Is this a stable material property?

YES



Is this a stable material property?

NO



But it meets the spec requirements – isn't it ok?

NO

What if cert tests are conducted on parts fabricated using batch 20?

And then batch 2 strength material is used for parts?

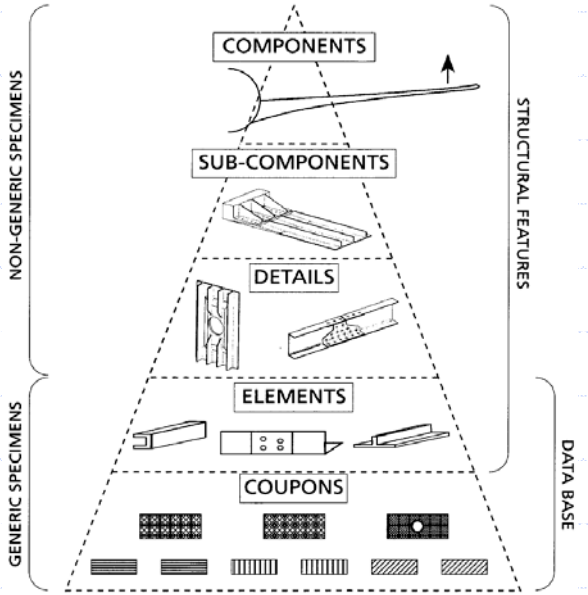
Why are “higher” properties not ok?

- The material is not stable nor in control!
- If a certification test is conducted with a material batch with “higher” properties and subsequently material with “lower” properties is used for production parts,
  → the certification basis for the part is violated
- In some cases, parts are designed to fail at specific load levels (fuses). Converse to the above situation, if “lower” property material is used to design and certify the part, and subsequently “higher” material is used for production parts,
  → the “fuse” will not function as designed

Why are “higher” properties not ok?

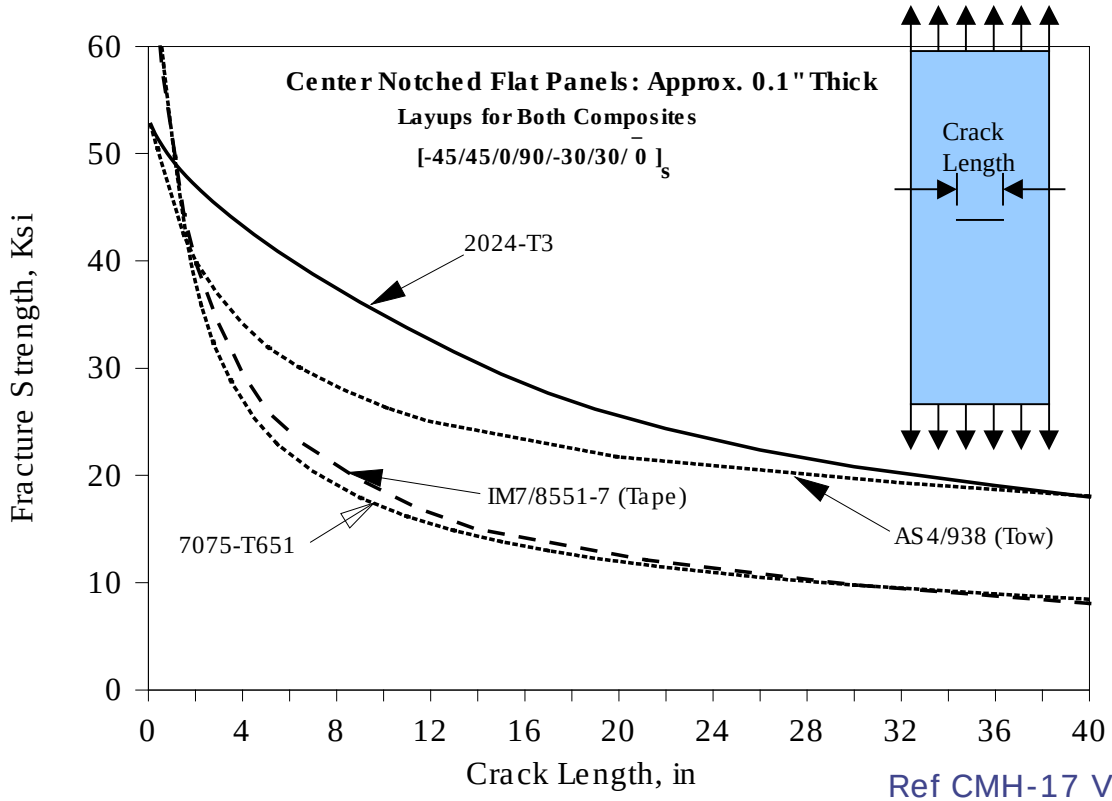
- Typical batch acceptance tests:
  - Uni tension, compression, shear
  - OHC, CAI (sometimes)
- If one of these properties increase, what happens to the rest of the building block data?
  - Do all other properties increase?

NO



Why are “higher” properties not ok?

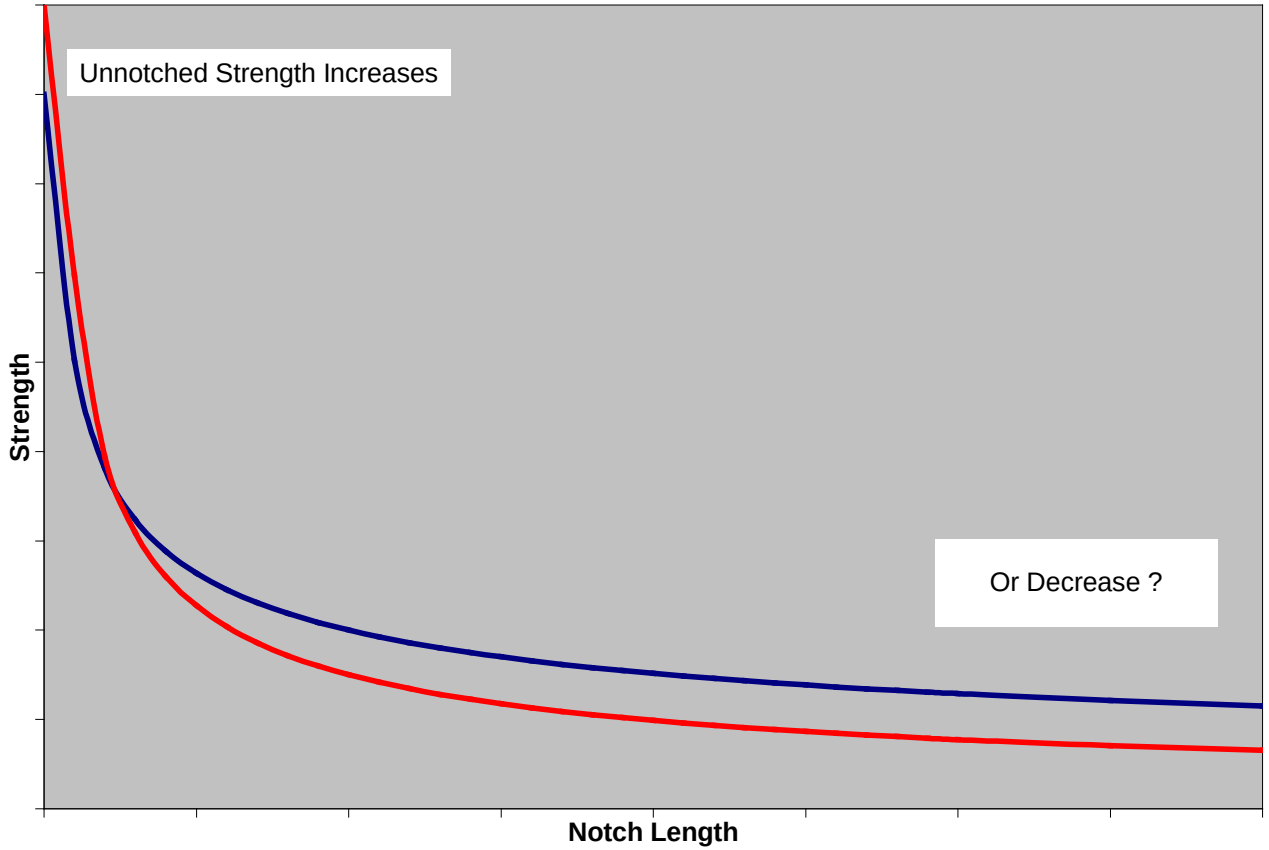
- For example, increasing material tensile strength does not always increase large notch strength



Ref CMH-17 Volume 3, Chapter 12

Why are “higher” properties not ok?

- Unnotched vs small notch vs large notch strength



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- Why do we need stable materials
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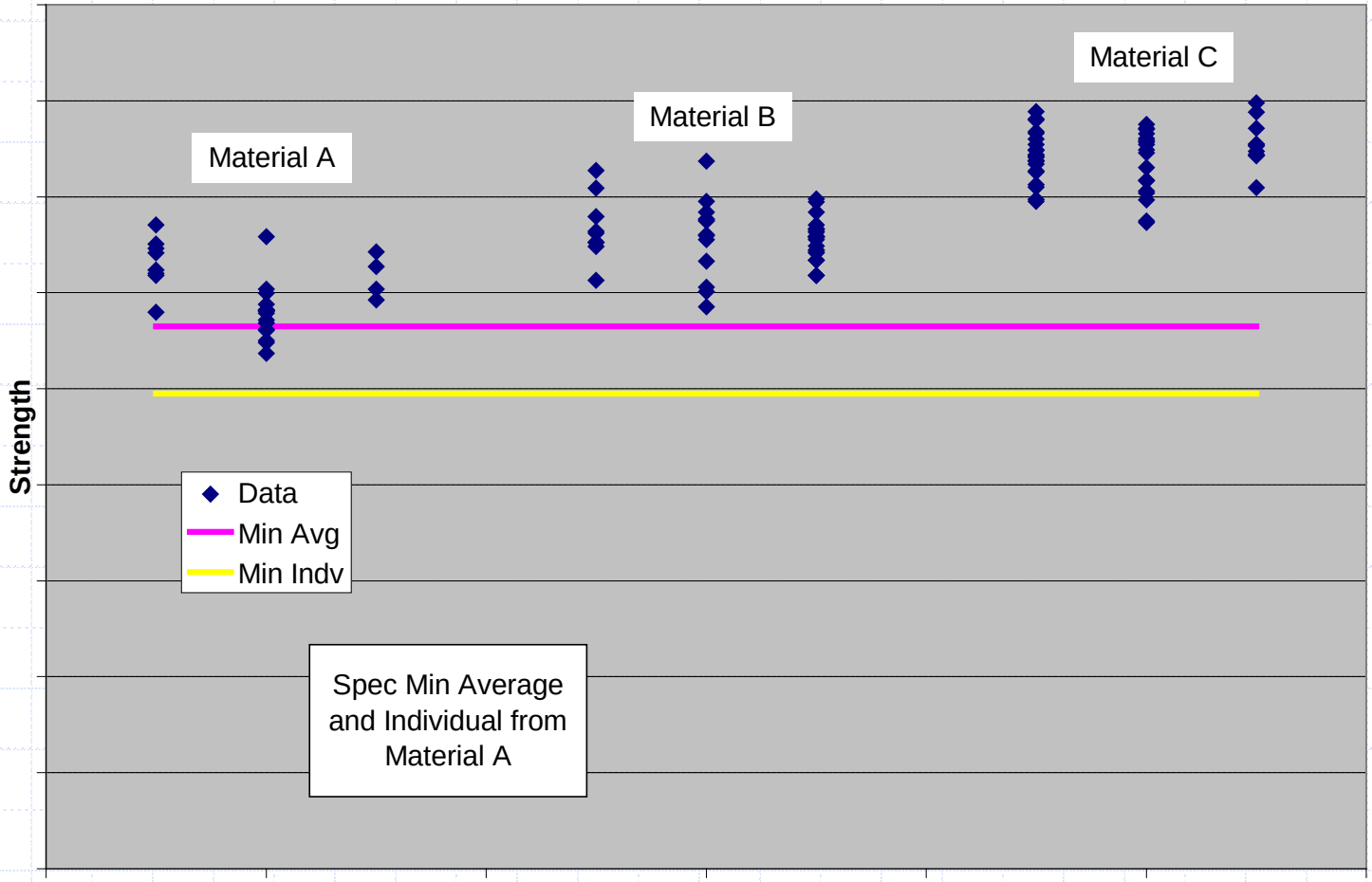
- What is (is not) material qualification
- What is needed before material qualification

Multiple material sources with the same spec designation and requirements – ok?

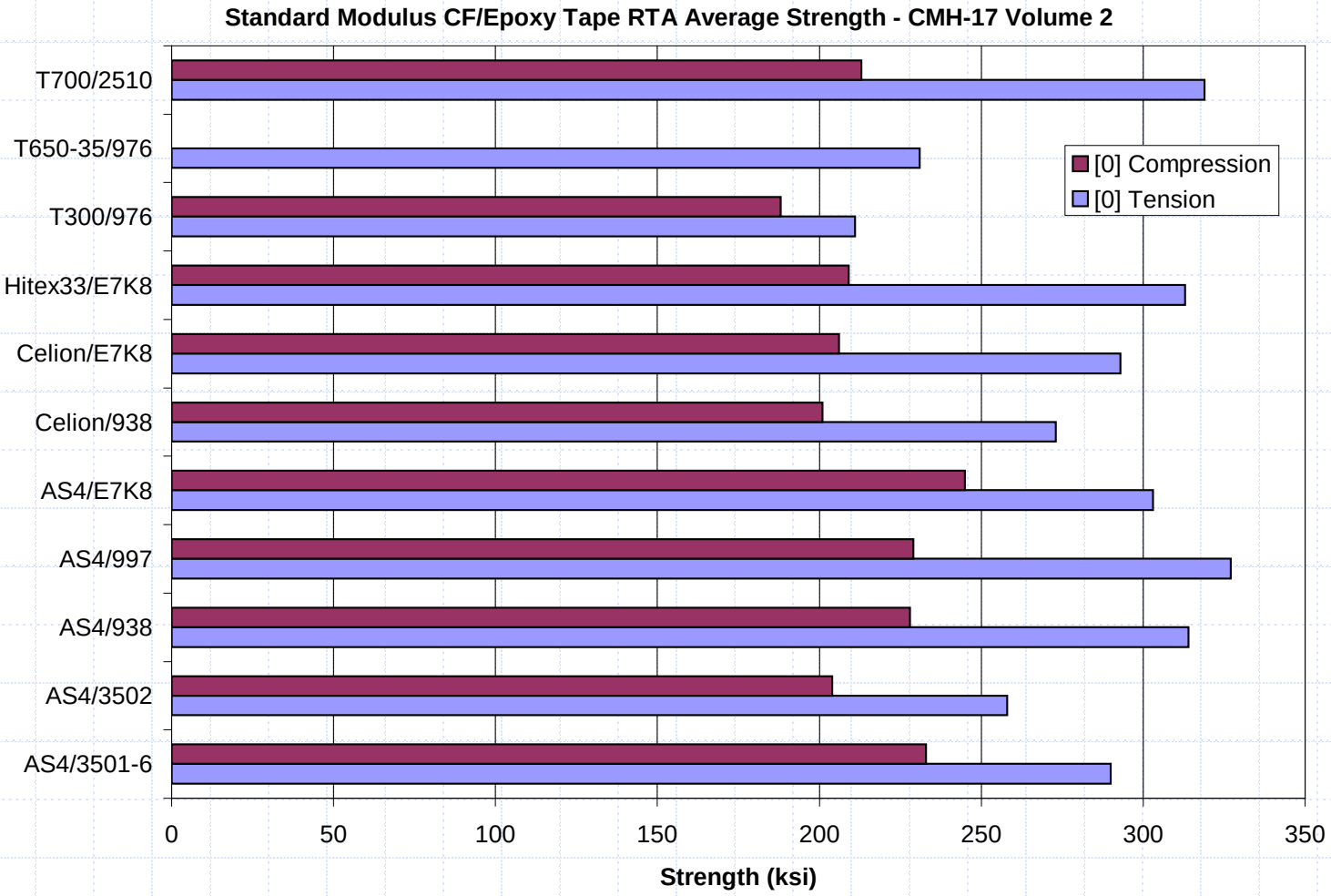
NO



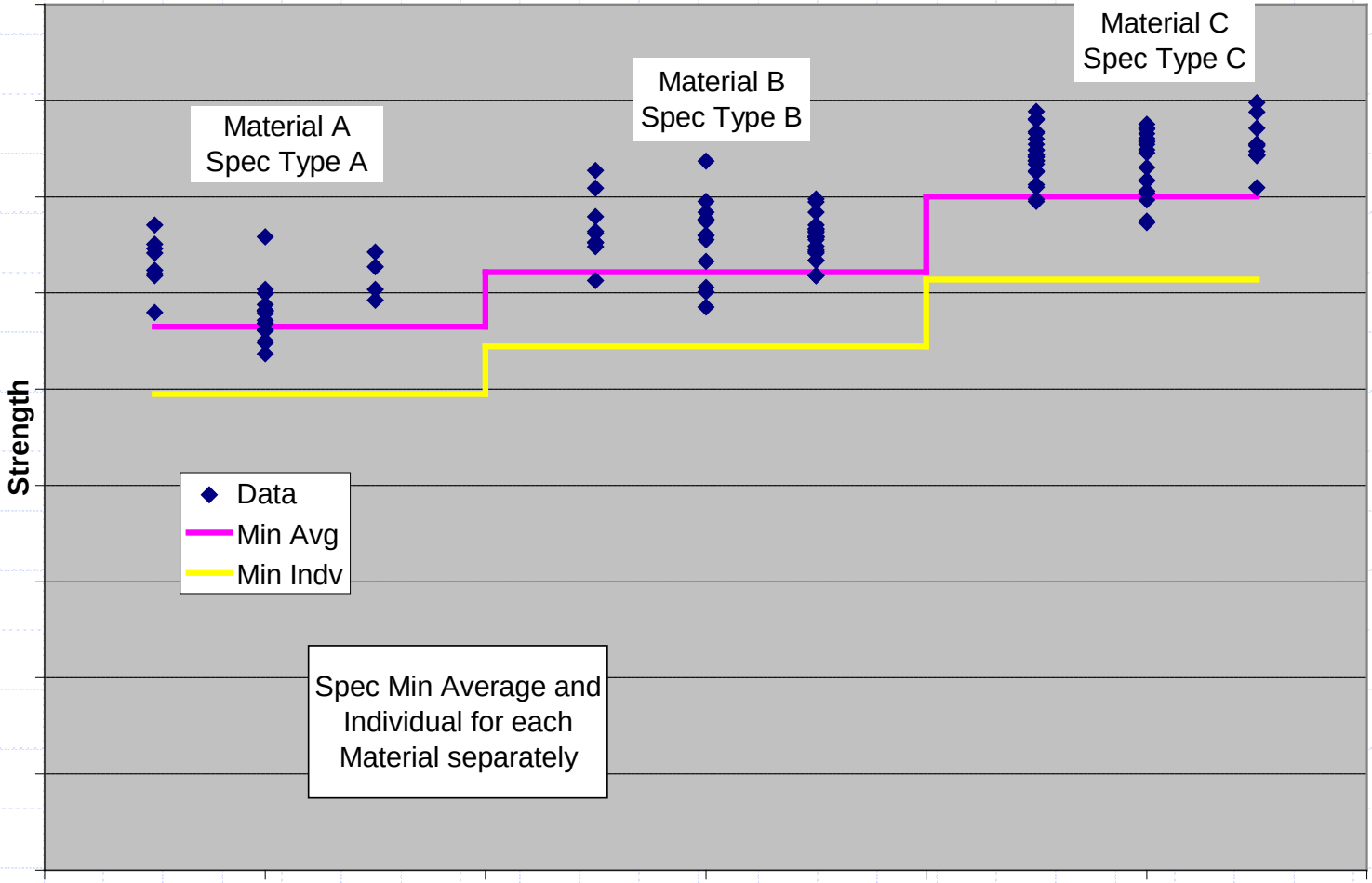
Multiple material sources with the same spec designation and requirements – ok?

- Several problems:
- Material B or C properties can drop significantly and meet the specification, but
  → the material is not stable nor in control!
- If material B or C is used in a certification test article, and subsequently material A is used for production parts,
  → the certification basis for the part is violated
- If material A is used to design a “fuse” and subsequently material B or C is used for production parts,
  → the “fuse” will not function as designed

Multiple material sources with the same spec designation and requirements – ok?



Multiple material sources – should have separate requirements / designations



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- What is (is not) material qualification
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What is “Material Qualification”?

- Qualification for civil airplane applications:
  - FAA does not “certify” materials
  - Obtain data to:
    - Demonstrate meeting desired properties
    - Validate material stability (multi-batch)
    - Set specification requirements
  - Allow purchase of material for “conformed” certification tests
  - Validate use of material with fabrication processes

What is NOT “Material Qualification”?

- Qualification of a material to a specification does NOT mean it is acceptable or certified for a part/structure/aircraft application
- Application certification requires additional testing, analysis and approval
- Military programs often use “qualification” to mean the entire scope of structural certification
  - Causes confusion relative to FAA process

What is NOT “Material Qualification”?

- A process for developing a material
- A process for stabilizing a material

What is needed before Material Qualification

- Draft material spec and requirements
- Anticipated usage environments
  - Temps, moisture, fluids, damage threats, etc
- Defined fabrication processes to use material
- Screening / pre-qual data
- Stable material
  - multiple batches have been produced and tested
- Production scale equipment and settings
  - Fiber line, resin mixing, prepreg line
  - Watch out for line speeds, mix sizes, etc

What is needed before Material Qualification

- Qual test plan
  - Tests, methods, layups, fab/cure process
  - Batch definitions, requirements
- Defined batch certification / acceptance tests

- Think risk reduction
  - Don’t qualify the wrong material
  - Don’t qualify the right material for the wrong process
  - Don’t qualify an unstable material

Controls Needed

- Material Specifications
  - Requirements established to ensure the consistency of the raw materials being purchased to manufacture products
  - Process Control Documents (PCDs)
    - Controlled “recipes” for the production of raw materials
    - Conform to the requirements of material specifications
    - Incorporate SPC
- Process Specifications
  - Documented procedures to ensure control of the manufacturing process used to fabricate parts

Summary

- Control of materials and processes are required to ensure safety
- Material control
  - Does NOT mean only minimum requirements
  - Means stable, repeatable properties
- Qualification is the testing of a stable material to determine material performance and establish requirements for batch certification/acceptance tests and for re-qualification equivalency tests

QUESTIONS ?

Composite Structure Engineering Safety Awareness Course

Module 3:
Qualification Test Programs

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Qualification Test Programs

AGENDA

- Tests
- Fabrication Trials
- Batches
- Liquid Molding
- Adhesives
- Test Plan

Qualification Program

- Specification tests
  - Batch acceptance tests
  - Material characterization
  - Fiber
    - Often qualified at same time as prepreg
  - Prepreg
- Structures tests
  - Key properties
- Fabrication trials

Qualification Program – Resin Tests

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

Ref: DOT/FAA/AR-07/3

Qualification Program – Fiber Tests

Fiber Property	Test Condition	Test Method
Form		ASTM E1309
Twist		TBD
Size Content		ASTM D4018
Tensile Modulus	RTD	ASTM D4018
Tensile Strength	RTD	ASTM D4018
Density	RTD	ASTM D3800

Ref: DOT/FAA/AR-07/3

Qualification Program – Prepreg Tests

- Physical properties
- Chemical properties
- Mechanical tests (fiber, laminates)
- Moisture gain properties
- Storage life, working life
- Cure studies
  - Time / temperature / ramp rate / multiple cures
  - Thickness
  - Autoclave, vacuum
  - Co-cure with core

Qualification Program – Uncured Prepreg

Uncured Prepreg Property	Test Condition	Test Method
Fiber Content, areal weight **FL1**		SACMA SRM 23
Resin Content, % by weight **FL1**		ASTM D3529 SACMA SRM 23
Insoluble Content		ASTM D3529
Volatile Content, % by weight **FL1**		ASTM D3530
Flow, % by weight **FL1**		ASTM D3531 SACMA SRM 22
Gel Time, minutes		ASTM D3532
HPLC (ingredient ratios) **FL1**		SACMA SRM 20
IR (Ingredients Chemical Signature)	RTD	ASTM E1252
Chemical Reactivity and Degree of Advancement via DSC		ASTM E1356 ASTM D3418 SACMA SRM 25
Tack	RTD	TBD
Drape	RTD	TBD

FL1 - Batch acceptance tests

Ref: DOT/FAA/AR-07/3

Qualification Program – Physical Properties

Cured Laminate Physical Property	Test Condition	Test Method
Cured Ply Thickness **FL1**		
Fiber Volume, % by volume		ASTM D3171
Resin Content, % by volume		
Void Content, % by volume		ASTM D2734
Laminate Density	RTD	ASTM D792
Glass Transition Temperature, Tg **FL2**	• Dry • Wet (85% RH)	SACMA SRM 18
Equilibrium Moisture Content	85% Relative Humidity	ASTM D5229
Moisture Absorption	85% Relative Humidity	ASTM D5229
Thermal Induced Microcracking	Cycles over expected range of usage temperatures; fast heat-up spikes, etc.	

FL1 - Batch acceptance test
FL2 - Equivalency baseline database test

Ref: DOT/FAA/AR-07/3

Qualification Program – Lamina Mechanical Tests

Layup FL3	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	70F/ Ambient	Highest Temperature/ Ambient	Highest Temperature/ Wet
[0]n	0 Tension ASTM D3039	Modulus		3 x 2 x 3 **FL2**	1 x 2 x 3	
[0]n **FL3**	0 Compression ASTM D6641	Modulus		3 x 2 x 3 **FL2**	1 x 2 x 3	1 x 2 x 3
[90]n **FL3**	90 Tension ASTM D3039	Ultimate Strength and Modulus		3 x 2 x 3	1 x 2 x 3	3 x 2 x 3
[90]n **FL3**	90 Compression SACMA SRM 1	Ultimate Strength and Modulus s		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 D3039	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 **FL1**	3 x 2 x 3	3 x 2 x 3
[90/0]ns **FL3**	90/0 Compression ASTM D6641	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 **FL2**	3 x 2 x 3 **FL1**	3 x 2 x 3
[+45/-45]ns **FL4**	In-plane Shear ASTM D3518	Ultimate Strength and Modulus	3 x 2 x 3	3 x 2 x 3 **FL2**	3 x 2 x 3	3 x 2 x 3
[0]n **FL5**	Short Beam Shear ASTM D2344	Ultimate Strength		3 x 2 x 3 **FL1**		

Ref: DOT/FAA/AR-07/3

Qualification Program – Laminate Tests

			No. of Batches x No. of Panels x No. of Tests/Batch/Panels			
			Test Temperature/Moisture Condition			
Layup FL3	**Test Type and Direction**	**Property**	**Lowest Temperature/ Ambient**	**70F/ Ambient**	**Highest Temperature/ Ambient**	**Highest Temperature/ Wet**
[45/0/-45/90]ns	Open Hole Tension **FL3** ASTM D5766	Ultimate Strength	3 x 2 x 3	3 x 2 x 3 **FL2**	3 x 2 x 3 **FL2**	3 x 2 x 3
[45/0/-45/90]ns	Open Hole Compression **FL3** ASTM D6484	Ultimate Strength		3 x 2 x 3 **FL2**	3 x 2 x 3 **FL2**	3 x 2 x 3

FL2 - Equivalency baseline database tests
FL3 - Open hole test configuration: 0.25 inch hole diameter, 1.5 inch width
FL3 – Layups should be selected such that laminate thickness is between 0.100 to 0.150 inch.

Ref: DOT/FAA/AR-07/3

Qualification Program – Batch Acceptance Tests

- Must have robust test specimens, methods
- Verify that supplier and purchaser get same values
- Do not use “wet” tests
- Focus on critical properties to “monitor” material
  - Purpose is to detect a change
  - Not to determine the full effect of the change
  - Link to key design properties

Qualification Program – Batch Acceptance Tests

- Recommended in DOT/FAA/AR-07/3 for CFRP tape prepreg
  - Fiber tensile strength and modulus, gm/m
  - Fiber, resin, volatile contents
  - Flow, HPLC
  - Cured thickness
  - RT [0/90] tensile strength and modulus
  - Hot [90/0] compressive strength and modulus
  - RT shear (short beam, CILS)

Qualification Program – Structures tests

- Laminates
  - Hard, quasi, soft
  - Unnotched, open hole strength
- Integrate Qualification and Allowables test programs

Qualification Program - Environments

- Typical for aircraft materials:
  - Cold/ambient
  - Room temp / ambient
  - Hot / ambient
  - Hot / wet
    - Often several ‘hot’ temperatures to cover design range

Qualification Program – Fabrication Trials

- Tack, drape, contour layup
- Slitting process – AFP, CTLM material
- Debulk process
- Sandwich panel core crush, ramps
- Machining, water jet cutting
- Drilling, countersinking
- Painting; paint/finish durability
- Peel ply compatibility
- Co-cured, secondary bond adhesive compatibility
- NDI process, standards

Qualification Test Programs

AGENDA

- Tests
- Fabrication Trials
- Batches
- Liquid Molding
- Adhesives
- Test Plan

Qualification Program – Batches

- Test program to evaluate batch effects, variation
  - Physical, chemical, mechanical properties
  - Fabrication parameters
- Evaluate
  - Fiber batches
  - Resin batches
  - Prepreg batches
    - Roll to roll
    - Within roll – length, width

Qualification Batches – How Many?

- Why test multiple batches
  - Capture / characterize material variations
  - Detect sensitivity of part fabrication process to material variations
  - Avoid “surprises” in:
    - Allowables, structures, certification testing
    - Part fabrication
  - For metals, MMPDS requires 100 data points from 10 heats and lots for A and B-basis values
    - And composites generally have higher variability, so ???

Qualification Batches – How Many?

- Traditional 3 batch qualification for composites and adhesives
  - Driven by cost perceptions, not technical rationale
  - Historical basis from secondary structures
- NOT adequate for primary structure
  - Batches are grouped closely together in time
  - Often are not 3 unique batches
    - Same fiber precursor, resin raw ingredients
    - Same prepreg line setup
    - Many cases only have 2 fiber lots, 2 resin lots
- Pre-Qual / Qual should include 6 – 10 prepreg batches

Qualification Batches

- Pan precursor lots
- Fiber lots
- Resin mixes, w/ separate raw ingredient lots
- Prepreg batches
  - Change setup between runs
- Fabric
  - Separate weaving setups
  - Separate fiber lots for warp and fill yarns

Qualification Batches – Multiple Items

- Multiple fiber lines (same company)
  - 3 lots, one from each line ← NO
  - 3 lots minimum from 1 line
  - 1 lot minimum from each additional line
- Multiple fiber sources (different companies)
  - 3 lots, one from each source ← NO
  - 3 lots minimum from each source

Qualification Batches – Multiple Items

- Multiple tape grades, fabric weaves
  - Treat as separate materials
  - 3 batches minimum per grade or weave
- Multiple prepreg lines (same company)
  - 3 lots, one from each line ← NO
  - 3 lots minimum from 1 line
  - 1 lot minimum from each additional line
- Multiple prepreg sources (licensed)
  - Treat as completely separate materials

Qualification Test Programs

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Qualification – Liquid Molding

- RTM, VARTM, RFI, etc
- Unique problems relative to prepreg
- Material specifications and qualification only involves separate fiber/fabric and resin
- Part fabricator must
  - Have internal “material specification” for combined materials
  - Perform “qualification” using multiple batches of fiber and resin
  - Perform batch tests on cured laminates
    - Batch panels, witness panels, prolongs

Qualification – Adhesives

- Two parts, both should be included in specification
- “Basic” qualification using metal adherends
  - Lap shear, thick adherend shear, peel, flatwise
  - Durability tests (wedge, etc)
  - Characterize adhesive failure modes
  - Use for batch acceptance tests
- “Application” qualification
  - Representative part adherends and fab process
    - Surface preparation, cure, etc
    - Adherend material, ply orientation, core type
  - Stress, fracture toughness type tests

Qualification – Adhesives

- Qualify each grade (thickness) separately
- Cure study
  - Similar to prepreg qualification
  - Include high temperatures, long cures, multiple cures
    - Adhesives often more sensitive
  - Include repair conditions if applicable
- Environments

Qualification Test Programs

AGENDA

- Tests
- Fabrication Trials
- Batches
- Liquid Molding
- Adhesives
- Test Plan

Qualification Program

- If only material qualification, do not need FAA conformity, witnessing
- If data is to be used for allowables,
  - FAA approved test plan and conformity
  - But are supposed to have a specification and qualified material before allowables testing
  - For joint qualification/allowables test program, need a released draft specification
  - Supplier must provide batch certification test data
  - Purchaser to review data before qual/allowables tests
- Need to define required material production witnessing (fiber, resin, prepreg), PCD audits, etc
- Need to define specimen layup, cure, machining, test witnessing

Qualification Test Plan

- Responsible organizations
- Witnessing, auditing plan
- Specific materials, suppliers, sites, PCDs
- Batches defined
- Test matrix
- Test specimens
- Layup sequences
- Layup procedures (including storage life, out-times before layup)
- Cure procedures (including resin injection, etc)
- Specimen machining
- Test methods/procedures (see CMH-17 for guidance)
- Specimen preconditioning (moisture, fluids, etc)

Qualification Test Plan

- Documentation / data reporting
  - Batch records
  - Batch certification data
  - Layup records / cure records
  - Test specimen conformity (AER)
  - Test witnessing (AER / delegate)
  - Data tables
  - Load-displacement, load-strain curves

Summary

- Before qualification
  - Understand and document all requirements
  - Have a stable, production ready material
- Qualification program
  - Material from production scale equipment
  - Adequate number of batches
  - Well defined test specimens and procedures
  - Integrate with allowables program
  - Include fabrication assessments
  - Complete before production part design

QUESTIONS ?