

Composite Structure Engineering Safety Awareness Course

Information Contained in Material Specifications

Composite Materials Specifications

AGENDA

- ◆ What are specifications?
- ◆ Prepreg example
- ◆ Sources of data and limits
- ◆ Supplier/purchaser responsibilities
- ◆ Other examples
- ◆ Case studies

Specifications

“ a detailed description of the criteria for the constituents, construction, appearance, performance of a material, apparatus etc, or of the standard of workmanship required in its manufacture”

- ◆ Historically, large airframers developed their own material specs, such as for prepreg (fiber and uncured resin), and suppliers qualified their product. Alternatively, composite part fabricators can now use industry specs created for a supplier material.
- ◆ Material specifications for composite materials flow vertically and horizontally.
- ◆ Material specifications are essential in protecting the cured composite mechanical properties used for designing the part. It is the responsibility of the part manufacturer to ensure form fit and function by controlling materials which are critical for composites because the part and the material are made at once.

Specifications

- ◆ Material allowables and specification limits are linked, this will be covered in a later session.
 - One is a quality control tool with testing to fewer properties and environments and may change over time.
 - It is used to see if a new batch, say of prepreg, is in the same population as the qualification batches.
 - The other is used for design and is fixed.
- ◆ The OEM, prepreg supplier, part manufacturer and the FAA all have roles to play, these inter-relationships too will be covered.
- ◆ I stole all the above from John Adelman's presentation.

Prepreg Specifications

- ◆ This is one you will run into at any part fabricator and it is a good example of the level of detail required to adequately control the material.
- ◆ Not only the materials must be defined: the process equipment must be qualified and unqualified equipment must not be used.
- ◆ The resin reactor and the mix process must have been qualified to make resin to the resin spec and its configuration must be defined and controlled.
 - Maintenance is ok, but parts that perform in an identical manner must be used.
 - Any changes to configuration and process must be validated and approved.
- ◆ Only an approved filmer may be used.

Prepreg Specifications-Continued

- ◆ Only qualified prepreg line(s) may be used to combine resin and fiber using the same controls as for resin.
- ◆ Only qualified fiber lines may be used to make fiber.
- ◆ Changes and modifications must go through a notification process and a qualification or a requalification process.
 - The nature and degree of proving equivalency will depend on the nature and degree of change.
 - The bigger the change, the more testing will be required to prove equivalency.
- ◆ What you will typically see is a property , a method to determine it (standard methods like ASTM are preferred), and +/- limits for acceptance. I don't like only a min or a max limit.

Scope

- ◆ General description of the product, its area of application and use limits.
- ◆ 350F curing plain weave fabric epoxy prepreg for use up to 180F.
- ◆ Defines form (slit tape, tape), type (RC), grade (FAW), class (tow count, size).

References or Applicable Documents

- ◆ Test methods i.e. ASTM, SACMA or vendor specific.
- ◆ FAA documents such as DOT/FAA AR-02/109 (2003), DOT/FAA AR-02/110 (2003) and DOT/FAA AR-06/10 (2007).
- ◆ Other relevant documents such as drawings and work instructions.

Definitions

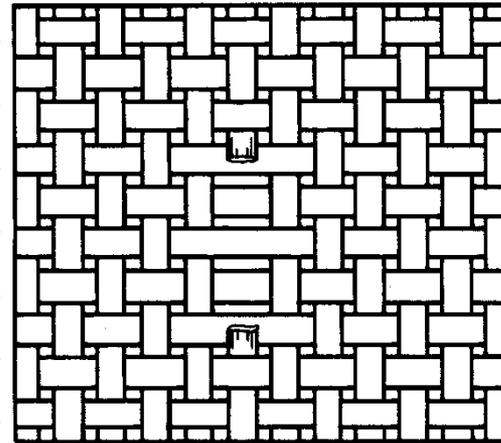
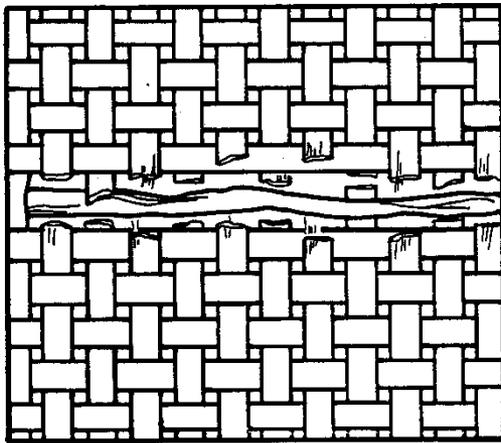
- ◆ Hard to get too specific
- ◆ Bagside, foreign material, roll, storage life, lot.
- ◆ Industry standards are also preferred here.
- ◆ I think of this as a contract, where the parties agree on what will be supplied.

Material Requirements

- ◆ This section calls out the constituent materials and their designations.
- ◆ For resin the actual formulation is usually supplier proprietary and takes a proprietary information agreement to access.
- ◆ For fiber in an industry spec the prepreg supplier will have a fiber spec, some airframers will have their own fiber spec.
- ◆ Ditto for the fabric spec.

Visual defect criteria and dimensional requirements

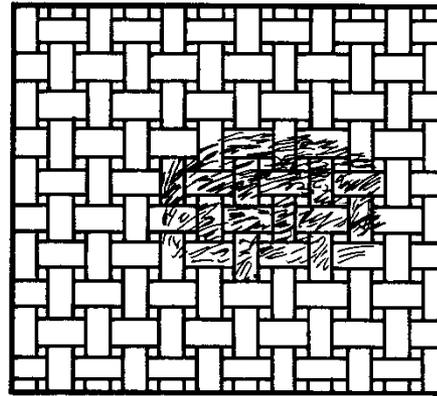
- ◆ No cut or broken fiber.



- ◆ Yarn or tow splices
- ◆ No dry fibers, no resin rich area.
- ◆ Some paper flecks (slitter litter).

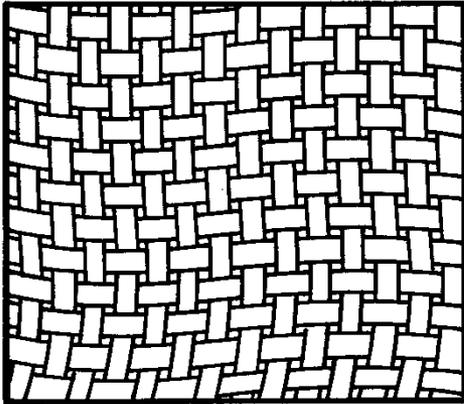
Visual defect criteria and dimensional requirements

- ◆ Fuzz balls are less than an inch and don't distort fiber.
- ◆ Fuzz balls are less than 3 in² in 10 ft².

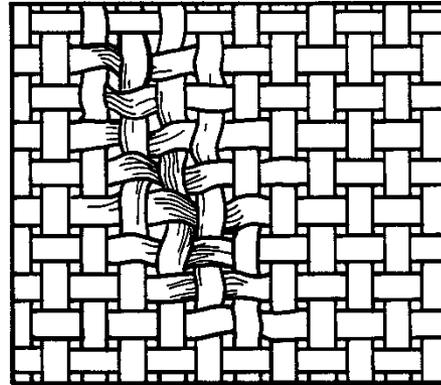


- ◆ Roll width shall be 38 +/- 1 inches.
- ◆ Roll length shall be 100 +/- 5 feet

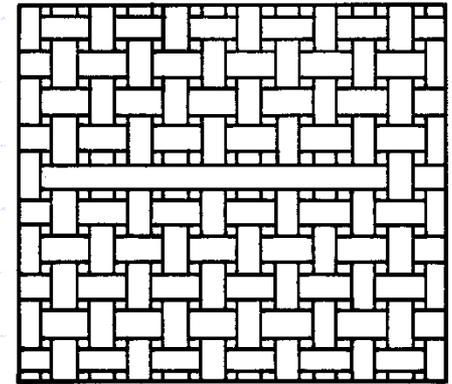
Visual defect criteria and dimensional requirements



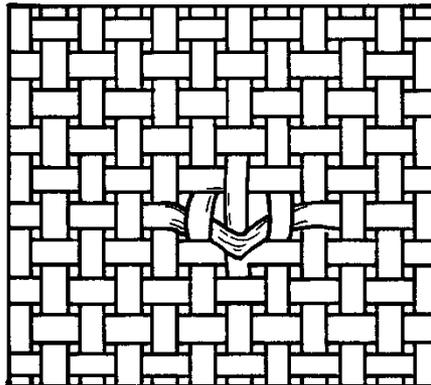
Waviness



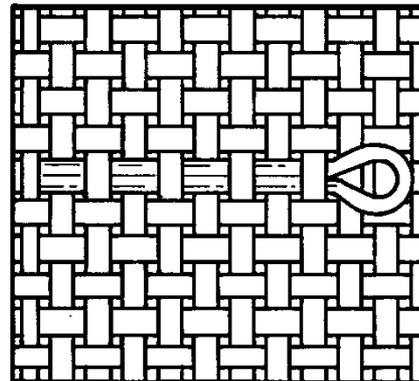
Crease or wrinkle mark



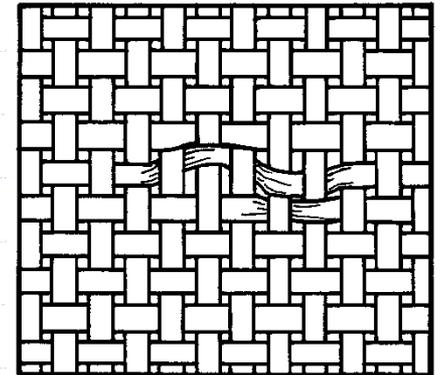
Float



Hang pick

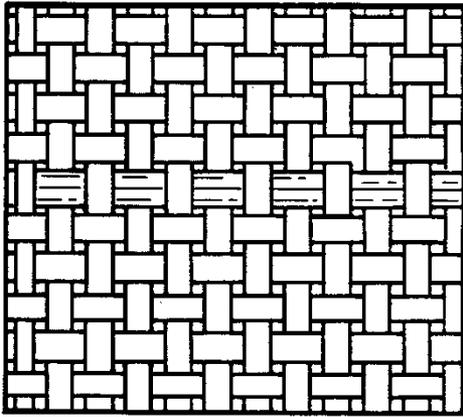


Kink

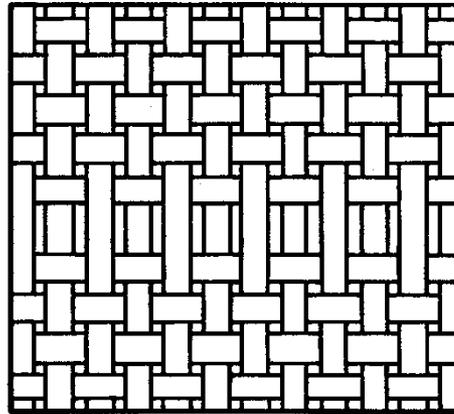


Loose pick

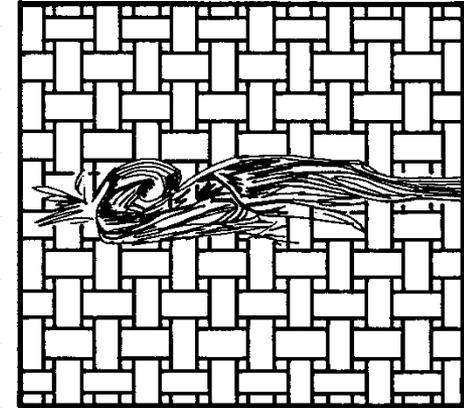
Visual defect criteria and dimensional requirements



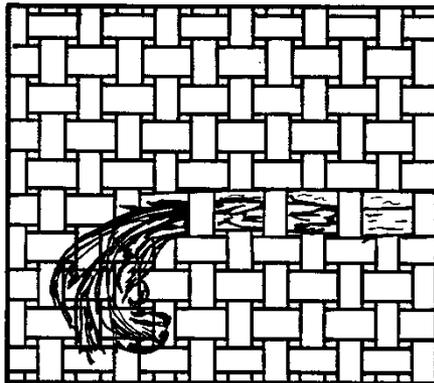
Mispick



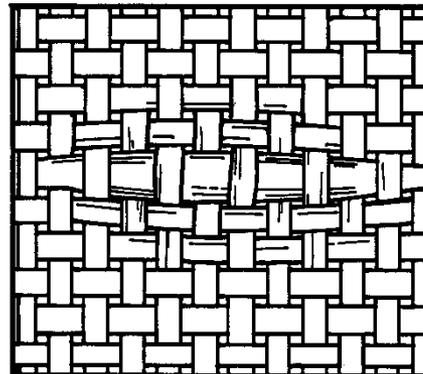
Missing pick



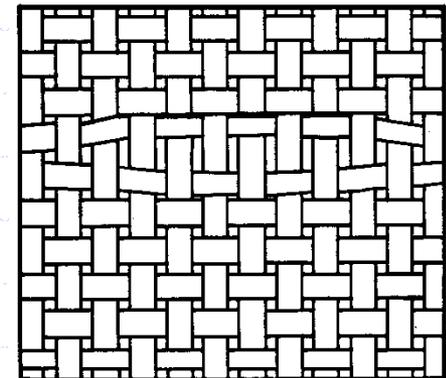
Nep



Pulled in filling

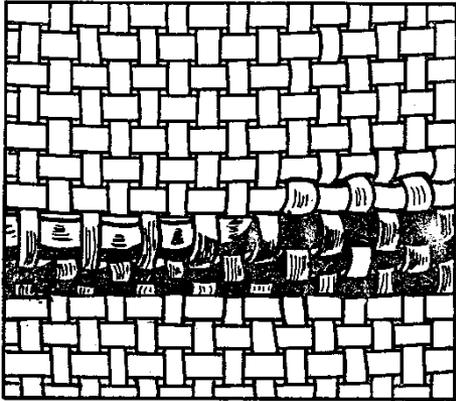


Slub

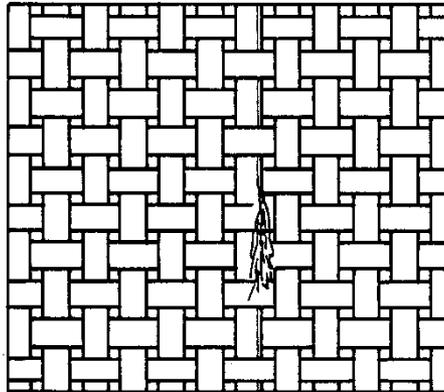


Weave separation

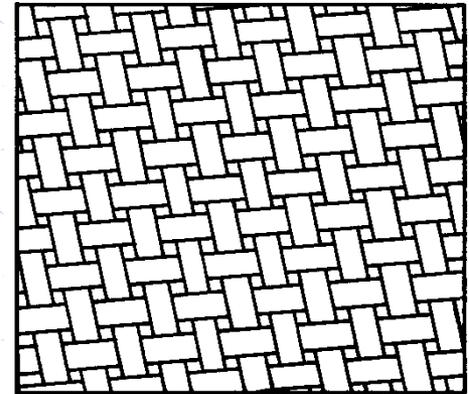
Visual defect criteria and dimensional requirements



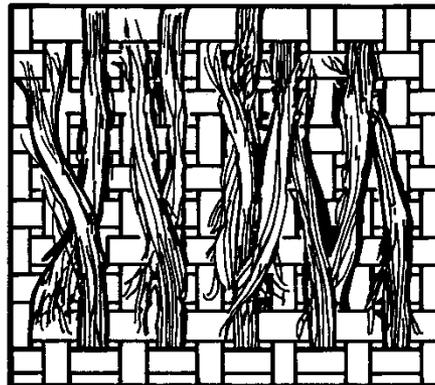
Baggy or wavy cloth



Broken tracer yarn



Skew



Smash

Non-conformance

- ◆ Fuzz ball can be removed with a spatula.
- ◆ Defects must be marked with a marker visible on the outside of the roll-usually a paper tag.
- ◆ Single point defects use a single marker.
- ◆ Linear defect mark start, end and every 2 feet along the defect.
- ◆ Prepreg may be cut or spliced to remove defects according to XXX criteria.
- ◆ The type, location and length of each defect shall be indicated on a defect log accompanying each roll.
- ◆ Defects are not counted towards amount purchased.

Prepreg life

- ◆ Thermoset resins change over time, faster at higher temperatures, slower at lower temperatures.
- ◆ Epoxy prepregs are typically kept frozen at all times until use, including shipping.
- ◆ This includes distributors.
- ◆ Storage time at a temp such as 12 months at 10F or lower after DOM.
- ◆ Tack life for layup and how long a laid up part can sit on the shop floor before cure.
- ◆ Define shop floor conditions such as temperature limits, humidity limits, dust concentration.
- ◆ These conditions must be documented.

Uncured physical and chemical properties

- ◆ Resin content percent by weight, 42.0 ± 3.0
- ◆ Fiber areal weight g/m^2 193 ± 8 .
- ◆ Chemical composition and attributes measured by IR, HPLC and DSC.
- ◆ For example for the HPLC analysis P1 % range, P2 % range etc. To keep formulation proprietary the peaks are not identified.
- ◆ An IR trace and a % correlation can be used as a fingerprint identifying the resin.

Cured physical properties

- ◆ Density in g/cc.
- ◆ Cured ply thickness in mils (related to FAW and RC) important for part fit up as well as normalizing data.
- ◆ Cured glass transition temperature range, a check if the material will cure properly.

Cured laminate mechanical properties

- ◆ Measured under various environmental conditions such as cold dry, room temperature dry, elevated temperature wet and dry where wet means humidity ageing to equilibrium moisture uptake.
- ◆ Since a higher % fiber will give higher results data may or may not be normalized.
- ◆ Some specs assume if FAW and resin content are controlled then there is no normalizing, panel thickness is based on nominal cured ply thickness (CPT) and number of plies.
- ◆ Some specs normalize the data according to the cure ply thickness of nominal FAW and resin content.
Normalized = Actual value X actual PPT/nominal PPT
- ◆ The specification may call out a minimum average and a minimum individual value for a given test.

Cured laminate mechanical properties, continued

- ◆ RT tension is to monitor the fiber and fiber interface properties.
- ◆ Hot wet compression monitors cured resin capabilities.
- ◆ Short beam shear tests the fiber-resin interface.
- ◆ Resistance to impact may be a critical attribute.
- ◆ Properties with holes, both unfilled and filled with a fastener are critical to bolted assembly.
- ◆ Solid laminate and sandwich panels may be included.
- ◆ Resistance to fluids found in the intended application- short term and long term.
- ◆ Fatigue testing of undamaged and damaged specimens.
- ◆ Hard and soft laminates.
- ◆ Some sub set will actually be used for incoming inspection.

Supplier Quality Assurance

- ◆ It is expected that statistical process control based on key characteristics (KC) and key process parameters (KPP) will be established and maintained.
- ◆ The responsibility for selecting and documenting is designated such as Quality or Technical.
- ◆ The responsibility for conducting analysis to determine control limits is designated.
- ◆ The responsibility for determining if the process is under control is designated as well as corrective action.
- ◆ With the approval of the customer and the FAA reduced testing can be implemented based on capability.

Product Certification

- ◆ The supplier will conduct those tests called out by the specification and prepare a certification report for each prepreg batch.
- ◆ Each roll will be tested for resin content and FAW.
- ◆ HPLC and FTIR will be taken on a random sample once per batch.
- ◆ Selected laminate mechanical properties are to be taken once a batch.
- ◆ Records will be kept for a period of time typically 10 years and will be available for inspection by the FAA or customers.
- ◆ The certification report warrants that the material has met the criteria of the specification.
- ◆ The report may include individual and averaged values with requirements, batch and roll identification, defect log (also one attached to each roll).
- ◆ Material that fails any of the requirements shall be rejected subject to retest conditions.

Purchaser Quality Control

- ◆ The purchaser QA will conduct those tests called out by the specification as incoming acceptance.
- ◆ Purchaser QA will review the supplier certification as well as conduct any other inspections.
- ◆ Purchaser QA must verify that the material met the storage conditions during shipment.
- ◆ Material that fails any of the requirements shall be rejected subject to retest conditions.
- ◆ Records will be kept for a period of time typically less than the supplier.
- ◆ Material will then be released to production.

Material test methods

- ◆ I like to see the specific methods spelled out in the spec, it makes for a bulky document but makes for easier reading in that you don't have to flip back from document to document.
- ◆ Describes each test in detail as well as how to report the data.
- ◆ These include details such as how to layup and cure the various panels for laminate testing.
- ◆ Process aids such as bagging material, vacuum sealant, breathers, caul plates must be defined in process spec, material spec or process instruction.
- ◆ The cure cycle for test laminates must be the same as used for part manufacture or equivalency shown between the cures.
- ◆ Life is not perfect, there must be provisions made for retesting if the materials fails such as known upsets during testing, panel fabrication, machining or is a statistical outlier. By the way all data must be reported as well as a description of how abnormalities were resolved.

Material identification

- ◆ Clearly and unambiguously assigns batch number, roll number, name of material, specification designation, length and width.
- ◆ Usually put inside of core, on the outside of the shipping bag and outside of shipping box.

Packaging, marking and shipping

- ◆ Release film or paper physical and chemical requirements, these are engineered products!
- ◆ Roll size.
- ◆ Support core details.
- ◆ Clean defect free plastic bag, no holes, cuts, tears.
- ◆ Additional packaging such as a box.
- ◆ Markings must be permanent and legible.
- ◆ Ship with temperature recorder to demonstrate storage requirements are met.

Material Safety Data Sheet MSDS

- ◆ To comply with hazardous communication regulations, a copy of the MSDS must accompany the shipment.
- ◆ A copy must also be available to the workforce.
- ◆ Community right-to-know laws may also require that a copy of the MSDS be kept even after the material is not used.

Qualified supplier list

- ◆ An appendix to the specification that lists the suppliers that have been qualified to supply material to the it.
- ◆ It typically lists the product designation, name, address, location and contact information.

Revision log

- ◆ Tracks changes to the specification.
 - ◆ When the change occurred.
 - ◆ What was the change.
 - ◆ Sign off by both supplier purchaser.
 - ◆ Designation of new revision.
- ◆ End of prepreg details!!!

Sources of data supporting requirements and specification limits

- ◆ The nominal values and spec limits for a prepreg specification are initially derived from the original qualification.
- ◆ The statistical evaluation is conservative and spec limits may be broad.
- ◆ Over time, data is added and spec limits could be tightened.
- ◆ Spec limits are only broadened after it can be shown that the design is unaffected.

Sources of data supporting requirements and specification limits

- ◆ Material at the limits of a specification requirement should be evaluated prior to qualification to determine effects on a higher level to validate limits.
- ◆ Resin ingredients at their limits should be combined into resin which is evaluated.
- ◆ Fiber batches at the spec limits should be made into laminates and tested.
- ◆ Studies should be conducted at the proposed tolerances and scale accuracies.

Sources of data supporting requirements and specification limits

- ◆ Proposed process conditions and limits should also be explored prior to qualification.
- ◆ Time and temperature limits to resin mixing
- ◆ Time, temperature, roll gap and speed for filming.
- ◆ Time, temperature, speed, compaction pressure, and fiber tension for prepregging.
- ◆ Cumulative extremes need to be explored.
- ◆ Upset conditions and corrective action must be defined, i.e. how long resin can stay heated in the fountain bar.

Supplier responsibilities

- ◆ Must have the capability to conduct raw material and final product testing.
- ◆ Must have the capability for record maintenance, calibrations and SPC.
- ◆ The workforce must be trained to perform specific jobs and this should be documented in a training record for each employee.
- ◆ Personnel must be capable of conducting testing and running equipment.
- ◆ Only qualified equipment shall be used to make a product.
- ◆ Major equipment maintenance and modification records should be available.
- ◆ Continuously test to populate database on an ongoing basis to ensure material has not changed.
- ◆ An organizational structure that ensures operations and quality can perform their functions.
- ◆ Certify outgoing materials to specifications.
- ◆ Maintain environmental controls to specified levels.

Purchaser responsibilities

- ◆ Must have the capability to conduct raw material and final product testing.
- ◆ Must have the capability for record maintenance, calibrations and SPC.
- ◆ The workforce must be trained to perform specific jobs and this should be documented in a training record for each employee.
- ◆ Personnel must be capable of conducting testing and running equipment.
- ◆ Only qualified equipment shall be used to make a product.
- ◆ Major equipment maintenance and modification records should be available.
- ◆ An organizational structure that ensures operations and quality can perform their functions.
- ◆ Maintain environmental controls to specified levels.

Specifications for other materials

- ◆ The complexity and level of detail must be adequate for the material.
- ◆ The degree of qualification depends on the criticality of the use.
- ◆ Acetone is a common cleaning solvent and several industry standard specs exist including ASTM D329.
 - The purchaser can reference this spec and require that incoming material show conformance.
 - No further testing by the purchaser is needed.
 - No qualification is needed as the use is non critical.
 - Any number of suppliers can be used.
- ◆ Bis phenol A epoxy resin is used as an ingredient in resin formulations.
 - There are many suppliers of nearly equivalent materials.
 - Since the resin is critical to cured mechanical properties receiving inspection is required, some reduced set of specification requirements.
 - Additional suppliers can be qualified through higher level testing including mixed resin testing as well as cured laminate property evaluations.
 - Material from the desired suppliers could be included in the original qualification.

Specifications for adhesives- structural paste for sandwich repair

- ◆ Will look much like the prepreg one in terms of content.
- ◆ Will call out mechanical properties that are pertinent to the use and application.
- ◆ Bonding aluminum alloy facings to nonmetallic core, core to core for repair, panel inserts and edge attachments.
 - Form bonds that withstand temperature range and humidity
 - Withstand combinations of stress, temperature, humidity and time.
- ◆ Working characteristics such as shelf life, curing, mix ratio adhesive life.
- ◆ Mechanical properties such as tensile lap shear, flatwise tension, climbing drum peel and wedge stress test for durability.
- ◆ Test methods will include sections on substrate preparation such as nature of metal surface cleaning and treatment and cutting and machining of core samples.

Specifications for non-metallic honeycomb core

- ◆ Physical properties may include material of construction, cell configuration, cell size, cell wall thickness and density.
- ◆ It may be a material but there are environmental, environmental chemical and flammability requirements.
- ◆ Mechanical properties and methods for their evaluation: compressive, impact, shear, fatigue and flatwise tension at the limits of operating range.
- ◆ The mechanical properties are conducted on a structure that includes face sheets and adhesives so they must be specified.
 - Aluminum or composite laminates as face sheets.
 - Paste or film adhesives.
- ◆ Machinability and formability may be critical to part manufacture.
- ◆ Other key properties may be thermal conductivity, insulation, heat transfer, electrical conductivity.
- ◆ It may be received by the plant pre-machined so these dimensions must be specified.

Case Study- Powderized Curative

- ◆ A ground aromatic amine curative was qualified as an ingredient for an epoxy resin formulation.
- ◆ After several years successful resin and prepreg production, certified curative meeting receiving inspection could not be mixed into the resin.
- ◆ The curative would roll and form hard lumps during mixing and would not disperse.
- ◆ After months of plant trips and testing a paperwork audit showed that to get passing particle size with less oversize the residence time in the grinder was increased which resulted in electrostatically charging the material making it clump.
 - This PCD change was not determined to be critical, no notification
- ◆ Return to original process time limit eliminated the problem.
- ◆ Not all critical attributes can be captured in a specification and must be controlled through the PCD.
- ◆ Detailed record keeping is critical to prevent problems or help solve them.

Case Study- Resite Failure

- ◆ A prepreg supplier was consolidating production and loading up the process equipment in the remaining plant.
- ◆ During the qual all looked good except the prepreg from the new machine failed tensile modulus requirements at cold dry conditions.
- ◆ RT tensile modulus had passed cert testing for years.
- ◆ A review of records showed that at the original plant modulus was determined by hand, picking a line segment that met the requirement which did not specifically detail how modulus was to be determined.
- ◆ Reanalysis of old qualification data per modern standards resulted in a shift of modulus downward.
- ◆ Re-evaluation of design criteria showed an acceptable margin of safety and many years of flight history validated the safety of the material.
- ◆ So the specification was lowered using a better defined modern standard method.
- ◆ Detailed record keeping is critical to prevent problems or help solve them.