Material Specifications For Fabric Prepregs

Comments on Unofficial Draft Recommendations – August 22nd 2003.
Presented at FAA Workshop, Chicago, September 16th, 17th, 18th 2003
ACG Activities in the Area of Specifications, Shared Databases and Material Control

- Participated in 2002 workshop which resulted in the publication of DOT/FAA/AR-02/109 and DOT/FAA/AR-02/110 documents.
- Participation in AMS P17 subcommittee responsible for creating industry specifications for composites.
- Decision made to apply the proposed methodology to an Air Force funded program on oven vacuum bag cured (OVBC) prepregs, which involves a substantial data generation program on a resin system called MTM45 with various product forms. MTM45 is a 175°F to 250°F curing resin with 250°F or higher service capability.
- The US Air Force is fully supportive of making the data public domain and following the guidelines of this workshop and other industry bodies.
- The intention is to share the data with multiple end users and to eventually submit as a candidate for inclusion in national specifications being developed under auspices of AMS P17 subcommittee.
MTM45 Data Generation Plan
Material Supplier Generated Data to be Made
Available to Multiple End Users

- Multiple product forms
  - 6K 5 harness satin AS4C fabric prepreg
  - AS4C unidirectional prepreg
  - 7781 style glass fabric prepreg
  - 4581 style quartz fabric prepreg
  - 3K PW AS4C carbon fabric
  - IM7C unidirectional prepreg
  - 1K plain weave carbon prepreg
  - Style 4503 quartz fabric prepreg

- DOT/FAA/AR-02/109 document used as a model for Test plan, modified to accommodate fabric prepregs. Fabric prepreg recommendations had not been published at the time the plan was written, however the test plan is reasonably close to the latest document.
  - Test plan includes both lamina level and laminate level data on Q/L, “soft” and “hard” laminates, plus bearing data, compression after impact data and interlaminar tension data.
  - Draft material and process specifications produced in accordance with the 109 and 110 document guidelines

- Input from industry partners encouraged to achieve consensus and buy-in.
  - Regular interactive progress meetings to be held – next is to be Monday September 30th, 3PM at the SAMPE Technical Conference, Dayton Ohio

- Execution of test plan preceded by extensive process development work involving design of experiments techniques to establish material and process specification and PCD parameters and limits
  - Although rarely referred to as such, a PCD is a process spec. for the manufacture of prepreg.

- FAA special project number requested and received in order to provide a vehicle for FAA acceptance of data. Test plan and specifications to be submitted for FAA approval.

- Conformance, witnessing etc. performed in accordance with FAA policy and recommendations.

- Intention is to submit the allowables data set for publication in Mil handbook 17 and to offer material for inclusion in an AMS spec. when this becomes a reality.

September 12th 2003 Chris Ridgard
Comments on Fabric Prepreg Specification Document – Section 1.5 Recommended Specification Format

- Tiered approach of higher level spec. with lower level specs. for each material form has major advantages.
  - Multiple product forms can be covered by individual slash sheets – can include both fabric and unidirectional materials and even different resin systems if the higher level spec. is written to permit this.
  - Each slash sheet has the acceptance and equivalency requirements derived from the allowables set according the methodology in DOT/FAA/AR-03/19
Comments on Fabric Prepreg Specification Document –

Section 2 Development of Material Controls

- “The investigation (of sensitivities of the material to variations in the tolerances set on the material chemical and physical properties and processing) can be performed in a structured design of experiments that will give the relative sensitivities to the process variables with minimum testing”

- Some material parameters are amenable to this approach, some are not and require collection of data over a number of material batches. DSC acceptance limits, for example, are typically established from data gathered from around 20 batches of material and hence will not be available after the completion of the initial 3 or 5 batch qualification program. Such data will initially be “report only” in any material specification.

- ACG has used the Taguchi design of experiments approach in work on the MTM45 resin system. Process parameters investigated include tolerances on:
  - Resin content
  - Mixing method
  - Degree of impregnation
  - Thermal history
  - All of which are related to parameters in the PCD – the “process specification” for making the prepreg itself.

- It is impossible to investigate most prepreg manufacturing parameters in isolation from the process for making laminates – the two are intertwined and the designed experiment must include both types of variables. This is particularly critical for oven/vacuum bag processed materials.

- “Deliverables” from such work are:
  - Process Control Document
  - Baseline Material Specification (physical properties only at this stage)
  - Process specification for test panel manufacture

September 12th 2003

Chris Ridgard
**Typical Taguchi Designed Experiment for Optimization of Both the Prepreg and the Laminate Manufacturing Processes**

<table>
<thead>
<tr>
<th>Variables (Factors)</th>
<th>Level 1</th>
<th>Level 2</th>
<th>Position in Taguchi L8 Array</th>
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<tbody>
<tr>
<td>Resin Content</td>
<td>A</td>
<td>B</td>
<td>Column 1</td>
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<tr>
<td>Vacuum Mixing</td>
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<td>Yes</td>
<td>Column 2</td>
</tr>
<tr>
<td>Impregnation</td>
<td>Sided</td>
<td>Impregnated</td>
<td>Column 3</td>
</tr>
<tr>
<td>Out Life</td>
<td>Fresh</td>
<td>10 days</td>
<td>Column 4</td>
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<tr>
<td><strong>Bagging</strong></td>
<td>Non perforated FEP with</td>
<td>P3 with glass bleeder/breather</td>
<td>Column 5</td>
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<tr>
<td></td>
<td>glass strings</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Debulks</td>
<td>None</td>
<td>P3 15 minutes every 2(^{nd}) Ply</td>
<td>Column 6</td>
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<tr>
<td>Interaction between impregnation and out</td>
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<td>N/A</td>
<td>Column 7</td>
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## Taguchi L8 Array

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<th>Col.2</th>
<th>Col.3</th>
<th>Col.4</th>
<th>Col.5</th>
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</table>
Example of ANOVA Parameter Sensitivity
Plots (Of Void Content)
Comments on Fabric Prepreg Specification Document –
Sections 2.4 and 2.5 Material Qualification Process using an Industry vs. an End User Specification

- Industry specifications don’t yet exist, so the supplier may need to create his own material and process specifications as an interim measure.
- Supplier’s specifications are used to make the initial prepreg qualification batches and panels and may subsequently (once the equivalency and acceptance values have been derived from the allowables data) also be used in one of three ways:
  - As a supplier specification for a commodity product, in much the same way as carbon fiber is supplied.
  - As a draft for an end user specification, where the end user is demonstrating equivalency to the original allowables database and wishes to have a document under his control rather than under the control of the supplier.
  - As a draft format for submission of data for inclusion in an industry specification at a later date.
- A test plan is still required to define the qualification route.
- The spirit of the qualification plan should be such that it’s validity is independent of who writes the plan, writes the specifications, makes panels and specimens, performs testing, where the data is published etc. providing the approach is technically sound, makes logical sense and is fully embodied and described in the test plan itself. Such issues of procedure often seem to obscure or even hinder the real objective of the program, which is to produce valid data for design and material control purposes.
“The process should be capable of producing consistent laminates of high quality”
- More importantly, the process should be capable of producing laminates with quality and variability representative of those of production parts. For oven/vacuum bag processing this may mean less than perfection.

Nondestructive inspection of Test Panels.
- This is correctly stated as a recommendation rather than a requirement.
- It is difficult to justify using NDI data to disposition laminates with higher than desirable levels of porosity – this may be simply representative of process capability rather than grounds for rejection unless some identifiable cause, such as a bag leak, can be found.
Comments on Fabric Prepreg Specification Document – 5.6.1.3 Definition of number of fabric and fiber batches

- Document recommends a minimum of three different material batches consisting of a minimum of two different fiber batches and three different resin batches.
- This may not be acceptable to some end users.
- A commonly used batch definition for carbon fiber (not glass fiber!) fabric prepregs is:

<table>
<thead>
<tr>
<th>Fabric Prepreg Batch</th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fabric Batch</td>
<td>Fabric Batch A</td>
<td>Fabric Batch B</td>
<td>Fabric Batch C</td>
</tr>
<tr>
<td>Warp Yarn</td>
<td>Fiber Lot 1</td>
<td>Fiber Lot 3</td>
<td>Fiber Lot 4</td>
</tr>
<tr>
<td>Fill Yarn</td>
<td>Fiber Lot 2</td>
<td>Fiber Lot 1</td>
<td>Fiber Lot 5</td>
</tr>
<tr>
<td>Resin Batch</td>
<td>1</td>
<td>2</td>
<td>3</td>
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</tbody>
</table>
ACG strongly supports the proposed approach and has attempted to adopt these evolving recommendations for a major program of work currently in progress under Air Force funding.

The guidelines documents must tread a fine line – it is very common to have a recommendation or an example interpreted as policy. Flexibility needs to be emphasized where it is appropriate, particularly in respect of the specifics of the qualification process (i.e. who makes and tests what etc. in any specific case).

In executing an independent qualification program, a material supplier cannot afford to operate in a vacuum. Input from the intended end users is critical in order to ensure that the end result is what is needed.

It is quite likely that data generated independently by a material supplier can equally be used for both an end user specification and for a national specification – it does not need to be an either/or. Equally there is no reason why the supplier could not continue supplying the material to its own specification as a commodity “off the shelf” product.

Change management and agreement on the levels of change, intentional or otherwise, and their consequences, remain difficult issues.

The issue of such qualifications being executed overseas under the jurisdiction of other airworthiness authorities should be considered. The value of this methodology is in principle independent of geography, but the procedures and policies of other authorities differ from those of the FAA although designed to achieve the same end result.