Bond Process Qualification Protocols & Adhesive Qualification Guidance for Aircraft Design and Certification

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  • Upul Palliyaguru, Anushi Amaranayake, and Tammy Nguyen

• FAA Technical Monitor
  • Ahmet Oztekin

• Other FAA/CMH-17 Personnel Involved
  • Larry Ilcewicz, PhD, Cindy Ashforth, and Curtis Davies,

• DoD & Industry Participation
  • AFRL, Boeing, Bell Helicopter, Henkel, Honda Aircraft Co., Lockheed Martin, MMM, MTech Engineering Services, NAVAIR, Solvey Industries, Textron Aviation, Boom Aerospace
• Adhesive Characterization Qualification (ACQ)
  • Develop test matrices
    • Bulk physical, chemical, and mechanical test matrices
    • Adhesive (joint) mechanical tests
    • Fluid sensitivity
    • Equivalency tests
  • Develop databases
    • Select adhesive bond systems
    • Inclusion to CMH-17 data review group
Road Map - Adhesive Qualification Guidance

Development of NCAMP Specifications

Guideline Document (FAA Report)
Guidelines and Recommended Criteria for the Development of Material/Process Specifications for Adhesive

Adhesive Process Control Document (PCD) Preparation and Maintenance Guide

Qualified Product List (QPL)
- NMS 300/1
- NMS 394/1
- ...

Material Specifications

NCAMP Material Specification (NMS)
Solvey FM300-2
Loctite EA9394
NMS 394/1 EA 9394 AERO

NCAMP Process Specification (NPS)
Solvey FM300-2
Loctite EA9394

Process Specifications

Conformity

Material Property Data Report
Statistical Analysis Report

Fabrication of bulk and mechanical test panels

Development of NCAMP Test Plans

NCAMP Test Plan (NTP)
Solvey FM300-2
Loctite EA9394

Authorized Inspection Representative (AIR)

Authorized Engineering Representative (AER)

Data Collection Templates (PMC)
Development of NCAMP Specifications, Test Plans & Guidelines

- Adhesive System 1 – FM300-2M (Film Adhesive)
  - NCAMP Material Specification (Base) – NMS300
  - NCAMP Material Specification (Slash) – NMS300/1
  - NCAMP Process Specifications - NPS 83002
  - NCAMP Test Plan - NTP AC-3002Q1
- Adhesive System 2 – EA9394 bare (Paste Adhesive)
  - NCAMP Material Specification (Base) – NMS394
  - NCAMP Material Specification (Slash) – NMS394/1
  - NCAMP Process Specifications - NPS 89394
  - NCAMP Test Plan - NTP AC-9394Q1
- Adhesive Process Control Document (PCD) – NRP 105
- NCAMP - Adhesive Data Collection Forms
NCAMP Material Specifications

Summary of documents -&gt; NMS 300 (Base)
250°F Cure High Temperature and High Toughness Structural Film Adhesive

- Covers Solvay FM300-2 types
  - Carrier material types
    - Type K – Wide open knit
    - Type M – Random mat
    - Type U – Unsupported film
  - Nominal weights
    - 0.10 psf
    - 0.08 psf
    - 0.06 psf
    - 0.03 psf
- Changes to the qualified materials
- Tests performed on adhesive

Uncured adhesive physical and chemical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard</th>
<th># of Replicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>HPLC</td>
<td>Uncured</td>
<td>SACMA SRM 20R-94</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>FTIR</td>
<td>Uncured</td>
<td>ASTM E1-188, ASTM E1252</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Viscosity &amp; Gel Time</td>
<td>Uncured</td>
<td>ASTM D4473</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Resin Flow</td>
<td>Uncured</td>
<td>ASTM D3531</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Volatile Content</td>
<td>Uncured</td>
<td>ASTM D3530</td>
<td>3 per Batch</td>
</tr>
</tbody>
</table>

Cured adhesive physical and thermal properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard</th>
<th># of Replicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cured Panel Thickness</td>
<td>Cured</td>
<td>SACMA SRM 10R-94</td>
<td>10 per Panel</td>
</tr>
<tr>
<td>Tg</td>
<td>Cured</td>
<td>ASTM D7028</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Density</td>
<td>Cured</td>
<td>ASTM D792 Method A</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Thermal Conductivity &amp; Thermal Diffusivity</td>
<td>Cured</td>
<td>ASTM E1952</td>
<td>3 per Batch</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ASTM E1530</td>
<td>3 per Batch</td>
</tr>
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</table>

Cured adhesive mechanical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Temperature</th>
<th>Test Method</th>
<th># of Replicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension Strength &amp; Modulus</td>
<td>RT</td>
<td>ASTM D638</td>
<td>5</td>
</tr>
<tr>
<td>Compression Strength and Modulus</td>
<td>RT</td>
<td>ASTM D695</td>
<td>5</td>
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<tr>
<td>Shear Strength and Modulus</td>
<td>RT</td>
<td>ASTM D3846</td>
<td>5</td>
</tr>
</tbody>
</table>
NCAMP Material Specifications
Summary of documents -> NMS 394 (Base)
150°F Cure High Temperature and High Toughness Structural Paste Adhesive

- Covers Henkel EA9394 types
- Bondline Control Mechanisms
  - EA9394.3 - 0.005 Glass beads
- Adhesive variations weights
  - EA 9394
  - EA 9394/C-2
  - EA 9394.2
- Changes to the qualified materials
- Tests performed on adhesive

### Uncured adhesive physical and chemical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard</th>
<th># of Replicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>HPLC</td>
<td>Uncured</td>
<td>SACMA SRM 20R-94</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>FTIR</td>
<td>Uncured</td>
<td>ASTM E-168 ASTM E-1252</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Viscosity &amp; Gel Time</td>
<td>Uncured</td>
<td>ASTM D4473</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Resin Flow</td>
<td>Uncured</td>
<td>ASTM D3531</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Volatile Content</td>
<td>Uncured</td>
<td>ASTM D3530</td>
<td>3 per Batch</td>
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### Cured adhesive physical and thermal properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard</th>
<th># of Replicates</th>
</tr>
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<tbody>
<tr>
<td>Cured Panel Thickness</td>
<td>Cured</td>
<td>SACMA SRM 10R-94</td>
<td>10 per Panel</td>
</tr>
<tr>
<td>Tg</td>
<td>Cured</td>
<td>ASTM D7028</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Density</td>
<td>Cured</td>
<td>ASTM D792 Method A</td>
<td>3 per Batch</td>
</tr>
<tr>
<td>Thermal Conductivity &amp; Thermal Diffusivity</td>
<td>Cured</td>
<td>ASTM E1952</td>
<td>3 per Batch</td>
</tr>
<tr>
<td></td>
<td>Cured</td>
<td>ASTM E1830</td>
<td>3 per Batch</td>
</tr>
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</table>

### Cured adhesive mechanical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Temperature</th>
<th>Test Method</th>
<th># of Replicates</th>
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<tr>
<td>Tension Strength &amp; Modulus</td>
<td>RT</td>
<td>ASTM D638</td>
<td>5</td>
</tr>
<tr>
<td>Compression Strength and Modulus</td>
<td>RT</td>
<td>ASTM D695</td>
<td>5</td>
</tr>
<tr>
<td>Shear Strength and Modulus</td>
<td>RT</td>
<td>ASTM D3846</td>
<td>5</td>
</tr>
</tbody>
</table>
NCAMP Material Specifications
Summary of document -> NMS 300 (Base)
250°F Cure High Temperature and High Toughness Structural Film Adhesive

• Storage and Handling Requirements
  • General requirements
  • Adhesive life requirements
  • Safety requirements
• Quality Assurance
  • Classification of tests and Inspections
  • Receiving inspections
• Delivery of Materials
  • Packaging
  • Shipping Requirements
• Material Rejection

Out-Time
Handling Life*
(function of factory environment)
Staging Life**
(function of factory environment)

Storage Life
(function of storage temperature)
Date of Shipment
Out of Storage
End of Storage Life
Adhesive laid up on the substrates
Part Cure

*a.k.a. application, assembly, or ambient work life
**a.k.a. mechanical or tool life
NCAMP Material Specifications
Summary of document -> NMS 300/1 (Slash)
(Solvay FM300-2M – 0.06psf)

- QPL - Solvay FM300-2M – 0.06psf
- Technical Requirements

Uncured adhesive physical and chemical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard</th>
<th># of Replicates (Every roll)</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>HPLC</td>
<td>Uncured</td>
<td>SACMA SRM 20R-94</td>
<td>3</td>
<td>TBD</td>
</tr>
<tr>
<td>FTIR</td>
<td>Uncured</td>
<td>ASTM E168</td>
<td>3</td>
<td>TBD</td>
</tr>
<tr>
<td>Viscosity &amp; Gel Time</td>
<td>Uncured</td>
<td>ASTM D4473</td>
<td>3</td>
<td>TBD</td>
</tr>
<tr>
<td>Resin Flow</td>
<td>Uncured</td>
<td>ASTM D2183</td>
<td>3</td>
<td>Minimum Average – 575%; Ind. Min 540.5%</td>
</tr>
<tr>
<td>Volatile Content</td>
<td>Uncured</td>
<td>ASTM D3530</td>
<td>3</td>
<td>Average maximum – 1%</td>
</tr>
</tbody>
</table>

Cured bulk adhesive mechanical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Temperature</th>
<th>Test Method</th>
<th>Number of Replicates</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension Strength &amp; Modulus</td>
<td>RT</td>
<td>ASTM D638</td>
<td>5</td>
<td>TBD</td>
</tr>
<tr>
<td>Compression Strength and Modulus</td>
<td>RT</td>
<td>ASTM D695</td>
<td>5</td>
<td>TBD</td>
</tr>
<tr>
<td>Shear Strength and Modulus</td>
<td>RT</td>
<td>ASTM D3846</td>
<td>5</td>
<td>TBD</td>
</tr>
</tbody>
</table>

Adhesive mechanical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Temperature</th>
<th>Test Method</th>
<th>Number of Replicates</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lap Shear Strength</td>
<td>RT</td>
<td>ASTM D1002</td>
<td>5</td>
<td>Minimum Average 4850 psi; Ind. Min 4559 psi</td>
</tr>
<tr>
<td>Flatwise Tensile Strength</td>
<td>RT</td>
<td>ASTM D897</td>
<td>5</td>
<td>Minimum Average 892 psi; Ind. Min 838 psi</td>
</tr>
<tr>
<td>Peel Strength</td>
<td>RT</td>
<td>ASTM D3167</td>
<td>5</td>
<td>Minimum Average 31 in-lbf/in; Ind. Min 29 in-lbf/in</td>
</tr>
</tbody>
</table>
NCAMP Material Specifications
Summary of document -> NMS 394/1 (Slash) (Henkel EA9394 bare)

- QPL - Henkel EA9394 bare
- Technical Requirements

### Uncured adhesive physical and chemical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard¹</th>
<th># of Replicates</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>HPLC</td>
<td>Uncured</td>
<td>SACMA SRM 20R-94</td>
<td>3</td>
<td>TBD</td>
</tr>
<tr>
<td>FTIR</td>
<td>Uncured</td>
<td>ASTM E168</td>
<td>3</td>
<td>TBD</td>
</tr>
<tr>
<td>Viscosity &amp; Gel Time</td>
<td>Uncured</td>
<td>ASTM D4473</td>
<td>3</td>
<td>Minimum Average = 160 Pa·S Ind. Avg = 150 Pa·S</td>
</tr>
<tr>
<td>Resin Flow</td>
<td>Uncured</td>
<td>ASTM D22183</td>
<td>3</td>
<td>TBD</td>
</tr>
<tr>
<td>Volatile Content</td>
<td>Uncured</td>
<td>ASTM D3530-97</td>
<td>3</td>
<td>TBD</td>
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</table>

### Cured bulk adhesive mechanical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Temperature</th>
<th>Test Method¹</th>
<th># of Replicates</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension Strength &amp; Modulus</td>
<td>RT</td>
<td>ASTM D638</td>
<td>5</td>
<td>Minimum Average 6675 psi; Ind. Min 6274 psi</td>
</tr>
<tr>
<td>Compression Strength and Modulus</td>
<td>RT</td>
<td>ASTMD695</td>
<td>5</td>
<td>Minimum Average 10000 psi; Ind. Min 9400 psi</td>
</tr>
<tr>
<td>Shear Strength and Modulus</td>
<td>RT</td>
<td>ASTMD3846</td>
<td>5</td>
<td>TBD</td>
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</tbody>
</table>

### Adhesive mechanical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Temperature</th>
<th>Test Method¹</th>
<th>Number of Replicates</th>
<th>Requirements</th>
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<td>Lap Shear Strength</td>
<td>RT</td>
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<td>5</td>
<td>Minimum Average 4850 psi; Ind. Min 4559 psi</td>
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<td>Flatwise Tensile Strength</td>
<td>RT</td>
<td>ASTM D897</td>
<td>5</td>
<td>Minimum Average 892 psi; Ind. Min 838 psi</td>
</tr>
<tr>
<td>Peel Strength</td>
<td>RT</td>
<td>ASTM D3167</td>
<td>5</td>
<td>Minimum Average 31 in-lb/in; Ind. Min 29 in-lb/in</td>
</tr>
</tbody>
</table>

¹: Test Method indicates the standard used for testing.
Materials used for bonded panel fabrication

Adhesive preparation

Substrate preparation

- Aluminum (Al2024 T3) – PAA (ASTM D3933) + BR 127
  - Pre-bond measurements
    - Thickness
    - Surface roughness
    - Primer thickness

- Composite (Epoxyglass G10) – 120 grit sanding + water break test + cleaning
  - Pre-bond measurements
    - Thickness
    - Surface roughness
NCAMP Process Specifications

Summary of document -> NPS 83002
Fabrication of NMS 300 Qualification, Equivalency and Acceptance Test Panels

• Fabrication/Bonding of mechanical test panels
  • Bonding of panels with a lap area
  • Bonding of panels without a lap area (Full surface bond)
  • Bonding of panels with a disbond

• Fabrication of bulk adhesive panels

• Bagging Scheme

• Cure Cycle –250°F for 90 minutes with 40 psi pressure.

• Quality control
  • Visual inspection
  • TTU c-scan
NCAMP Test Plan
Summary of document -> NTP AC-3002Q1
Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

- Nomenclature for specimen identification
- Physical, Chemical and Thermal tests performed on adhesive

### Uncured adhesive physical and chemical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard</th>
<th>Min Replicates per batch</th>
</tr>
</thead>
<tbody>
<tr>
<td>HPLC</td>
<td>Uncured</td>
<td>SACMA SRM 20R-94</td>
<td>3</td>
</tr>
<tr>
<td>FTIR</td>
<td>Uncured</td>
<td>ASTM-E168 &amp; ASTM-E1252</td>
<td>3</td>
</tr>
<tr>
<td>Viscosity &amp; Gel Time</td>
<td>Uncured</td>
<td>ASTM D4473</td>
<td>3</td>
</tr>
<tr>
<td>Resin Flow</td>
<td>Uncured</td>
<td>ASTM D2183</td>
<td>3</td>
</tr>
<tr>
<td>Volatile Content</td>
<td>Uncured</td>
<td>ASTM D3530</td>
<td>3</td>
</tr>
</tbody>
</table>

### Cured adhesive physical and thermal properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Product Form</th>
<th>Test Standard</th>
<th>Min Replicates per batch</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tg</td>
<td>Cured</td>
<td>ASTM D7028</td>
<td>3</td>
</tr>
<tr>
<td>Density</td>
<td>Cured</td>
<td>ASTM D792 Method A</td>
<td>3</td>
</tr>
<tr>
<td>Thermal Conductivity &amp; Thermal Diffusivity</td>
<td>Cured</td>
<td>ASTM E1952</td>
<td>3</td>
</tr>
</tbody>
</table>
NCAMP Test Plan

Summary of document -> NTP AC-3002Q1
Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

- Adhesive Mechanical Property Testing
  - Test Environments -> CTD (-65F), RTD (70F), ETD (180F Dry), and ETW (180F Wet)
  - Dry – as fabricated moisture content
  - Wet – specimens conditioned at 145°F/85%RH for 1000 hrs. (FM300-2M)

- Specimen distribution methodology
## NCAMP Test Plan

### Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

- **Adhesive Mechanical Property Testing**
  - **Test Matrix**

<table>
<thead>
<tr>
<th>Test Description</th>
<th>Test Code</th>
<th>Test Standard</th>
<th>Property</th>
<th>Number of Batches x No. of Panels x No. of Specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thin Metal Adherend Single Lap Shear</td>
<td>SLS – MThin</td>
<td>ASTM D1002</td>
<td>Strength</td>
<td>3 x 2 x 3, 3 x 2 x 3, 1 x 2 x 3, 3 x 2 x 3</td>
</tr>
<tr>
<td>Thick Metal Adherend Single Lap Shear</td>
<td>SLS - MThick</td>
<td>ASTM D5656</td>
<td>Strength and Modulus</td>
<td>1 x 2 x 3, 1 x 2 x 3, 1 x 2 x 3, 1 x 2 x 3</td>
</tr>
<tr>
<td>Thin Laminate Adherend Single Lap Shear</td>
<td>SLS - CThin</td>
<td>ASTM D3165</td>
<td>Strength</td>
<td>3 x 2 x 3, 3 x 2 x 3, 1 x 2 x 3, 3 x 2 x 3</td>
</tr>
<tr>
<td>Mode I Fracture Toughness</td>
<td>M-I</td>
<td>ASTM D3433</td>
<td>Fracture Toughness Gic</td>
<td>1 x 2 x 3, 1 x 2 x 3, 1 x 2 x 3, 1 x 2 x 3</td>
</tr>
<tr>
<td>Mode II Fracture Toughness</td>
<td>M-II</td>
<td>ASTM D7905</td>
<td>Fracture Toughness Giic</td>
<td>1 x 2 x 3, 1 x 2 x 3, 1 x 2 x 3, 1 x 2 x 3</td>
</tr>
<tr>
<td>Floating Roller Peel</td>
<td>FRP</td>
<td>ASTM D3167</td>
<td>Peel Load</td>
<td>3 x 2 x 3, 3 x 2 x 3, 1 x 2 x 3, 3 x 2 x 3</td>
</tr>
<tr>
<td>Flatwise Tensile</td>
<td>FWT</td>
<td>ASTM D897</td>
<td>Strength</td>
<td>3 x 2 x 3, 3 x 2 x 3, 1 x 2 x 3, 3 x 2 x 3</td>
</tr>
</tbody>
</table>
### Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

**Summary of document -> NTP AC-3002Q1**

**Adhesive Mechanical Property Testing – Fluid Sensitivity**

**Test Matrix**

<table>
<thead>
<tr>
<th>Extended Contact:</th>
<th>Exposure</th>
<th>Test Condition</th>
<th>Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAE AMS 2629 Jet Reference Fluid</td>
<td>30 days min. @ 70°F±10°F</td>
<td>70°F</td>
<td>FS11RT</td>
</tr>
<tr>
<td></td>
<td>30 days min. @ 70°F±10°F</td>
<td>180°F</td>
<td>FS11ET</td>
</tr>
<tr>
<td>MIL-PRF-5606 Hydraulic Oil</td>
<td>30 days min. @ 70°F±10°F</td>
<td>70°F</td>
<td>FS12RT</td>
</tr>
<tr>
<td></td>
<td>30 days min. @ 70°F±10°F</td>
<td>180°F</td>
<td>FS12ET</td>
</tr>
<tr>
<td>Sea Water (ASTM D1141 or equiv.)</td>
<td>30 days min. @ 70°F±10°F</td>
<td>70°F</td>
<td>FS13RT</td>
</tr>
<tr>
<td></td>
<td>30 days min. @ 70°F±10°F</td>
<td>180°F</td>
<td>FS13ET</td>
</tr>
<tr>
<td>Skydrol LD-4 (SAE AS1241, Type IV, Class 1)</td>
<td>30 days min. @ 70°F±10°F</td>
<td>70°F</td>
<td>FS14RT</td>
</tr>
<tr>
<td></td>
<td>30 days min. @ 70°F±10°F</td>
<td>180°F</td>
<td>FS14ET</td>
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<td>FS21RT</td>
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<tr>
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<td>85% Relative Humidity</td>
<td>1000 hrs. at 145°F±5°F /85%±5% RH</td>
<td>70°F</td>
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<td>1000 hrs. at 145°F±5°F /85%±5% RH</td>
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Preliminary Test Results
EA 9394 – D1002 – Property Value – Shear Strength (ksi)

Batch A
Cure Cycle 1  Cure Cycle 2

Higher % of Cohesive / Adhesive
Preliminary Test Results
EA 9394 – D3165 – Property Value – Shear Strength (ksi)

Batch A
Cure Cycle 1  Cure Cycle 2

Adhesive Failure  Adhesive Failure
Preliminary Test Results
EA 9394 – D3167 – Property Value – Peel Load (lbf)

Batch A
Cure Cycle 1
Adhesive Failure

Cure Cycle 2
Adhesive Failure
Preliminary Test Results
EA 9394 – D3433 – Property Value – Fracture Toughness (lbf/in)

Batcch A

<table>
<thead>
<tr>
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<td>Cohesive Failure</td>
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Preliminary Test Results

EA 9394 – D5656 (T1) – Property Value – Shear Strength (ksi)

T1
Batch A

Cure Cycle 1  Cure Cycle 2

Cohesive / Adhesive

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<td>RTA 75°F</td>
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<td>8</td>
<td>EPA1 180°F</td>
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Legend:
- MEAN
- B-Basis (Pooled)
- B-Basis (Single Point)
- B-Basis (Pooled Modified C.V.)
- A-basis (Pooled)
- A-Basis (Single Point)
- A-basis (Pooled Modified C.V.)
- Maximum
- Minimum
Preliminary Test Results
EA 9394 – D5656 (T2) – Property Value – Shear Strength (ksi)

EA 9394 – D5656 (T2)

Cohesive / Adhesive

Cure Cycle 1
Cure Cycle 2

Batch A

T2
Preliminary Test Results
EA 9394 – D1002 – Fluid Sensitivity Test Results

Shear Strength (ksi)

Fluid Sensitivity

- SAEAMS 262: Jet Fuel
- MILPRF-5606: Hydraulic Oil
- Sea Water (ASTM D1141)
- Skydrol LD-4 (SAE AS1241)
- MK washing fluid (ASTM)

Moisture Condition

RT

ET
### Current Status

**EA 9394**

<table>
<thead>
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<th>Test Method</th>
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**FM300-2M**

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<td>CTD</td>
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<td>Mode II Fracture Toughness</td>
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<td>CTD</td>
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<td>Flatwise Tensile</td>
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<td>Fluid Sensitivity</td>
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<td>ET</td>
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</table>

- **Color Legend**
  - **Waiting on Adhesive Batch**
  - **Specimen/Panel Bonding in Progress**
  - **Specimen Machining in Progress**
  - **Conditioning in Progress**
  - **Testing in Progress**
  - **Testing Complete**
Look Forward

• **Future Activities**
  - Generate the B-Basis allowable for EA9394 and FM300-2M material systems
  - Focus on performing equivalency on adhesive materials.
  - Analyze failure modes for different test environments and report them accordingly

• **Benefit to the Aviation Community**
  - Guidance on test matrices for mechanical, physical and chemical characterization of adhesives
  - Generate adhesive material databases under NCAMP protocols that can be used for a wide variety of applications by different end users
**Bond Process Qualification Protocols - Road Map**

- **Bond Process Qualification (BPQ)**
  - Develop an acceptance criteria
    - Requirements (based on information in AC’s and FAR’s, etc.)
    - Applicability of existing standards and/or develop new standards
  - Select known bond system failures
    - Simulate and investigate the BPQ methodology flags the “bad” bonds
  - **Develop protocols**
    - Quantify process reliability
    - Assess repeatability/maturity
• Substrate type
  • Metal
  • Composite
• Adhesive types
  • Film
  • Paste
Summary of Activities

• Current Activities
  • Task 1 – Substrate and adhesive compatibility
  • Task 2 – Use of peel ply for composite substrate preparation

• Completed Activities
  • Effects of Mix-ratio in two part paste adhesives
  • Evaluation of assembly time in paste adhesives
    • Amine blush effects
  • Fluid Sensitivity of adhesive
  • Efficient adhesive screening method testing.
# Qualification of a Bond Process

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Adhesive System</th>
<th>Substrate Surface Preparation</th>
<th>Bonding Process/Curing</th>
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<tr>
<td>2. Metal</td>
<td>2. Film</td>
<td>1. Grid Blasting</td>
<td>2. Vacuum or Pressure</td>
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<td>2. Hand Sanding</td>
<td>3. Bondline Control</td>
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<td>2. Peel Ply</td>
<td>4. Assembly Time</td>
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<td>4. Other</td>
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<td>1. Plasma Treatment</td>
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Qualification of a Bond Process

Task 1

1. Composite
2. Metal

1. Paste
2. Film

1. Abrasion
   1. Grid Blasting
   2. Hand Sanding

2. Peel Ply
   1. Nylon
   2. Polyester

3. Chemical
   1. PAA + Priming
   4. Other
     1. Plasma Treatment

Task 2

1. Cure Cycle – Compatibility with Substrate
2. Vacuum or Pressure
3. Bondline Control
4. Assembly Time
5. Adhesive Preparation
6. Environment
7. Bond Configuration – Co-bond/Secondary bond
When using bonded joints for primary or secondary structure applications, there is a wide variety of substrates and adhesive materials that are available for use. Providing Guidance on selecting a compatible substrate and a adhesive combination is important for designers.

Main factors to consider during adhesive and substrate selection are the mechanical property requirements, physical compatibility of the substrates (hybrid and non-hybrid) and adhesives for bonding, thermal compatibility of the bond system during the bonding process and service life.

Objective of this task is to provide establish a set of guidelines to use when selecting an adhesive and substrate combination for a given bond process.

Goal – Develop guidelines on how to select compatible substrate and adhesive combinations to obtain a robust bond system
Task 1 - Qualification of a Bond Process – Substrate and Adhesive Compatibility

Substrate + Adhesive System + Substrate Surface Preparation + Bonding Process/Curing

Compatibility

Mechanical
1. Material allowable properties
   1. Substrate data -> NCAMP
   2. Adhesive data -> NCAMP (in progress)
2. Joint mechanical capability
   1. Static
   2. Durability

Physical
1. Surface Characterization
   1. Surface roughness
   2. Surface energy/Contact angle
   3. Wettability envelope

Thermal
1. CTE Mismatch
2. Glass Transition
3. Cure Cycle Compatibility
   1. Secondary bond
   2. Co-bonded
Task 1 - Substrate and Adhesive Compatibility

**Substrates**
- Carbon Fiber Composites
  - UNI – T800/3900-2
  - PW - T300/3900-2
- Glass Fiber Composites
  - Fabric – Epoxyglass G10
- Metallic
  - AL 2024-T3
  - Ti – Grade 2

**Adhesives**
- Paste Adhesives
  - Henkel EA9394
  - Henkel EA9390
  - Cytec 680-3
- Film Adhesive
  - Cytec FM300-2M
  - 3M-AF163

**Cure Cycles**
- Manufacturer recommended cure cycle

**Surface Preparation Methods**
- Carbon fiber composites
  - As fabricated – MEK wipe only
  - Peel ply only
  - Peel ply + Light abrasion (180 grit)
  - Abrasion only (120 grit)
- Glass fiber composites
  - As fabricated
  - Abrasion only (120 grit)
- Metallic substrates
  - MEK wipe only
  - PAA+BR127
  - 3M - AC 130-2 surface treatment
  - Abrasion
  - Chemical treatment – ASTM D2651 (Ti)
Task 1 - Substrate and Adhesive Compatibility Assessment

Physical Compatibility

• Objective
  • Generate guidelines to ensure the surface preparation + substrates are physically compatible for bonding.

• Physical Compatibility
  • Surface morphology related tests for substrates
    • Surface roughness
    • Contact angle
    • Surface Energy
  • Wettability envelope development
    • Measure polar and dispersive surface energies for substrate and compare the surface energy of the adhesive.
    • Objective:- A simplistic rapid approach to evaluate if the adhesive surface tension falls within the wettability envelope == Good bond
  • Perform qualitative tests to assess the bond failures
    • Wedge crack
    • Rapid adhesion test
    • Flatwise tensile
Task 1 - Substrate and Adhesive Compatibility Assessment

Physical Compatibility

- Develop the relationship between the
  - Contact angle of substrates vs. bond quality.
  - Elapsed time vs contact angle
- Use in-situ surface energy measurement techniques (BTG-Labs - Surface Analysts) to assess the substrate characterizations
- Repeat the process for a given adhesive for different mechanical properties (Shear, Peel, Fracture Toughness)
Task 1 - Substrate and Adhesive Compatibility Assessment
Physical Compatibility – Current Status

- Fabrication of composites test panels completed.
- Preliminary experiments completed for surface energy/contact angle measurements
- Guideline development for substrate preparation timeline is in progress
Task 1 - Substrate and Adhesive Compatibility Assessment
Thermal Compatibility

• Objective
  • Generate guidelines to ensure the thermal properties of substrates and adhesives are compatible for bonding and during service life.

• Thermal Compatibility
  • CTE mismatch
    • Between substrates and adhesives cured at elevated temperatures
    • CTE mismatch in bonded structures during service life – Cold and elevated temperature environments – formation of micro cracks during thermal cycling
  • Glass Transition Temperatures
    • Mismatch in glass transition temperatures and out it could potentially effect the bond integrity
  • Pose cure effect on the substrates (composites) for secondary bonded structures
    • Understand how the critical mechanical and thermal properties change after exposing to high temperatures for long durations (cure profile of a adhesive)
Substrate and Adhesive Compatibility Assessment

Mechanical Properties

- **Mechanical Properties**
  - **Coupon level testing**
    - Perform coupon level testing to evaluate static and durability capability. (using the actual bond process that will be used for the application)
      - Shear
      - Peel
      - Fracture toughness
  - **Element/Component level testing**
    - Fabricate a representative bonded structures
    - Perform mechanical testing (static and fatigue)
    - Assess the bond quality / Perform NDI

Future Activities
Task 1 - Substrate and Adhesive Compatibility

Additional Tasks

• Extend current research with industry partners to further investigate substrate adhesive compatibility.
• Substrate Material – Tencate T350-1/IM7
• Adhesive System – EA 9394

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<th>#Spec.</th>
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Test Type

- Single Lap Shear
- Lap Shear and Stress/Strain
- T-Peel
- Floating Roller Peel
- Fracture Toughness
- Flatwise Tensile
Use of peel ply as a surface preparation method reduces the amount of labor involved and simplify the substrate preparation process. It also provides a uniform and repeatable surface for bonding.

Peel ply prepared surface quality vary on many substrate and surface preparation process parameters. Bond surface quality directly effects the bond integrity. Understanding the effects of these parameters is critical. Development of reliable and rapid inspection methods is crucial to ensure the bond process (surface preparation) method is appropriate for a given bond system.

After an appropriate peel ply surface preparation method is chosen, there are many other parameters associated with handling substrates that could potentially change the quality of the bond surfaces. These parameters and their adverse effect on the bond integrity needs to be evaluated to provide Guidance and Develop Protocols to have a robust bond system.

Goal – Develop guidelines and protocols to handle peel ply prepared surfaces to obtain a robust bond system.
Peel Ply Surface Preparation Evaluation

- Peel ply removal preparation method provides a repeatable uniformly prepared surface for bonding with, minimum labor.
  - For guideline development, need to understand
    - The effect of different peel ply materials and thicknesses
    - Surface contamination created and ways to reduce it (during application and removal of peel ply and the timeframe of removal)
    - Rapid inspection methods to ensure the surface quality of the substrates
    - Peel ply prepared surface exposure to extreme environments (hot/wet)
    - Any adverse effects to the laminate due to having the peel ply during cure cycle.
    - Effects of peel ply prepared surfaces going through multiple cure cycles.
Peel Ply Surface Preparation Evaluation

- **Types of Peel Ply**
  - **Polyester (Non Released)**
    - Wet (Henkel EA 9895)
    - Dry
      - T1 – 60002 (0.005 – 0.006-inch)
      - T2 – 60005 (0.006 – 0.007-inch)
  - **Nylon (Non Released)**
    - Wet (Henkel EA 9896)
    - Dry
      - T1 – 60004 (0.0045 – 0.0055-inch)
      - T2 – 60005 (0.006 – 0.007-inch)
  - **Polyester (Released)**
    - Dry
      - T1 – 60001 (0.005 – 0.006-inch) (SRB)

- **Carbon Composites Substrates**
  - Toray T800/3900-2

- **Adhesive Systems**
  - Film – FM300-2M
  - Paste – EA 9394

- **Cure Cycles**
  - FM300-2M – 250F for 2 hrs. at 40 psi pressure + full vacuum
  - EA 9394 – 150F for 1 hr. 6psi vacuum
Peel Ply Surface Preparation Evaluation

• Surface Preparation Details
  • Peel ply removal only
  • Peel ply removal and light sanding (120 grit)
• Exposure to environment (Room temperature ambient and Elevated temperature wet)
  • Remove peel ply immediately after curing
  • Remove peel ply immediately before bonding (30 days)
• Effects of prepared substrates going through multiple cure cycles
  • Co-bond and repair applications
  • Thermal cycle substrates for multiple times to evaluate the effects
• Controlled/non peel ply configuration
  • Carbon epoxy laminates without peel ply
  • Hand Abrasion (120 grit)
  • No surface preparation (MEK wipe only)
Peel Ply Surface Preparation Evaluation
Methods of Bond Surface Quality Assessment

• Surface Characterization
  • Surface roughness measurements
  • Contact angle measurements
  • Scanning electron microscopic (SEM) inspection for surface details
  • X-ray photoelectron spectroscopy (XPS) to detect surface contamination

• Wettability Envelope Development
Peel Ply Surface Preparation Evaluation
Methods of Mechanical and Physical Property Evaluation

• Fiber Volume Fraction Quantification
  • Due to the resin absorption in peel plies, fiber volume fraction is affected
  • Flatwise tensile testing to quantify the effect.

• Mechanical Property Assessment
  • ASTM D1002/D3165 type Single lap shear to determine the shear strength
  • ASTM D5528 to determine the fracture toughness properties

• Peel Ply Prepared surfaces going through multiple cure cycles
  • Measure the degree of cure for repeated cure cycle – simulation of core bond and repair applications.
Summary of Activities

• Current Activities
  • Task 1 – Substrate and adhesive compatibility
  • Task 2 – Use of peel ply for composite substrate preparation

• Completed Activities
  • Effects of mix-ratio in two part paste adhesives
  • Evaluation of assembly time in paste adhesives
    • Amine blush effects
  • Fluid sensitivity of adhesive
  • Efficient adhesive screening method testing
Effects of incorrect mix-ratio in two part paste adhesives

- Two part adhesive for smaller quantities are available in cartridge form. (Mix ratio is not a concern) For applications that require larger quantities, common method is to obtain them in separate containers and manually mix it. It is important to evaluate the sensitivity of mix ratio in these applications.

- Experimental Approach – used PAA+BR127 and Abrasion + AC120-2 prepared aluminum and carbon composite substrates and fabricated panels with different mix ratios for Part A and part B. Test methods evaluated are D1002 – single lap shear, mode I fracture toughness, and floating roller peel specimens (selected incorrect mix ratios).

- EA 9394 was used for the study with Part A mix ratio error ranging from -40% to +40%
Effects of incorrect mix-ratio in two part paste adhesives
Test Results – Single Lap Shear – ASTM D1002

-40%  -20%  0%  20%  40%
Average Strength [psi]

Incorrect Mix Ratio – Part A Mismatch
Less than nominal Component B
More than nominal Component B

Batch 1 - PAA
Batch 2 - AC-1 30-2

Adhesive/Cohesive Failures
Effects of incorrect mix-ratio in two part paste adhesives
Test Results – Mode I – ASTM D5528

Incorrect Mix Ratio – Part A Mismatch

First Ply/Adhesive Failures
Effects of incorrect mix-ratio in two part paste adhesives

Test Results – Viscosity Response

Viscosity during bonding
Minimum viscosity during cure
Effects of incorrect mix-ratio in two part paste adhesives

Test results – Summary

- EA9394 adhesive system showed a ~±15% change in the mechanical properties of single lap shear and fracture toughness at the extreme mix ratios between -40% & +40%.
- Repeatability of the experiment was validated with a second data set. Data correlates will with the original testing.
- No change in the failure modes was seen between the extreme ends of the experimental procedure.
- Static response of the properties are desirable. However, understanding of the mix ratio effect on fatigue properties needs to be investigated.
Evaluation of assembly time in paste adhesives

- Manufacturer provided pot life is to be used as a material specification. In bonding applications, assembly time is defined as the time it takes to mix, apply adhesive and mate the two parts together. Depending on the bond area and the complexity (contour) of the structure, this could be a critical parameter.

- Experimental Approach – used PAA+BR127 and Abrasion + AC120-2 prepared aluminum and carbon composite substrates and fabricated panels with different assembly times. Test methods evaluated are D1002 – single lap shear, mode I fracture toughness, and floating roller peel specimens (selected incorrect mix ratios).

- Assembly time for EA 9394 was varied from 0, 5, 45, 60, 90, and 120 minutes
Evaluation of assembly time in paste adhesives

Test Results – Single Lap Shear – ASTM D1002

<table>
<thead>
<tr>
<th>Assembly Time [min]</th>
<th>Batch 1 - P AA</th>
<th>Batch 2 - AC-1 30-2</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 min</td>
<td>45 min</td>
<td>60 min</td>
</tr>
<tr>
<td>Average Strength [psi]</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- Higher % Adhesive Failures
- Adhesive/Cohesive
- Cohesive Failures
Evaluation of assembly time in paste adhesives

Test Results – Mode I – ASTM D5528

- Coefficient of Variation [%]
- GIC (5% / MAX) [KJ/m²]

<table>
<thead>
<tr>
<th></th>
<th>5 min</th>
<th>60 min</th>
<th>120 min</th>
</tr>
</thead>
<tbody>
<tr>
<td>First Ply/Adhesive Failures</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Adhesive/Cohesive Failures</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cohesive Failures</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Evaluation of assembly time in paste adhesives
Test Results – Viscosity Response

Viscosity during bonding
Minimum viscosity during cure

Assembly Time [min]
Normalized Strength
Viscosity [p]
Evaluation of assembly time in paste adhesives

Test results – Summary

• EA9394 adhesive system showed very significant effect for longer assembly times.
• Single lap shear strength reduced by ~20% when assembly time was 60 minutes. This started dropping to ~50% for 90 minutes (pot life).
• Mode I fracture toughness data showed a large scatter in test data for increased assembly times.
• Mode I data showed an increase in the properties for 60 minutes assembly and rapidly dropped when the assembly time was increased to 120min.
• Failure modes throughout all the failure modes indicated cohesive/adhesive failures until 60 minute assembly time and changed to cohesive after 60 minute mark.
• Static response of the properties are desirable. However, understanding of the mix ratio effect on fatigue properties needs to be investigated.
Fluid sensitivity of adhesive

- Current method used to evaluate the fluid sensitivity of adhesives is the D1002 single lap shear specimen configurations.
- D1002 Lap Shear specimen configurations requires treated substrate materials which involves numerous steps from preparation to storage.
- The adhesive area exposed to fluids is minimum. (Adhesive Thickness)
- A relatively simpler (test method + specimen geometry) bulk adhesive specimens were fabricated using the adhesive systems and simpler test configuration (3-pt bending) was used to evaluate the effects and compared with the current D1002 method.
- EA 9394 and FM300-2m material systems were used for the evaluation.
- Fluids used in this study:
  - Skydrol LD-4 (SAE AS1241, Type IV, Class 1) – 90 days
  - MEK washing fluid. ASTM D740 – 90 minutes
  - 145°F/85% Relative Humidity 1000hrs – Controlled Condition
Fluid sensitivity of adhesive

Test Results

**EA9394**

<table>
<thead>
<tr>
<th></th>
<th>Normalized Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>BA SE LINE</td>
<td></td>
</tr>
<tr>
<td>MEK - 90M</td>
<td>1.0</td>
</tr>
<tr>
<td>Skydrol L</td>
<td>1.0</td>
</tr>
<tr>
<td>D 4 - 90 days</td>
<td>1.0</td>
</tr>
<tr>
<td>Moisture Conditioning</td>
<td>1.0</td>
</tr>
</tbody>
</table>

**FM300-2M**

<table>
<thead>
<tr>
<th></th>
<th>Normalized Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>BA SE LINE</td>
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<td>MEK - 90M</td>
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</tr>
<tr>
<td>Moisture Conditioning</td>
<td>1.0</td>
</tr>
</tbody>
</table>
Adhesive Screening Test Methods

- Currently ASTM D1002 test method is being used to perform screening test/receiving inspections of adhesive material. ASTM D1002 requires a specialized substrates – treated chemical treatment/preparation method.

- As an alternate to this test method/substrate, Epoxyglass G10 substrates has been evaluated to be used in this type of screening testing.

- Different substrate thicknesses has been evaluated as well as a added new test method.
  - ASTM D1002 – Al substrates – 0.063-in thick (PAA+BR127)
  - ASTM D1002 – Epoxyglass G10 substrates – 0.063-in thick (Abrasion) – Thickness matched
  - ASTM D1002 – Epoxyglass G10 substrates – 0.093-in thick (Abrasion) – El matched
  - ASTM D3165 – Epoxyglass G10 substrates – 0.25-in thick (Abrasion) - Standard

- Adhesive systems evaluated
  - FM300-2M and EA9394
Adhesive Screening Test Methods

Test Results

EA 9394

FM300-2M

Apparent Shear Strength [psi]

Coefficient of Variation [%]

Adhesive/Cohesive Failures

Adhesive Failures

Adhesive/Cohesive Failures

Adhesive Failures
Adhesive Screening Test Methods

Summary

• In a receiving inspection/screening tests, it is usually a Pass/Fail criteria

• Epoxyglass substrates can be used for screening/receiving inspection tests. Baseline tests needs to be performed for the identical specimen configurations.

• Higher variation is seen in the bondline thickness for 0.25-inch thick epoxyglass substrates. Bonding process/bondline control mechanism needs to be revisited to get the required bondline thicknesses.
Looking Forward

• Future Works
  • Generate bond process protocols for
    • Selecting compatible substrate and adhesive combinations for a robust bond structure
    • Provide guidelines on how to select and use peel ply for composite substrate preparation

• Benefit to Aviation
  • Generate bond process protocols
    • Provide guidance on the critical parameters in the bond process and how to mechanically test them to generate protocols
Acknowledgement

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  • Bryce Floryancic
  • Shannon Jones (Textron Aviation)
  • Wei Kwan
  • Royal Lovingfoss (NIAR)
  • Molly Stone
  • Mike Stuart (Solvay)
Questions and Comments ???
Development of NCAMP Specifications

Guideline Document (FAA Report)
- Guidelines and Recommended Criteria for the Development of Material/Process Specifications for Adhesive
- Adhesive NRP 102

Material Specifications
- NCAMP Material Specification (NMS)
  - Solvey FM300-2
  - Loctite EA9394
- NMS 300/1
  - FM300-2M 0.06 psf
- NMS 394/1
  - EA 9394 AERO

Process Specifications
- NCAMP Process Specification (NPS)
  - Solvey FM300-2
  - Loctite EA9394

Qualified Product List (QPL)
- NMS 300/1
- NMS 394/1
- ...

Fabrication of bulk and mechanical test panels
Development of NCAMP Test Plans

NCAMP Test Plan (NTP)

Solvey FM300-2

Loctite EA9394

Authorized Inspection Representative (AIR)

Authorized Engineering Representative (AER)

Data Collection Templates (PMC)

Conformity

Material Property Data Report

Statistical Analysis Report