Adhesive Bond Process Qualification Protocols Development & Development of Roadmap for Bonded Structure Certification

Waruna Seneviratne, John Tomblin, and Upul Palliyaguru

2019 Technical Review - (05/22/2019)
Adhesive Bond Process Qualification Protocols Development & Development of Roadmap for Bonded Structure Certification

- Principal Investigators & Researchers
  - John Tomblin, PhD, and Waruna Seneviratne, PhD
  - Upul Palliyaguru and Anushi Amaranayake

- 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
Aircraft companies tend to use bonded joins in their primary structure due to various time and cost savings. However, qualification of the bond process and certification of the bonded structure requires extensive amount of substantiation work.

Due to the complexity and numerous variables seen in a bond system, locking on to these parameters needs extensive exploration of all possible variations in the bond process. After locking onto this processes, effective and efficient methods for quality assurance needs to be implemented to qualify the bonding process.

After the bond process qualification tasks are completed, bonded structure needs to be certified per the requirements of the safety agencies.

The Primary goal of this research program is to develop a road map for qualification activities of a bond system and support development of certification road map for bonded structures per the safety requirements through substantiation.
Adhesive Bond Process Qualification Protocols Development

**Objective**

Manufacture a bonded wing structure

**Preliminary Design Requirements/Knowledgebase of the Bonded Structure**
1. General Size
2. Mechanical property requirement for Bonded Joints
3. Environmental Envelope
4. Manufacturing Requirements
5. Analysis of critical bonded joint types seen in the structure
6. Bondline thickness requirements

**Preliminary Selection and Screening of Substrate and Adhesive Materials**
1. Material allowable – (Material databases)
2. Adhesive & Substrate compatibility assessment & Wettability assessment
3. Selection of surface preparation methodology
4. Adhesive processing parameters of a representative design.
5. Conduct basic adhesive test methods at room temperature to validate the parameters

**Bond Process Qualification Plan and Protocol Development**
1. Finalized Bond System
2. Based on the parameters, compose a test matrix to ensure quality assurance of surface preparation and processing parameters
3. Generation of quality assurance methodologies

**Structural Certification of Bonded Structure & Maintenance**
1. Screening of bond system
2. Long term durability
3. Substrate & adhesive characterization
4. Bonded joint characterization
5. Durability & environmental scatter
6. Damage Tolerance and Crack growth

**Maintenance**
1. Inspection methodology development
2. Inspection methodology for bond strength degradation.
3. Identification of inspection level and frequency.
Adhesive Bond Process Qualification Protocols Development (Road Map)

Objective
Manufacture a bonded wing structure

Preliminary Design Requirements/Knowledgebase of the Bonded Structure
1. General Size
2. Mechanical property requirement for Bonded Joints
3. Environmental Envelope
4. Manufacturing Requirements
5. Analysis of critical bonded joint types seen in the structure
6. Bondline thickness requirements

Preliminary Selection and Screening of Substrate and Adhesive Materials
1. Material allowable – (Material databases)
2. Adhesive & Substrate compatibility assessment & Wettability assessment
3. Selection of surface preparation methodology
4. Adhesive processing parameters of a representative design.
5. Conduct basic adhesive test methods at room temperature to validate the parameters

Bond Process Qualification Plan and Protocol Development
1. Finalized Bond System
2. Based on the parameters, compose a test matrix to ensure quality assurance of surface preparation and processing parameters
3. Generation of quality assurance methodologies

Structural Certification of Bonded Structure & Maintenance
1. Screening of bond system
2. Long term durability
3. Substrate & adhesive characterization
4. Bonded joint characterization
5. Durability & environmental scatter
6. Damage Tolerance and Crack growth

Maintenance
1. Inspection methodology development
2. Inspection methodology for bond strength degradation.
3. Identification of inspection level and frequency.
Overview of the Presentation

- Preliminary screening and down-selection of adhesive-substrate combinations
- Critical parameters in the surface preparation
  - Surface preparation methodology
  - Quality assurance and handling of prepared substrates
- Critical parameters in the adhesive application and cure process
  - Adhesive handling guidelines
  - Mixing and application
  - Bondline thickness control
- Bond process qualification protocols generation to assess the effect of varying the parameters
Preliminary Selection and Screening of Various Substrate and Adhesive Materials

*Selection of preliminary candidates for the adhesive and substrate materials*

• Bond system parameter down-selection
  • Surface preparation and adhesive processing
    • Phase I – Facility and equipment limitations
      • Preliminary design dimensions – a general idea of the size
      • Facility processing equipment – Curing and pressure application
    • Phase II – Material performance limitations and incompatibility issues
      • Operational environmental envelope
      • Physical - wettability of substrates and adhesive
      • Thermal – CTE and Tg mismatch
      • Mechanical – poor static and durability performance – failure mode based assessment
Bond Process Qualification Protocol Generation
Quality Assurance of Surface Preparation Methodologies

- Pre-surface preparation checklist
  - Quality control and process specification of substrates (cured)
    - Ex. Mold release, surface finish (bag/tool)
  - Quality control of equipment/tools used in surface preparation
    - Material specifications
      - Ex. Sand paper/sanding disks, peel ply, chemicals
    - Operational settings of equipment
      - Ex. – Sander types, speed, force, Plasma generator parameters

- Quality control and training of technicians involved in the process
  - Quantification and assessment of variability in the hand sanding process

- Evaluation of surface preparation to bonding time limitation
  - Effect of environmental exposure duration on surface free energy

- Quality check of the prepared substrates to ensure the integrity of the bond system.
  - Development of surface preparation standards and quantification of the effects

Common Surface Preparation Methods
(Metallic & Composite substrates)

- Abrasion
  - Hand Sanding
  - Grit Blasting
- Peel Ply
- Atmospheric Plasma Treatment (ATP)
- Degreasing
- Chemical Treatments
- Corona Discharge
- Laser Ablation
Bond Process Qualification Protocol Generation
Quality Assurance Standard Development

• Surface Preparation
  • Goal – Increase the surface free energy -> better wettability -> good bonds
  • Method of verification -> Water contact angle measurement
  • Quality check -> Water contact angle measurement comparison to a known standard
  • Equipment used – Surface Analysts – BTG Labs
  • Contact angle measurements validated with Goniometer results.

• Surface preparation quality assurance standard
  • Utilizing different abrasion methods (pressures/grit size) – obtain a range of different surface free energies (contact angles)
  • Fabricate bonded joint specimen and evaluate the bond strength

± 2° variation
Quality Assurance of Surface Preparation – FM300-2M

Substrate - T800/3900-2
Adhesive – FM300-2M
Surface Preparation – Multiple
Test Method – D3165

Cohesion/First Ply
High Adhesion/Low Cohesion
Adhesion
Quality Assurance of Surface Preparation – EA9394

Substrate - T800/3900-2
Adhesive – EA9394
Surface Preparation – Multiple Test Method – D3165

Bond Strength [psi]

<table>
<thead>
<tr>
<th>Bond Strength [psi]</th>
<th>Average Water Contact Angle [degrees]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>25</td>
</tr>
<tr>
<td>500</td>
<td>35</td>
</tr>
<tr>
<td>1000</td>
<td>45</td>
</tr>
<tr>
<td>1500</td>
<td>55</td>
</tr>
<tr>
<td>2000</td>
<td>65</td>
</tr>
<tr>
<td>2500</td>
<td>75</td>
</tr>
<tr>
<td>3000</td>
<td>85</td>
</tr>
<tr>
<td>3500</td>
<td>95</td>
</tr>
<tr>
<td>4000</td>
<td></td>
</tr>
<tr>
<td>4500</td>
<td></td>
</tr>
<tr>
<td>5000</td>
<td></td>
</tr>
</tbody>
</table>

Cohesion/First Ply

Adhesion
Environmental Exposure Effects of Prepared Substrates

Substrate - T800/3900-2
Surface Preparation – Manual 120G

Contact Angle [degrees]

Time [mins]
Surface Preparation - Hand Abrasion Technician Process Variation

Substrate - T650/5320-1
Surface Preparation – Manual Abrasion 120G

Contact Angle [degrees]

Technician

1  2  3  4  5
Surface Preparation - Peel Ply Removal

- Initial assessment of peel plies
  - Commonly used Nylon and Polyester peel ply was used for the study.
- Peel ply usage studies were performed to identify critical parameters
  - Peel ply removal time frame and exposure duration
    - A – Immediately before bonding
    - B – Removed and surface exposed to 14 days.
    - C – Immediately before bonding; exposed to for to 14 days.
  - Mode I and Single Lap Shear Properties
- Post cure effects on peel ply prepared surfaces – Multiple Cure Cycles (MMC)
  - FM300-2M – T800/3900-2 (Substrate and Adhesive combination)
    - Cure Cycles
      - Baseline Initial Cure – 350°F for 2hrs with 85 psi pressure (Substrate Cure)
      - MCC1 – 350°F for 2 hrs.
      - MCC2 – 350°F for 2hrs (X2)
  - Degree of Cure and Fiber Volume Fraction
  - Mode I and Single Lap Shear Properties (in progress)

<table>
<thead>
<tr>
<th>Material</th>
<th>Code</th>
<th>Style</th>
<th>Finish</th>
<th>Thickness [in]</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nylon</td>
<td>40000</td>
<td>56129</td>
<td>60</td>
<td>0.0075 – 0.0085</td>
<td>Natural</td>
</tr>
<tr>
<td>Polyester</td>
<td>60001</td>
<td>60001</td>
<td>60</td>
<td>0.005 – 0.006</td>
<td>Natural</td>
</tr>
<tr>
<td></td>
<td>60002</td>
<td>56030</td>
<td>60</td>
<td>0.005 – 0.006</td>
<td>Natural</td>
</tr>
<tr>
<td></td>
<td>60004</td>
<td>56111</td>
<td>60</td>
<td>0.0045 – 0.0055</td>
<td>Natural</td>
</tr>
<tr>
<td></td>
<td>60005</td>
<td>56210</td>
<td>60</td>
<td>0.006 – 0.007</td>
<td>Natural</td>
</tr>
<tr>
<td></td>
<td>60005</td>
<td>56210</td>
<td>65</td>
<td>0.006 – 0.007</td>
<td>Very Low Porosity</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>MEK</th>
<th>MEK wipe only</th>
</tr>
</thead>
<tbody>
<tr>
<td>120G</td>
<td>Hand sanding with 120 grit</td>
</tr>
</tbody>
</table>

- Ny
  - Nylon peel ply
- Poly
  - Polyester peel ply
Evaluation of Peel Ply Removal and Exposure
D3165 - Single Lap Shear – FM300-2M

A – Immediately before bonding
B – Removed and surface exposed to 14 days.
C – Immediately before bonding; exposed to for to 14 days.
Evaluation of Peel Ply Removal and Exposure
D5528 - Mode I - FM300-2M

A - Immediately before bonding
B - Removed and surface exposed to 14 days.
C - Immediately before bonding; exposed to for to 14 days.

GIC (5% / MAX) [KJ/m2]

Average Water Contact Angle [degrees]

Adhesion Failure
Cohesion/First Ply Failure
Evaluation of Peel Ply Removal and Exposure
D3165 - Single Lap Shear – EA 9394

A - Immediately before bonding
B - Removed and surface exposed to 14 days.
C - Immediately before bonding; exposed to for 14 days.

Apparent Shear Strength [psi]

Average Water Contact Angle [degrees]
Evaluation of Peel Ply Removal and Exposure
D5528 - Mode I – EA9394

A – Immediately before bonding
B – Removed and surface exposed to 14 days.
C – Immediately before bonding; exposed for 14 days.

Adhesion Failure
Cohesion Failure
Evaluation of Peel Ply Removal – Multiple Cure Cycles
**FM300-2M - D3165 Single Lap Shear**

<table>
<thead>
<tr>
<th>Adhesive Bond</th>
<th>Apparent Shear Strength [psi]</th>
<th>Average Water Contact Angle [degrees]</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Adh</td>
<td>Ny-T1-PP-MCC1</td>
<td>100% Adh</td>
</tr>
<tr>
<td>100% Adh</td>
<td>Ny-T1-PP-MCC2</td>
<td>100% Adh</td>
</tr>
<tr>
<td>100% Adh</td>
<td>Poly-T1-PP-MCC1</td>
<td>21% LFT, 79% Adh</td>
</tr>
<tr>
<td>10% LFT, 90% Adh</td>
<td>Poly-T1-PP-MCC2</td>
<td>25% LFT, 75% Adh</td>
</tr>
<tr>
<td>8% LFT, 92% Adh</td>
<td>Poly-T1-PP-MCC3</td>
<td>8% LFT, 92% Adh</td>
</tr>
</tbody>
</table>

**Substrate** - T800/3900-2
**Adhesive** – FM300-2M
**Bond Cure Cycle** – 90 mins 250°F 40 psi
**Surface Preparation** – Peel Ply

**Adhesion Failure**
Low Cohesion/First Ply – High Adhesion
Evaluation of Peel Ply Removal – Multiple Cure Cycles
FM300-2M – D5528 Mode I

Substrate - T800/3900-2
Adhesive – FM300-2M
Bond Cure Cycle – 90 mins 250F 40 psi
Surface Preparation – Peel Ply

Adhesion Failure
Low Cohesion/First Ply – High Adhesion
Evaluation of Peel Ply Removal – Multiple Cure Cycles
Degree of Cure

<table>
<thead>
<tr>
<th>Material</th>
<th>Degree of Cure [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ny-T1-PP-MCC1</td>
<td>98 ± 0.5</td>
</tr>
<tr>
<td>Ny-T1-PP-MCC2</td>
<td>97 ± 0.5</td>
</tr>
<tr>
<td>Ny-T1-PPR-MCC1</td>
<td>98 ± 0.5</td>
</tr>
<tr>
<td>Ny-T1-PPR-MCC2</td>
<td>97 ± 0.5</td>
</tr>
<tr>
<td>Poly-T1-PP-MCC1</td>
<td>99 ± 0.5</td>
</tr>
<tr>
<td>Poly-T1-PP-MCC2</td>
<td>99 ± 0.5</td>
</tr>
<tr>
<td>Poly-T1-PPR-MCC1</td>
<td>99 ± 0.5</td>
</tr>
<tr>
<td>Poly-T1-PPR-MCC2</td>
<td>99 ± 0.5</td>
</tr>
</tbody>
</table>

Legend:
- Co - Initial cure
- C1 - 1 thermal cycle
- C2 - two thermal cycles
Evaluation of Mixing Method
Hand Mixing vs. Speed Mixer

• Hand mixing
  • Materials weighed into cup and mixed for 5 minutes. Mixture is then transferred to second cup and mix for an additional 5-10 minutes or until the consistency of the adhesive has changed to become smoother and easier to mix.

• Speed Mixer
  • Materials weighed into FlackTek compatible cup and placed inside machine with holder. An appropriate recipe (depending on weight) is chosen and the machine is run.

<table>
<thead>
<tr>
<th>Zone</th>
<th>RPM</th>
<th>Time (secs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1000</td>
<td>60</td>
</tr>
<tr>
<td>B</td>
<td>1600</td>
<td>40</td>
</tr>
<tr>
<td>C</td>
<td>2000</td>
<td>90</td>
</tr>
</tbody>
</table>

Recipe for 125g of adhesive
Evaluation of Mixing Method
Hand Mixing vs. Speed Mixer

Average Strength [psi]

HandMixed

Machine Mixed

Coefficient of Variation [%]

0%

5%

10%

15%
Bondline Control Mechanism

- Bondline control mechanisms available
  - Glass beads
  - Tracer Wires
- Evaluate the mix percentage for optimal bondline control
  - 0.0059-inch GB mixed at 0.05% and 0.1% by weight
  - 0.01-inch GB mixed at 0.05% and 0.1% by weight
- Effects of cure/pressure application
  - In Progress
- Effects on the mechanical properties
  - In Progress
Bondline Control Mechanism

![Chart showing the average bondline thickness and coefficient of variation for different conditions.](chart)

- Average Bondline Thickness [in]
- Coefficient of Variation [%]
Summary and Conclusion

• Quality assurance through water contact angle measurements for substrate provide reliable data to ensure the substrate preparation is acceptable.

• Surface preparation to bonding time assessment provide the state change substrates go through when exposed to environment. This can be used to fine tune the bond process.

• Technicians variability assessment is critical in understanding the sensitivity of some of the manual labor critical activities. Understanding the variability is critical to ensure proper training is provided.

• Exposure duration and configuration in peel ply removal technique show variation in the bond performance. Investigations are currently underway to evaluate the reason for the PP removed exposed substrate showed slightly higher properties.

• Polyester peel ply showed a change in the strength and failure mode when exposed to thermal cycles.
Looking Forward/Future Work

• Future Works
  • Generate bond process protocols for
    • Selecting compatible substrate and adhesive combinations for a robust bond structure
    • Provide guidance on protocol development for cure process related activities
    • Look into other surface preparation methods and look into critical parameters

• 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 to ensure the integrity of the final bonded product