Effect of Surface Contamination on Composite Bond Integrity and Durability

Gabriela Gutierrez-Duran & Dwayne McDaniel
Effect of Surface Contamination on Composite Bond Integrity and Durability

• Motivation and Key Issues
  – Past research has focused on determining/understanding acceptable performance criteria using the initial bond strength of composite bonded systems.
  – There is significant interest in assessing the durability of composite bonded joints and the how durability is affected by contamination.

• Objective
  – Develop a process to evaluate the durability of adhesively bonded composite joints
  – Investigate undesirable bonding conditions by creating scalable and repeatable weak bonds.
  – Investigate a means to mitigate the undesirable conditions via surface preparation methods.
  – Support CMH-17 with the inclusion of content for bonded systems
Effect of Surface Contamination on Composite Bond Integrity and Durability

- Principal Investigators
  - Dwayne McDaniel, Ben Boesl

- Students
  - Gabriela Gutierrez-Duran, Brian Hernandez, Julie Dubon, Mauricio Pajon

- FAA Technical Monitor
  - Ahmet Oztekin

- Industry Participation
  - Exponent, 3M, Embraer, BTG Labs
Manufacturing of Bonded Systems

**KEY QUESTION**
What happens to bonded joint’s strength when contamination occurs, if known can it be mitigated?

**CAUSES**
Contamination can occur in a manufacturing setting from oil on hands, mold release, leakage/spillage, etc.

- Fabrication of Laminates
- Laminate Cure (Cure Cycle @350F)
- Preparing/Cutting Samples
- Adhesive Cure
- Adhesive Bond Strength Testing
- Bonding of Laminates

(Cure Cycle @350F)
Contamination Approach

GOAL - Develop a process to create a scalable and repeatable weak bond via bondline contamination.

Contaminant – Frekote release agent

• Developed a station that can uniformly spray contaminant – vary nozzle size and spray rates
• Potential for creating a scalable weak bond by adjusting concentration of Frekote
• Total amount of contaminate applied is measured using an analysis of pre- and post- weight measurement.
Bond Quality Evaluation

- Dual Cantilever Beam Testing
  - Measures interlaminar fracture toughness
- Fracture toughness provides a measure of composite strength
  - The critical energy a material may absorb before failure and resistance to delamination
    - $G_{1C} = \frac{3P\delta}{2b(a+\Delta)}$
- Use of MTS machine to measure displacement
Materials

- **Material type and curing procedure for specimens:**
  Unidirectional carbon-epoxy system, film adhesive, secondary curing bonding and contaminants.

- **Materials utilized:**
  - Toray P 2362W-19U-304 T800 Unidirectional Prepreg System (350F cure)
  - 3M AF 555 Structural adhesive film (7.5x2 mills, 350F cure)
  - Precision Fabric polyester peel ply 60001
  - Frekote 700-NC from Henkel Corporation
Calibration of Contamination Levels

- Calibration of the contamination levels is important in order to be able to trace back the amount of contaminant used and relate that amount to the strength of the weak bond created
  - This enables us to determine the different bond strengths that can be created from different amounts of contaminant

- Adjusting spray speeds and mass measurements of the contaminant on a 1” x 1” section of a panel, allows for the determination of the strength of the weak bond

- Procedures
  - Modify the spray speed according to the amount of mass desired
    - Fast speeds: less mass
    - Slow speeds: more mass
  - Weigh a 1” x 1” section of a panel before spraying contaminant
  - Spray contaminant and weigh it again
  - Continue process until desired mass is reached
Mitigation Procedures

• **GOAL** - Develop a process to mitigate the influence of contamination of the bondline

• Two methods of mitigation
  
  – *Solvent Wipe* -
    Attempt to remove contaminate off of surface with soaked cloth
  
  – *Sanding of Material* -
    Actively remove material using abrasive
Results of Mitigation Approaches

<table>
<thead>
<tr>
<th></th>
<th>Baseline</th>
<th>Cont</th>
<th>Wire</th>
<th>WSW</th>
</tr>
</thead>
<tbody>
<tr>
<td>78%</td>
<td>1.030</td>
<td>0.800</td>
<td>0.673</td>
<td>1.050</td>
</tr>
</tbody>
</table>

$G_C (kJ/m^2)$
Mitigation Results

<table>
<thead>
<tr>
<th>Percentage</th>
<th>Base line</th>
<th>Cont</th>
<th>Wine</th>
<th>WSW</th>
</tr>
</thead>
<tbody>
<tr>
<td>19 %</td>
<td>1.270</td>
<td>0.238</td>
<td>0.221</td>
<td>0.609</td>
</tr>
<tr>
<td>42 %</td>
<td>1.320</td>
<td>0.554</td>
<td>0.575</td>
<td>0.869</td>
</tr>
<tr>
<td>78 %</td>
<td>1.030</td>
<td>0.800</td>
<td>0.673</td>
<td>1.050</td>
</tr>
</tbody>
</table>
Failure Modes – 19%

**Mixed-mode failure**
Variable combination of interlaminar and cohesion

**Adhesion failure**
Separates from the surface of adherent

Baseline
19% Wipe/Sand/Wipe
19% Only
19% Wipe

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13
Failure Modes – 42%

Mixed-mode failure
Variable combination of interlaminar and cohesion

Baseline

42% Wipe/Sand/Wipe

42% Only

42% Wipe

Adhesion failure
Separates from the surface of adherent

CECAM
JAMS
AMTAS
Failure Modes – 78%

Mixed-mode failure
Variable combination of interlaminar and cohesion

Adhesion failure
Separates from the surface of adherent

Baseline
78% Wipe/Sand/Wipe
78% Only
78% Wipe
In-situ Testing
Combined Load Frame and Electron Microscopy

Test Development

\( \mu \text{DCB (Dual Cantilever Beam)} \)
Assess the mechanisms of mode I fracture. Fixture was designed based on literature of metal-adhesive bond testing.

\( \mu \text{ENF (End Notch Flexure)} \)
Assesses the mechanisms of mode II fracture. Fixture was designed based of traditional ENF testing of composite bonds.
In-situ Testing
Combined Load Frame and Electron Microscopy

Specimen Details

Baseline
L/W: 40mm x 10mm
thickness: 5.2 mm
Pre-crack: 8 mm
10 layer unidirectional composite panels

Observations
- Initially bond is very stiff
- Controlled crack propagation begins at ~50N Load
- Unstable crack growth begins at the pre-crack then travels to composite-adhesive interface
In-situ Testing
Combined Load Frame and Electron Microscopy

Specimen Details

Contaminated
L/W: 40mm x 10mm
thickness: 5.2 mm
Pre-crack: 8 mm
4% contamination procedure was used at the interface

Observations

• Initial delamination between adhesive and composite panel
• High compliance during loading, reduction in peak load
• Unstable crack growth begins at the interface and pre-crack remains un-damaged
In-situ Testing
Combined Load Frame and Electron Microscopy

Complications with in situ testing
Small sample sizes and edge effects
Sample testing environment

At the moment, testing can be used to study mechanisms but not to quantify fracture properties

From Linear Elastic Fracture Mechanics theory we know the stress field very near the crack tip and from that we can solve for the displacement at any point if \( K_I \) is known.

Therefore if we know the displacements we can solve for the \( K_I \) value.
In-situ Testing
Combined Load Frame and Electron Microscopy

In situ Microscopy  Digital Image Correlation  Digitized Displacements

\[ u_x = \frac{K_I}{8\mu \pi} \sqrt{2\pi r} \left[ (2\kappa - 1) \cos \frac{\theta}{2} - \cos \frac{3\theta}{2} \right] \]
\[ u_y = \frac{K_I}{8\mu \pi} \sqrt{2\pi r} \left[ (2\kappa + 1) \sin \frac{\theta}{2} - \sin \frac{3\theta}{2} \right] \]

\[ \mu \text{DCB } G_{IC} = 0.58 \text{ kJ/m}^2 \]
\[ \text{DCB } G_{IC} = \sim 1.00 \text{ kJ/m}^2 \]
CMH-17 Support

Background and Motivation

- A Strategic Composite Plan has been developed by the FAA and has identified three focus areas regarding safety, certification and education. Within these areas, there are a number of initiatives related to structural issues and adhesive bonding.

- As part of the FAA’s bonding initiatives, the CMH-17 handbook is supporting the development of content related to bonding design and process guidelines.

Mission Statement

The Composite Materials Handbook organization creates, publishes and maintains proven, reliable engineering information and standards, subjected to thorough technical review, to support the development and use of composite materials and structures.
CMH-17 Bonding Process Task Group

Need for bonding process content in CMH

The Promise of Bonded Composites
lighter weight, monolithic structures
designed with fewer parts and
assembled with reduced
manufacturing costs (in terms of
time and labor)

The Reality of Bonded Composites
bonded parts that are bolted for
confidence, adhesives asked to act as
environment seals, challenges of
process control to capture and
quantify variability

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
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<tbody>
<tr>
<td>Bonded Joints</td>
<td>Limits to thickness that can be joined with simple joint configuration; Inspection other than for gross flaws difficult; Prone to environmental degradation; Sensitive to peel and through-thickness stresses; Residual stress problems when joining to metals; Cannot be disassembled; May require costly tooling and facilities; Requires high degree of quality control; May be of environmental concern</td>
</tr>
<tr>
<td>Small stress concentration in adherends; stiff connection; Excellent fatigue properties; No fretting problems; Sealed against corrosion; Smooth surface contour; Relatively lightweight; Damage tolerant</td>
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<table>
<thead>
<tr>
<th>Bolted Joints</th>
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<tbody>
<tr>
<td>Positive connection, low initial risk; Can be disassembled; No thickness limitations; Simple joint configuration; Simple manufacturing process; Simple inspection procedure; Not environmentally sensitive; Provides through-thickness reinforcement; Not sensitive to peel stresses; No major residual stress problem</td>
<td></td>
</tr>
<tr>
<td>Considerable stress concentration Prone to fatigue cracking in metallic component; Hole formation can damage composite; Composites's relatively poor bearing properties; Prone to fretting in metal; Prone to corrosion in metal; May require extensive shimming</td>
<td></td>
</tr>
</tbody>
</table>
Executive Summary

An outline for composite bonding processes was created and circulated for approval. The CMH-17 Bonding Process Task Group used the outline as a framework to create an online forum to capture, organize, and edit relevant content. The content in the online forum will be converted into draft for circulation, editing, and approval.

Bonding Process Task Group Leadership
Dwayne McDaniel     FIU
Tanila Faria        Embraer
Tim Barry           BTG Labs
Dan Ruffner         Emeritus
Howard Creel        3M

Bonding Process Task Group Champions
Curt Davies         FAA
Rachael Andrulonis  CMH-17

Bonding Process Task Group Steering
Nathan Weigand FAA
Bill Nickerson Navy
Michelle Johnson LMCO

Special Thanks to Founding Members
Holly Thomas, Margaret Roylance, Dan Ruffner, Scott Leemans, Carl Rousseau
5.9 Assembly Processes

5.9.1 Fastened Joints

5.9.2 Bonded Joints

5.9 ASSEMBLY PROCESSES

Assembly processes are not conventionally covered within composite material characterization, but can have a profound influence on the properties obtained in service. As seen with test coupons, edge and hole quality can dramatically affect the results obtained. While these effects are not usually covered as material properties, it should be noted that there is an engineering trade off between part performance and the time and effort expended toward edge and hole quality. These effects need to be considered along with the base material properties.
CMH17 Volume 3: Materials Usage, Design and Analysis
Chapter 5  Materials and Processes - The Effects of Variability on Composite Properties
Proposal for New Section in Revision H

5.9 Assembly Processes

5.9.1 Assembly for Bonded Joints
The section covers the process considerations for assembling bonded thermoset composite joints. It represents
guidelines drawn from best available knowledge and is not to be used for specification or certification purposes.
It is organized to provide the details of the process of secondary bonding, special considerations and advantages
of co-curing, and co-bonding processes and considerations for multi-step bond fabrication. The section is focused
on load bearing bonds and not on sealants or other adhesive or bonding systems.

5.9.1.1 Introduction
5.9.1.2 General Considerations
  • Types of Bonds
  • Definitions
5.9.1.3 Secondary Bonding
  • General Consideration
  • Quality considerations for bonding
  • Surface Preparation
  • Protecting the Prepared Surface
  • Adhesive Application
  • Bond Assembly
  • Adhesive Cure
  • Bond Inspection
5.9.1.4 Co-curing
  • Advantages
  • Special Considerations
5.9.1.5 Co-bonding
  • Advantages
  • Special Considerations
5.9.1.6 Multi-Stage Bonding
5.9.1.7 References

5.9.2 Assembly for Bolted Joints
5.9.3 Assembly for Hybrid Joints

Five Working Groups Formed for Bonded Joints
1. General Considerations  Creel, 3M
2. Surfaces  Faria, Embraer
3. Adhesives and Processing  Creel, 3M
4. Inspection, Testing, Quality  McDaniel, FIU
5. Co-cure, Co-bond, Multi-stage  TBD
CMH-17 Support

Using online forums to organize CMH-17 content
Summary

• A contamination procedure was developed using and Frekote to develop a scalable and repeatable weak bond. The weak bonds can be used to evaluate surface prep techniques and potentially NDI methods.

• Repeatable weakened bonds were obtained using a customized contamination rig for three levels of contamination (~20, 40 and 80% bond strength).

• Mitigation approaches included solvent wiping and solvent wiping/sanding/solvent wiping. Results from these tests indicted that wiping alone did not improve the bond strength, however, there was significant improvement with the wiping/sanding/solvent wiping.


• An on-line procedure was developed to provide a means for the bonding community to submit content to the CMH-17 handbook. The first draft of the surface prep section was assembled is currently being reviewed.
Path Forward

• Contaminated DCB coupons and coupons treated with the mitigation methods will be placed in an environmental chamber to determine the effects of contamination on environmental durability.

• Contaminated and treated DCB coupons will be fatigued in a hydraulic fatigue rig that can cyclically load specimens in shear via three point bending. After the specimens have been aged, effects of fatigue on the contaminated specimens will be evaluated.

• Mini-DCB coupons will be developed and tested in the SEM to provide a understanding of the modes of failure. Aspects that can be evaluated include, environmental exposure, contamination and bondline thickness. Efforts will also be made to quantity the fracture toughness using DIC to estimate the strain field around the crack tip.

• Content on bond testing and quality as well as materials will be assembled, organized and submitted for review for CMH-17.
Questions ?