



Effect of Surface Contamination on Composite Bond Integrity and Durability

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Effect of Surface Contamination on Composite Bond Integrity and Durability

- **Motivation and Key Issues**

- A number of issues can lead to reduced reliability of adhesively bonded composite systems.
 - Material compatibility, surface preparation, manufacturing, contamination
- Different levels of contamination on laminates surface prior to bonding can lead to varying levels of bond performance. In some cases, small levels may not effect initial bond strength.
- Understanding how well typical approaches in surface preparation can address contamination will provide valuable information regarding acceptable tolerances.

- **Objective**

- Develop a process to create a scalable and repeatable weak bond via contamination. The weak bond can be used to assess the effectiveness of surface preparation methods including sanding and solvent wiping.
- Validate the methods for both initial and long term strength.
- 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**
 - Shervin Tashakori, Daniella Gil

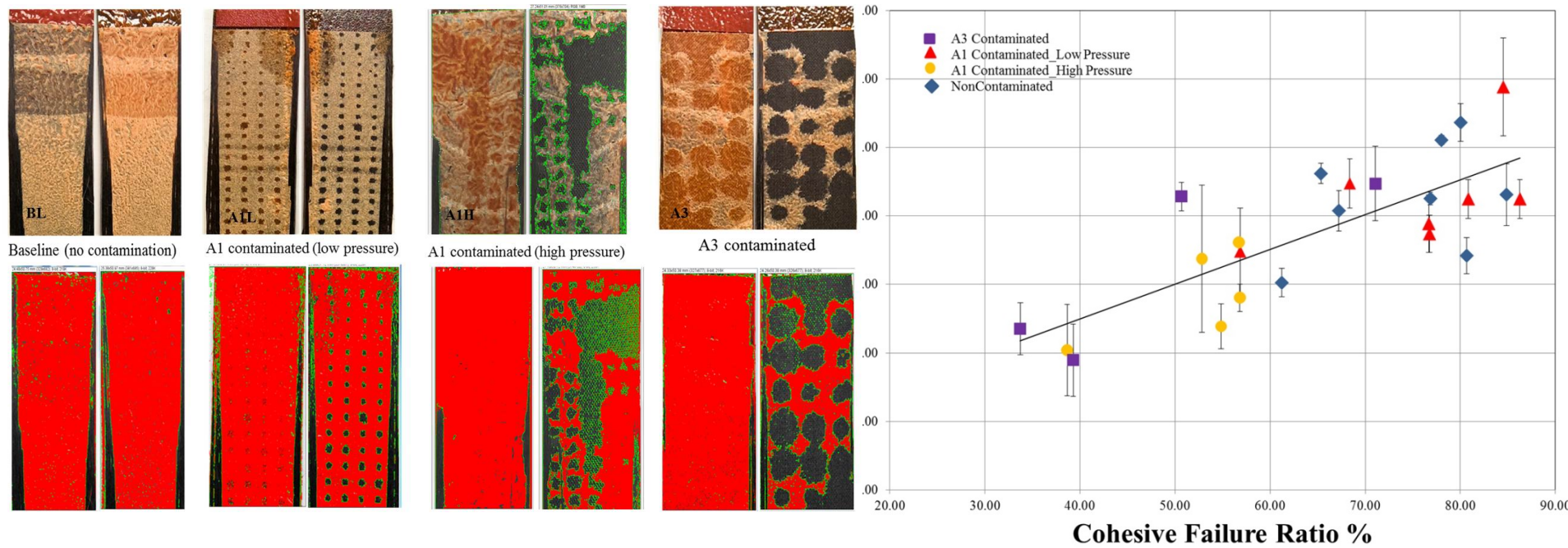
- **FAA Technical Monitor**
 - Curt Davies, David Westlund

- **Industry Participation**
 - Exponent, 3M, Embraer, Boeing

Previous Contamination Efforts

Contamination – Ordered Array

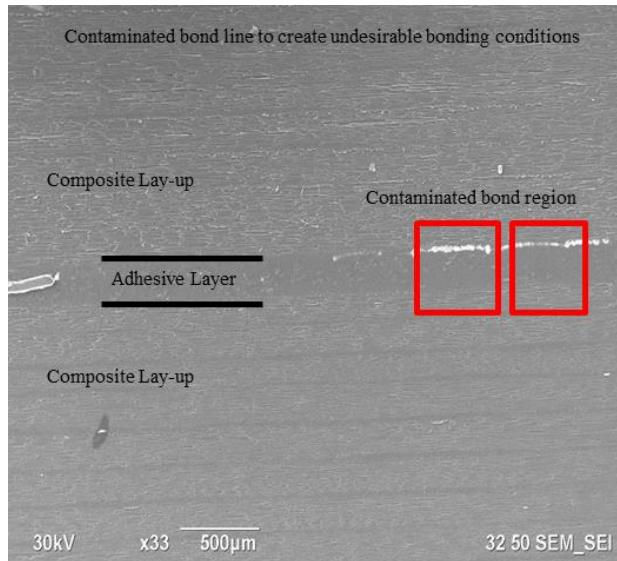
Create scaled bond strength – vary contamination size



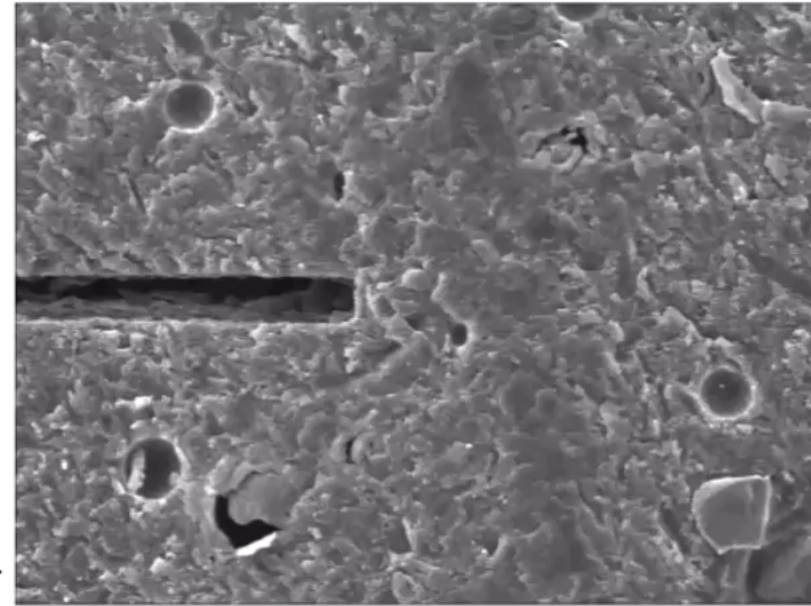
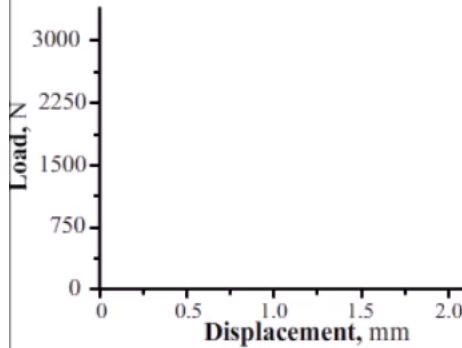
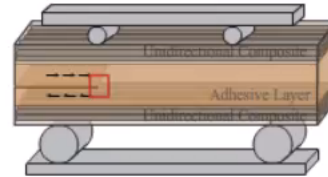
Previous Contamination Efforts

Micro Scale Testing

In-situ Electron Microscopy - End Notch Flexure Tests



Testing Details: End Notch Flexure
 Displacement Control: 0.5 mm/min
 Sample: Adhesive-MENS (1 vol. %)



0 30 60 90 120 150 180 210 Time, s

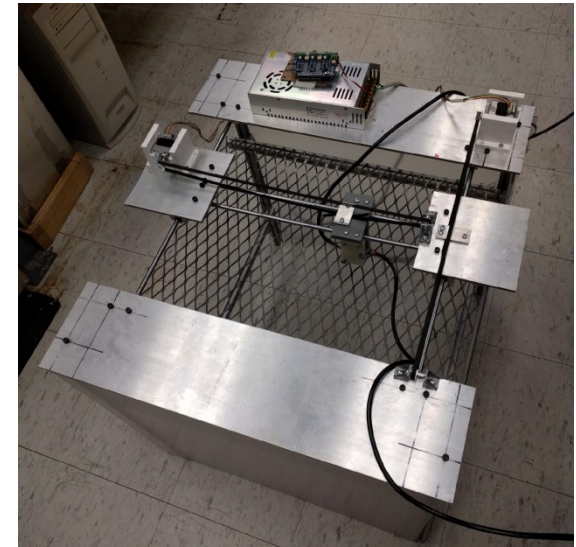
Uniform Contamination

- Feedback – interested in larger scale contamination areas
- uniformly distributed contamination to create weak bonds

Initial Uniform Contamination Approach

Contaminant – Frekote release agent (Siloxane)

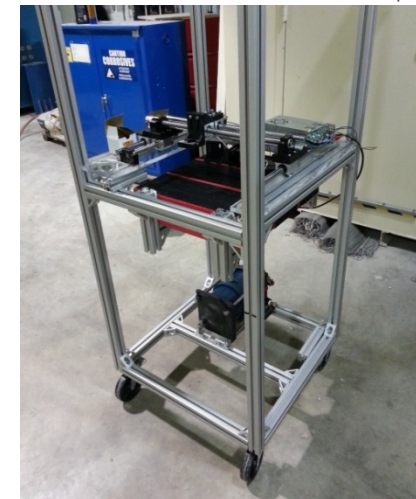
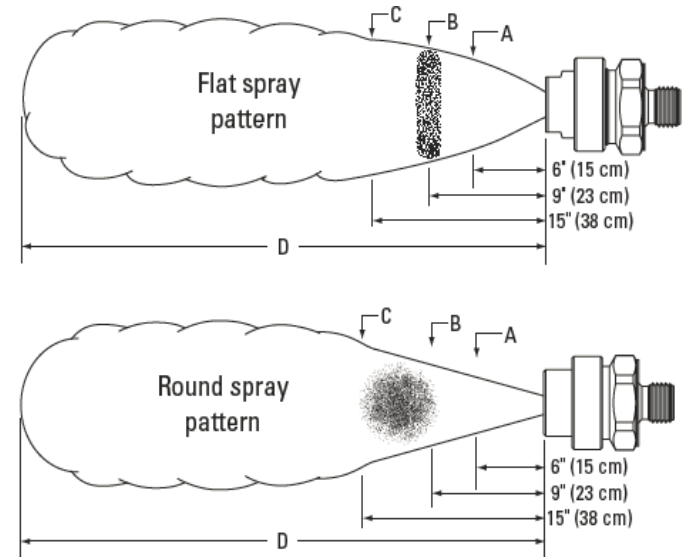
- Uniform spraying of contaminant comprised of Frekote and Hexane at various concentration levels.
- Evaporate Hexane – leaving various levels of Frekote on laminate surface prior to bonding.
- Potential for creating a scalable weak bond – by adjusting the concentration of Frekote
- Developed a station that can uniformly apply the contaminant – vary nozzle size and spray rates.
- System can allow for a variety of contaminants



Uniform Contamination

Initial Uniform Contamination Approach

- Evaluate nozzle size, nozzle head speed, nozzle head height and fluid pressure
- Manufacture panels
- Contaminate panels with solutions of Hexane and Frekote
 - 4 contamination levels – 25% 50% 75% and 100% Hexane (75% 50% 25% and 0% Frekote)
- Bond baseline panels with contaminated panels
- Manufacture DCB specimens – 4 specimens for each set



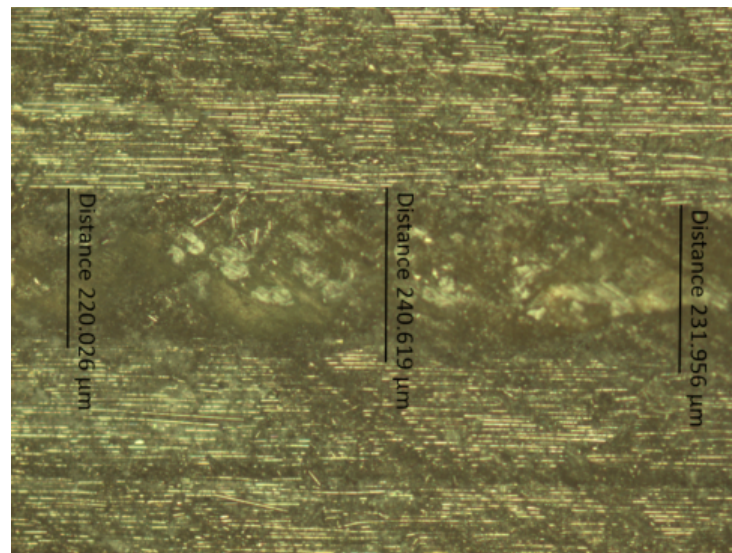
Bonding System Materials

- Material type and curing procedure for specimens: unidirectional carbon-epoxy system, film adhesive, secondary curing.

- 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
 - Freekote 700-NC from Henkel Corporation

Testing Results

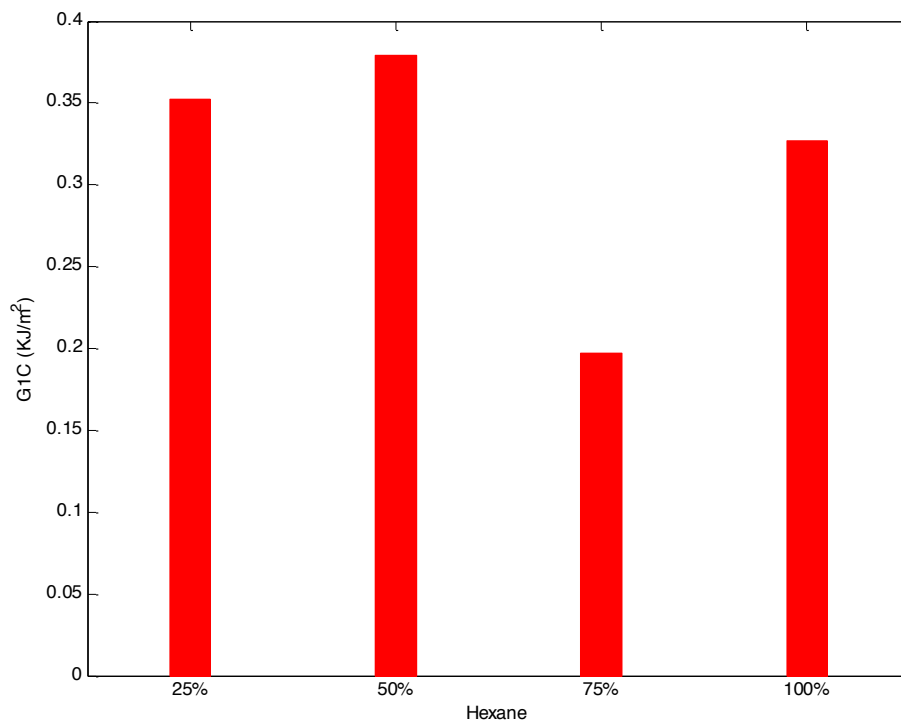
Bondline Measurement (um)				
Hexane percentage	25%	50%	75%	100%
Average for each panel	338.4	253.9	222.8	260.4
Total average	268.9			



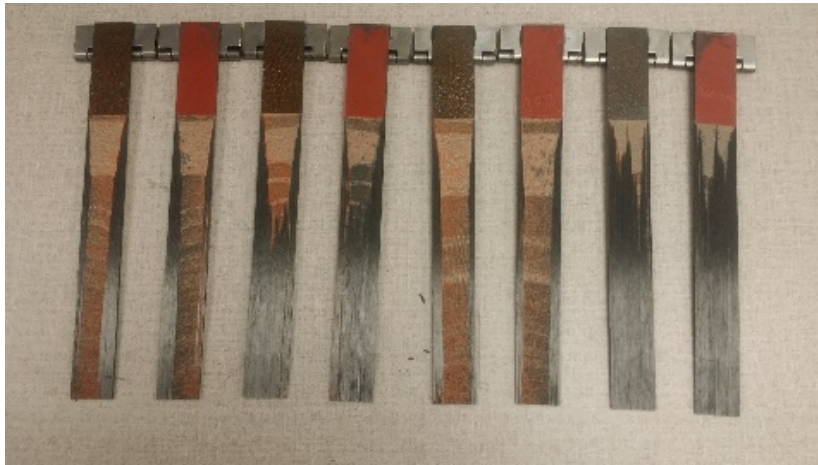
Testing Results

DCB Testing

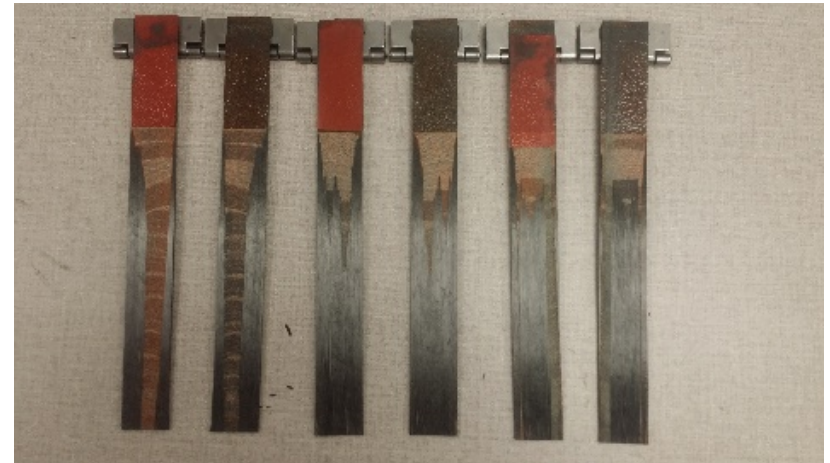
Hexane	25%				50%				75%				100%			
G_{1C} for each coupon	0.333	0.352	0.332	0.392	0.310	0.372	0.368	0.465	0.147	0.380	0.159	0.102	0.289	0.301	0.348	0.370
Average G_{1C}	0.352				0.379				0.197				0.327			



Modes of Failure



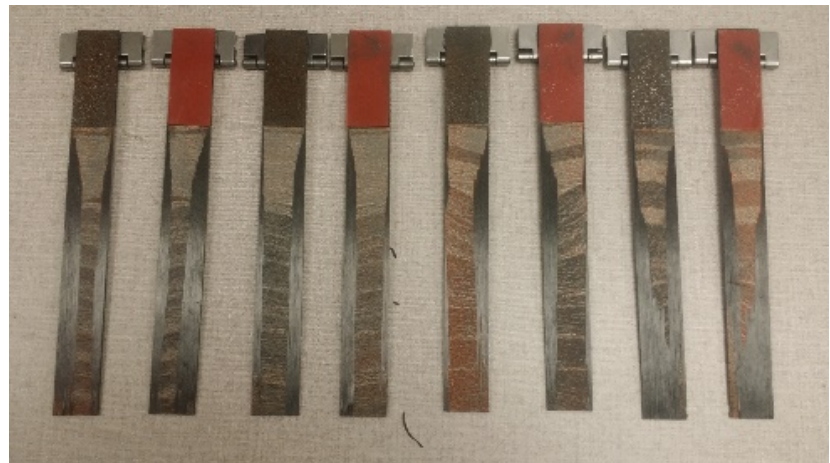
25% Hexane, 75% Frekote



50% Hexane, 50% Frekote



75% Hexane, 25% Frekote



100% Hexane

Hexane Evaporation Test

100% Hexane	
Time of measurement	Weight of 1 by 1 inch composite panel (mg)
Before spraying	1667.095
Immediately after spraying	1667.451
1 min	1667.365
5 min	1667.290
15 min	1667.253
30 min	1667.235
21 hours and 30 min	1667.212
45 hours and 30 min	1667.200

Alternative Uniform Contamination Approach

Direct Contamination Approach

- Due to mixed results and effects of Hexane – sought an alternative approach
- Contaminate panels directly, to determine if amount of Frekote can be varied/controlled to reduce bond strength
- Vary spray head speed to control amount of Frekote
 - Initially try slower speeds to find a lower level of bond strength
 - Subsequent trials will use faster head speeds
- Spray 1x1 inch coupon and compare mass changes
- Repeat bondline measurements and DCB tests



Quality Measurements

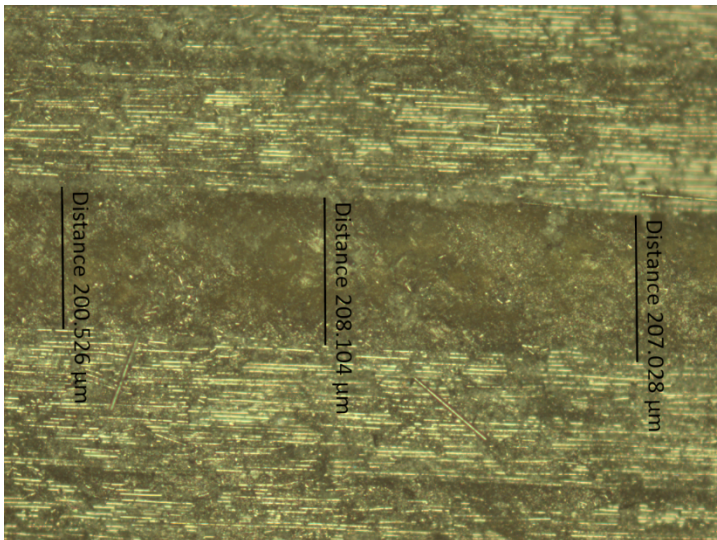
Bondline Measurement

Panel	Baseline	100% Frekote
Average for each panel	228.07	201.20
Total average	214.63	

Gravimetric Analysis

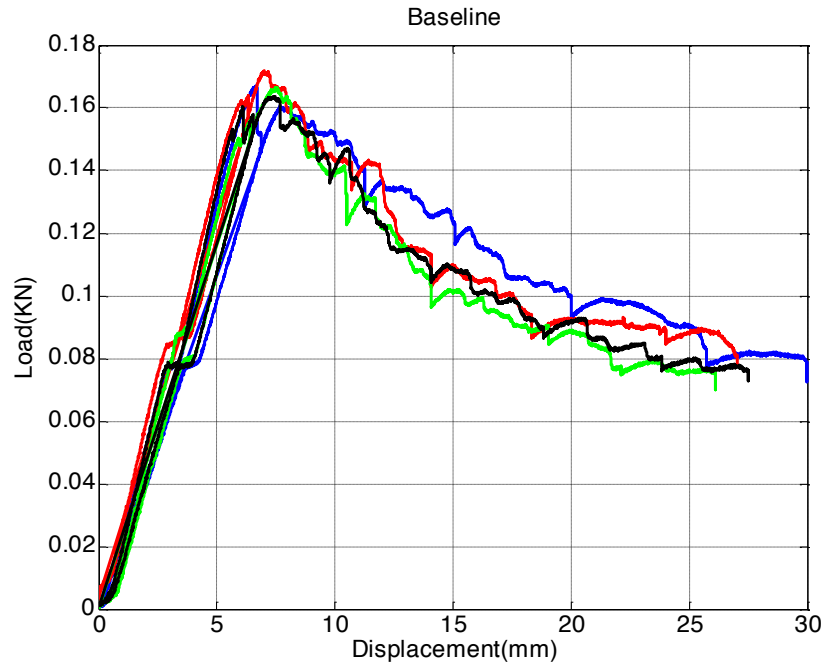
Weight of 1 x 1 Inch Composite Panel (mg)	
Baseline	100% Frekote
1662.040	1662.265

We will compare mass change obtained with additional nozzle speeds

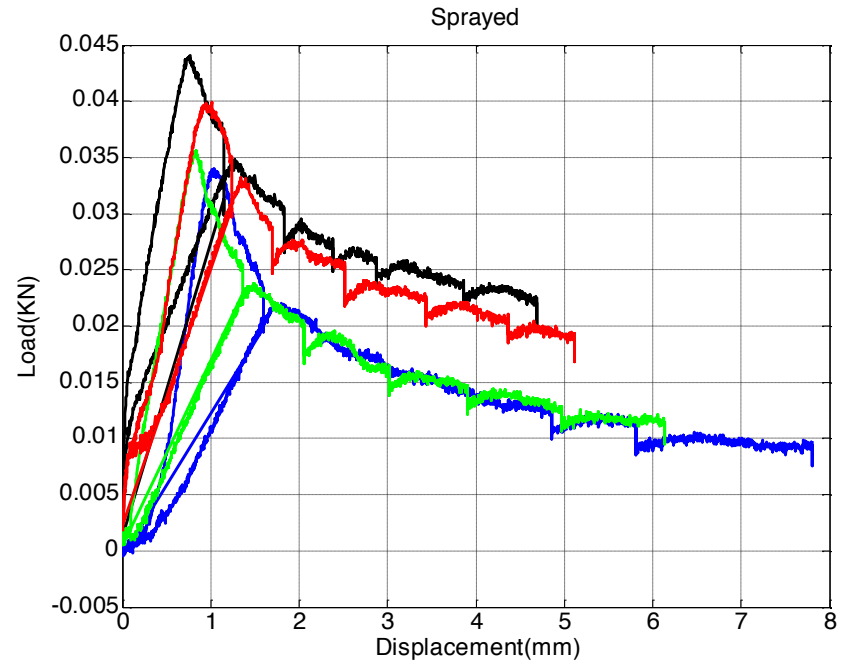


DCB Results

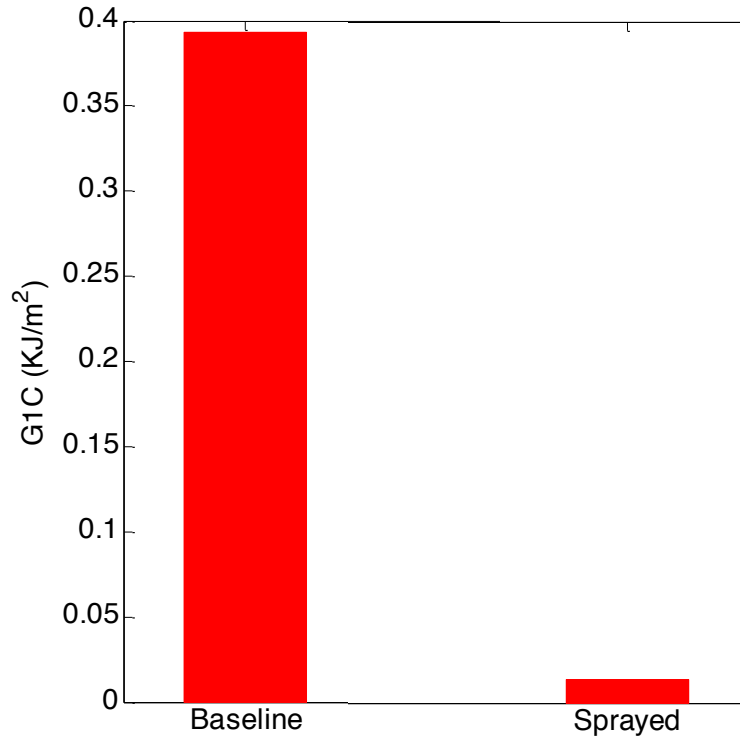
Baseline



Sprayed 100% Frekote

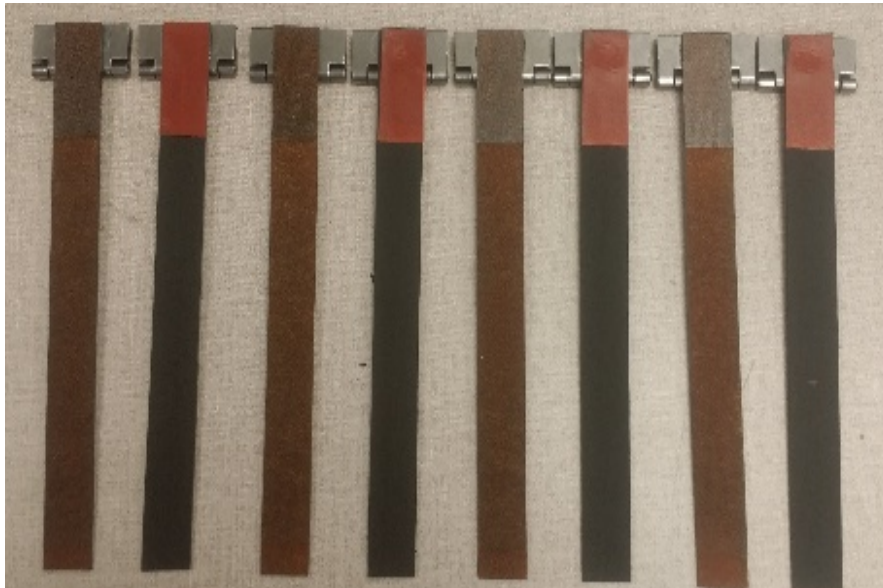


DCB Results

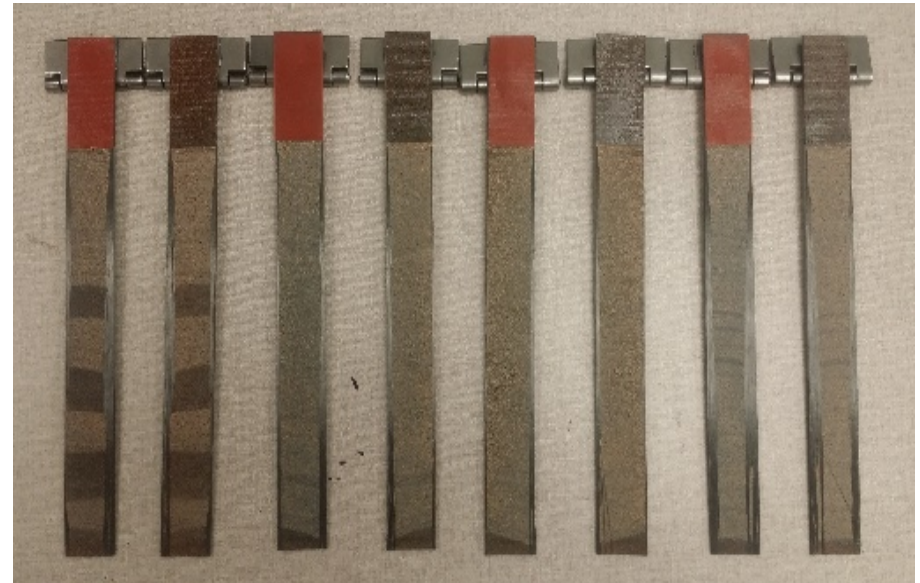


Hexane	Baseline				Sprayed			
G_{1C} for each coupon	0.412	0.407	0.371	0.384	0.0133	0.0154	0.0112	0.0165
Average G_{1C}	0.394				0.0141			

Modes of Failure



100% Frekote



Baseline

Conclusions and Path Forward

- A contamination procedure was evaluated using a solution of Hexane 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.
- Results were inconsistent, yielding unpredictable G_{1c} s. Failure modes were primarily mixed – interlaminar and cohesion.
- A direct approach was alternatively used and a baseline nozzle speed for contamination was established that provides a lower bound for bond strength.
- Testing will continue – varying the speed of the nozzle to change the level of contamination.
- Fracture toughness of the varying sets will be compared to establish correlations with contamination levels.
- Methods to remove the contamination will then be evaluated via sanding and solvent wiping. Specimens will be evaluated for their initial bond strength as well as their long term durability (aged in environmental chamber prior to fracture tests).
- ENF contaminated specimens will be manufactured and loaded in the SEM to evaluate the failure in real time.

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.

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 |

Expectations for August 2016 CMH-17 Meeting

1. Two working sessions by phone
2. Optimized outline for content
3. Identification of key sources of information
4. Map of existing content in CMH
5. Gap analysis

Volunteers for Bonded Joint Working Groups needed!

Also: Leadership for Bolted Joint Content

CMH-17 Support

- Map of existing content bonding content was provided to Curt Davies identifying relevant sections in the current handbook. Presentation was given at August CMH-17 meeting.
- Discussions were focused on updating Ch 3 Section 5.9 - path forward will depend on the amount of content developed.
- Individual groups are forming their teams
- Inspection, Testing and Quality – first objective is to update the bond inspection section of 5.9
 - Dwayne McDaniel, FIU
 - Ray Kaiser, Delta
 - Joe Rakow, Exponent
 - Marcio Donadon, ITA
 - Chuck Zhang, Georgia Tech
 - Others ...

Effect of Surface Contamination on Composite Bond Integrity and Durability

Questions?

Expanded Vision: New Volume focused on Bonding

CMH-17 Volume 7 Bonding of Thermoset Composite Structures

1. General Information
2. Material Data
3. Guidelines for Property Testing
4. Design and Analysis of Bonded Joints
5. Assembly Processes
6. Quality Control
7. Supportability
8. References

Current Vol 3 Section 5.9 becomes Vol 7 Section 5 with simultaneous optimization to Volume 3 Rev H

- Volume 3 Section 5.9.1 points to Volume 7
- New content for bolted and hybrid joints?
- Many revisions under development
- Relevant adhesive and join content moved to Volume 7 with pointers from Volume 3

By CMH-17 Meeting in August:

- General Outline for discussion and comment
- Existing content map with proposed changes to Vol 3
- Gap analysis

5 Assembly Processes

5.1 Introduction

5.2 General Considerations

5.2.1 Types of Bonds

5.2.2 Definitions

5.3 Secondary Bonding

5.3.1 General Considerations

5.3.2 Quality Considerations for Bonding

5.3.3 Surface Preparation

5.3.4 Protecting the Prepared Surface

5.3.5 Adhesive Application

5.3.6 Bond Assembly

5.3.7 Adhesive Cure

5.3.8 Bond Inspection

5.4 Co-curing

5.4.1 Advantages

5.4.2 Special Considerations

5.5 Co-bonding

5.5.1 Advantages

5.5.2 Special Considerations

5.6 Multi-Stage Bonding

5.7 References