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Effect of Surface Contamination on Composite Bond Integrity and Durability

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Florida International University

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 effected 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**
  - Dillon Watring, Shervin Tashakori

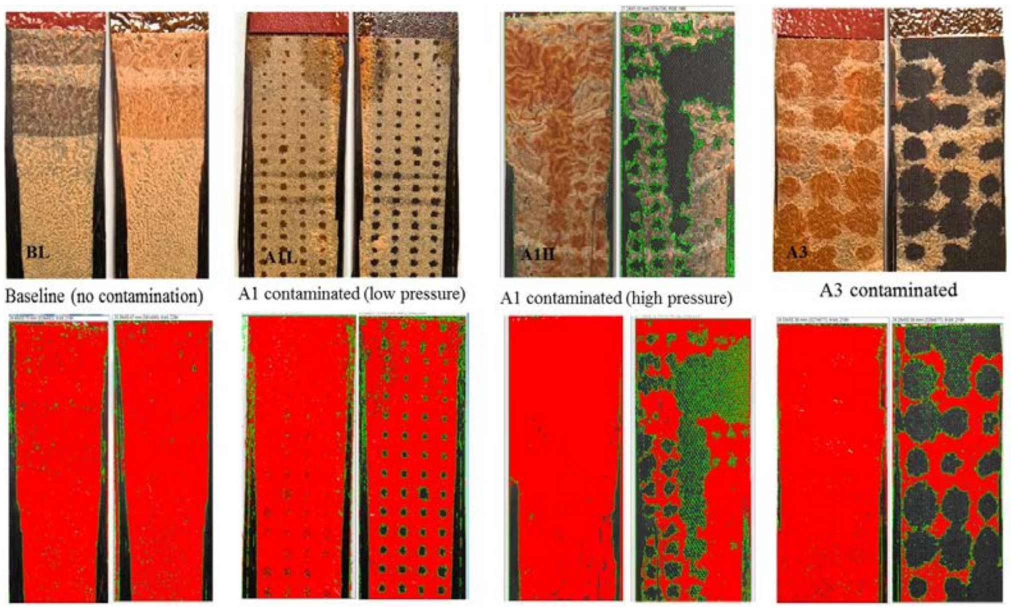
- **FAA Technical Monitor**
  - Curt Davies, David Westlund

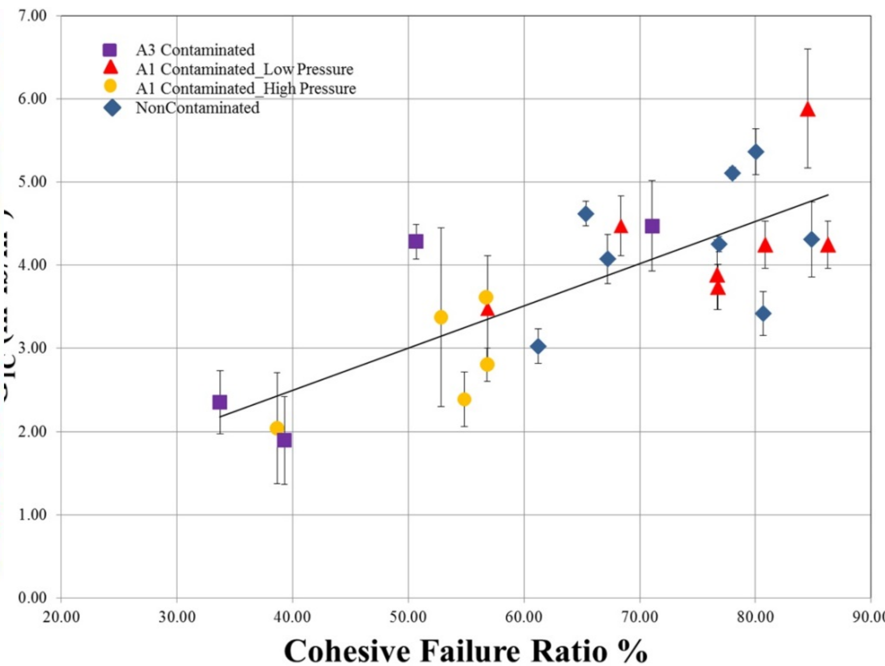
- **Industry Participation**
  - Exponent, 3M, Embraer, Boeing

Previous Contamination Efforts

Contamination – Ordered Array

Create scaled bond strength – vary contamination size

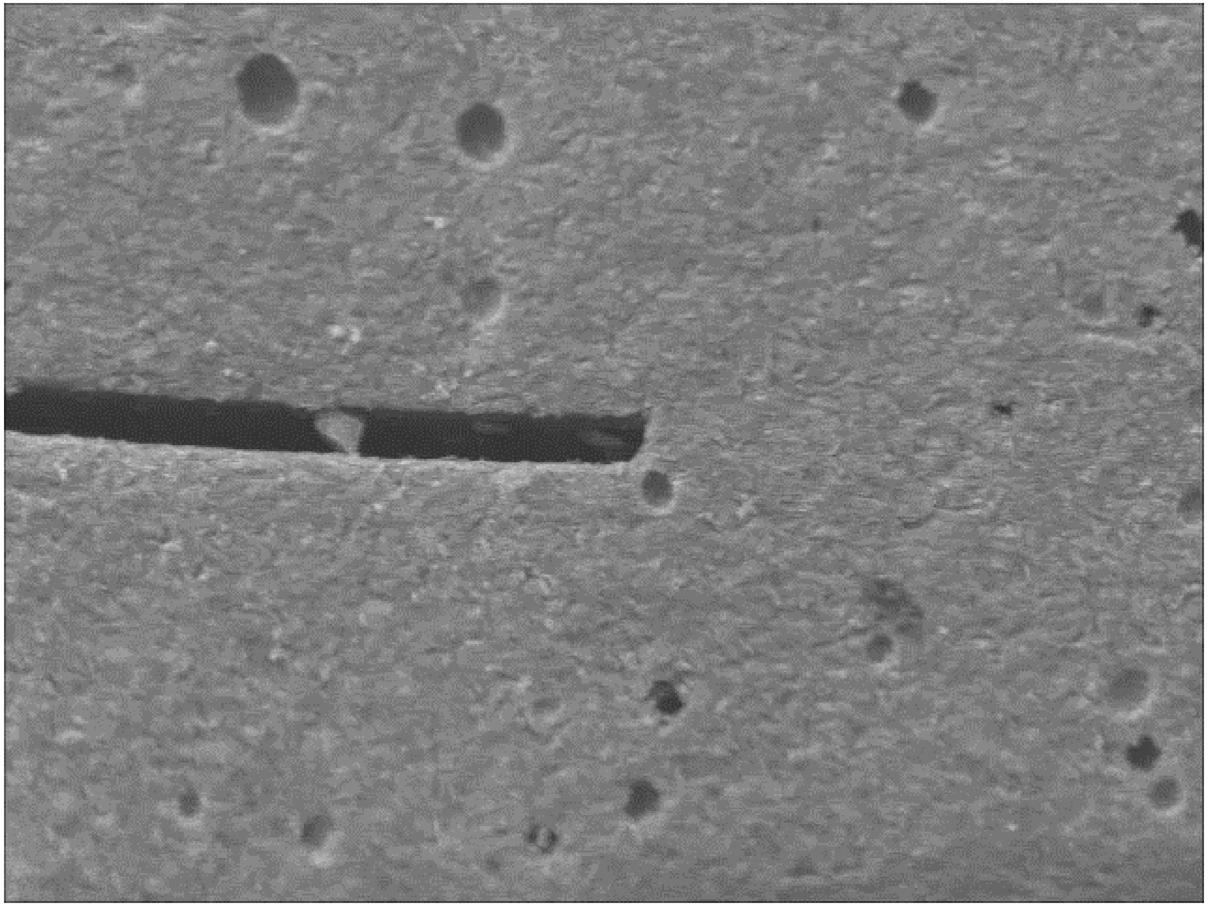




In-situ Testing

Micro Scale Testing

In-situ Electron Microscopy - End Notch Flexure Tests



In-situ Testing

Mini-DCB Testing

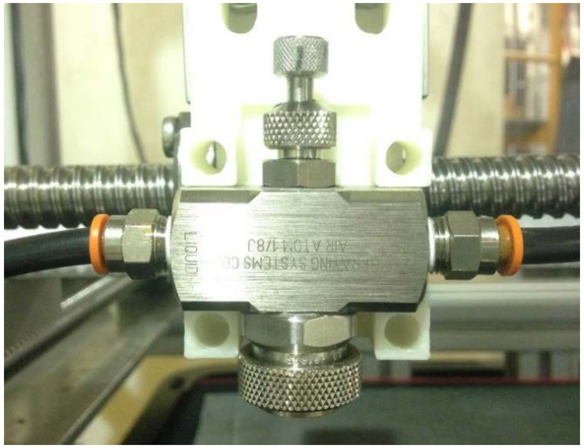
Uniform Contamination

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

Initial Uniform Contamination Approach

Contaminant –Frekote release agent (Siloxane)

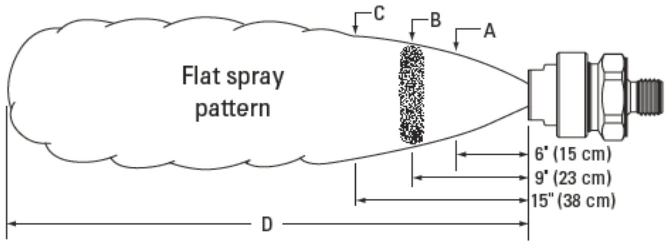
- Developed a station that can uniformly apply the contaminant – vary nozzle size and spray rates.
- System can allow for a variety of contaminants
- Uniform spraying of contaminant comprised of Frekote and Hexane at various concentration levels.
- Evaporate Hexane – leaving various levels of Frekote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

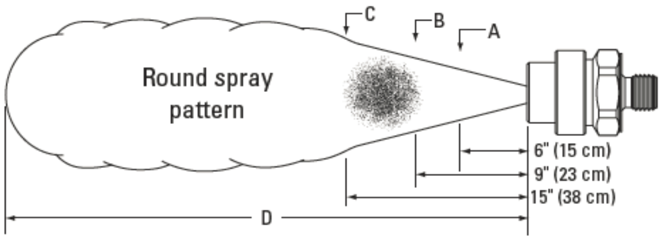


Uniform Contamination

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 pristine panels with contaminated panels
- Manufacture DCB specimens – 4 specimens for each set





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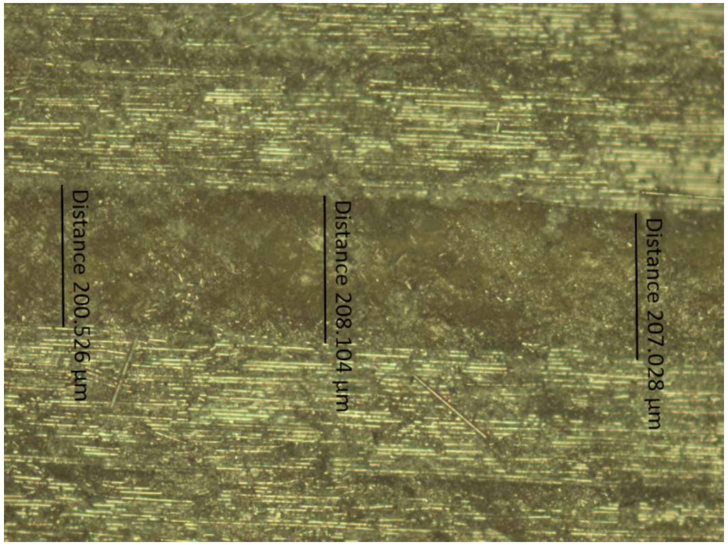
Materials

- 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 Systems (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

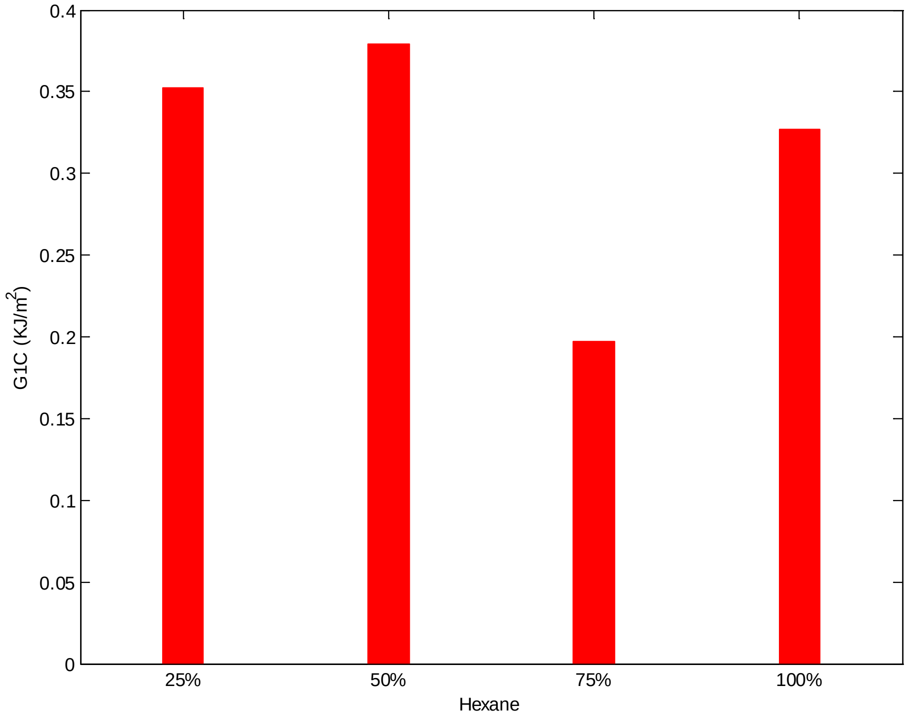
Quality Control

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			



DCB Results

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			



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

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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 and height 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

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Quality Measurements

Initial specimens prepared – baseline and contaminated

- Contaminated specimens created with 100% Frekote at a head speed of 2.5 inch/sec and at a height of 1 inch above panel.

Bondline Measurement (um)		
Panel	**Baseline**	**100% Frekote**
Average for each panel	228.1	201.2
Total average	214.6	

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

Note - we will compare mass change obtained with additional nozzle speeds

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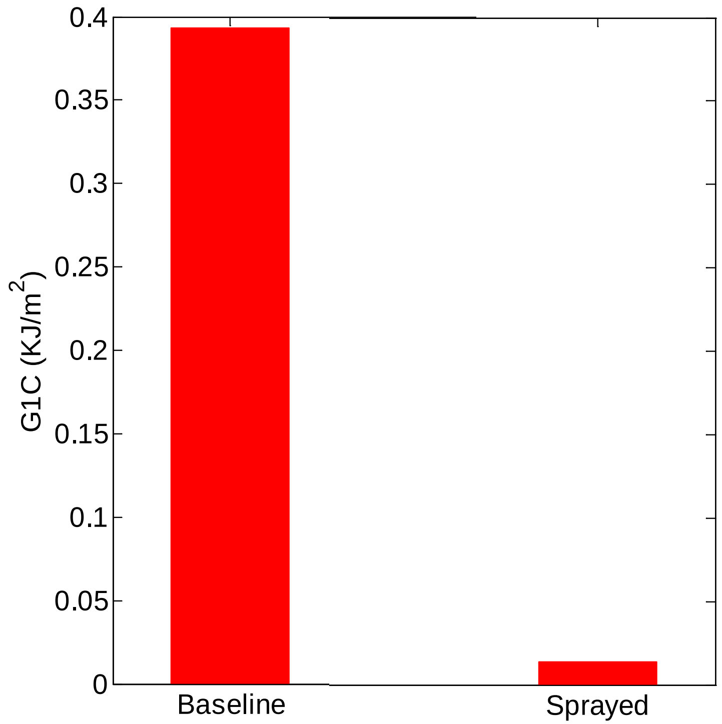
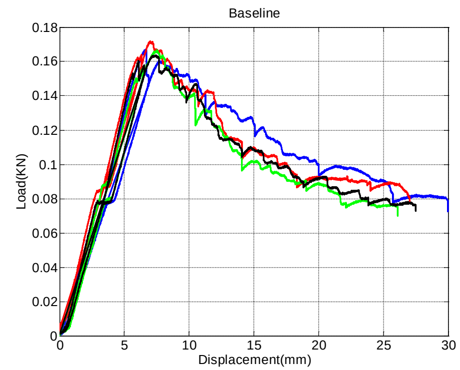
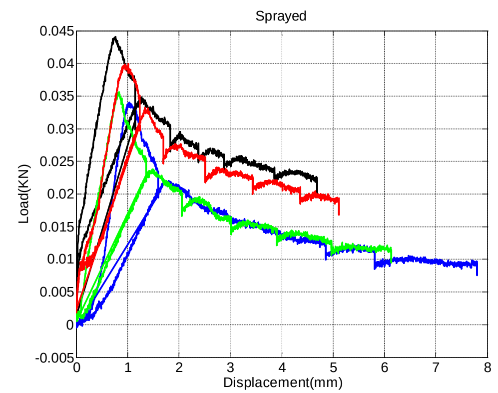
DCB Results







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			

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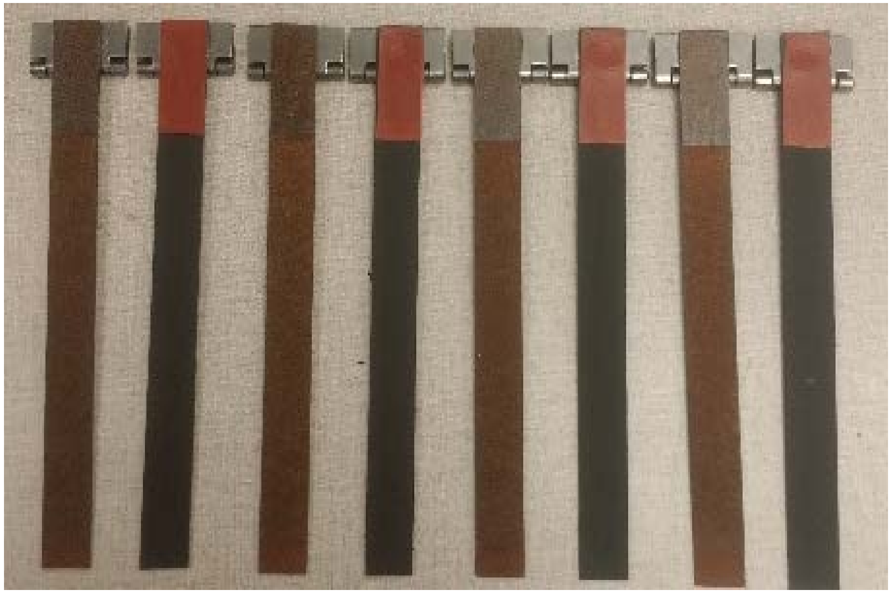
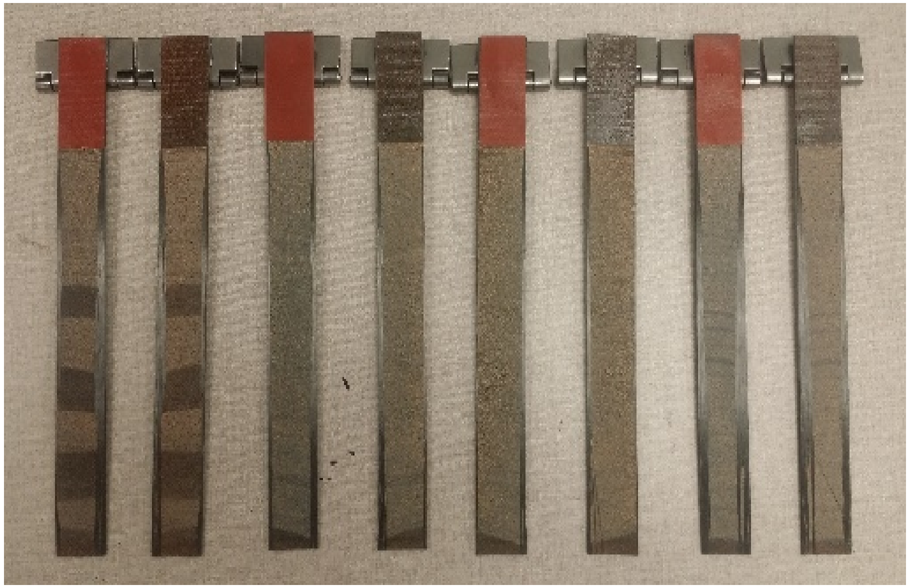
Modes of Failure



100% Frekote



Baseline

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Additional Analysis

- Results provided are based on a 2.5 in/s nozzle speed, 1 inch above panel
  - Approximately 4% of the baseline strength was obtained
- Additional tests were conducted at 3.75 in/s and 5 in/s to obtain weaker bonds.
- The nozzle height was also varied from 1 to 2 inches.

Bondline Thickness

Panel	Baseline	Spraying speed 2.5 (in/s)	Spraying speed 3.75 (in/s)	Spraying speed 5 (in/s)	Spraying speed 5 (in/s)	Spraying speed 5 (in/s) (increased height)
Panel Average	228.1	201.2	199.6	213.3	208.1	223.4
Overall Average	212.3					

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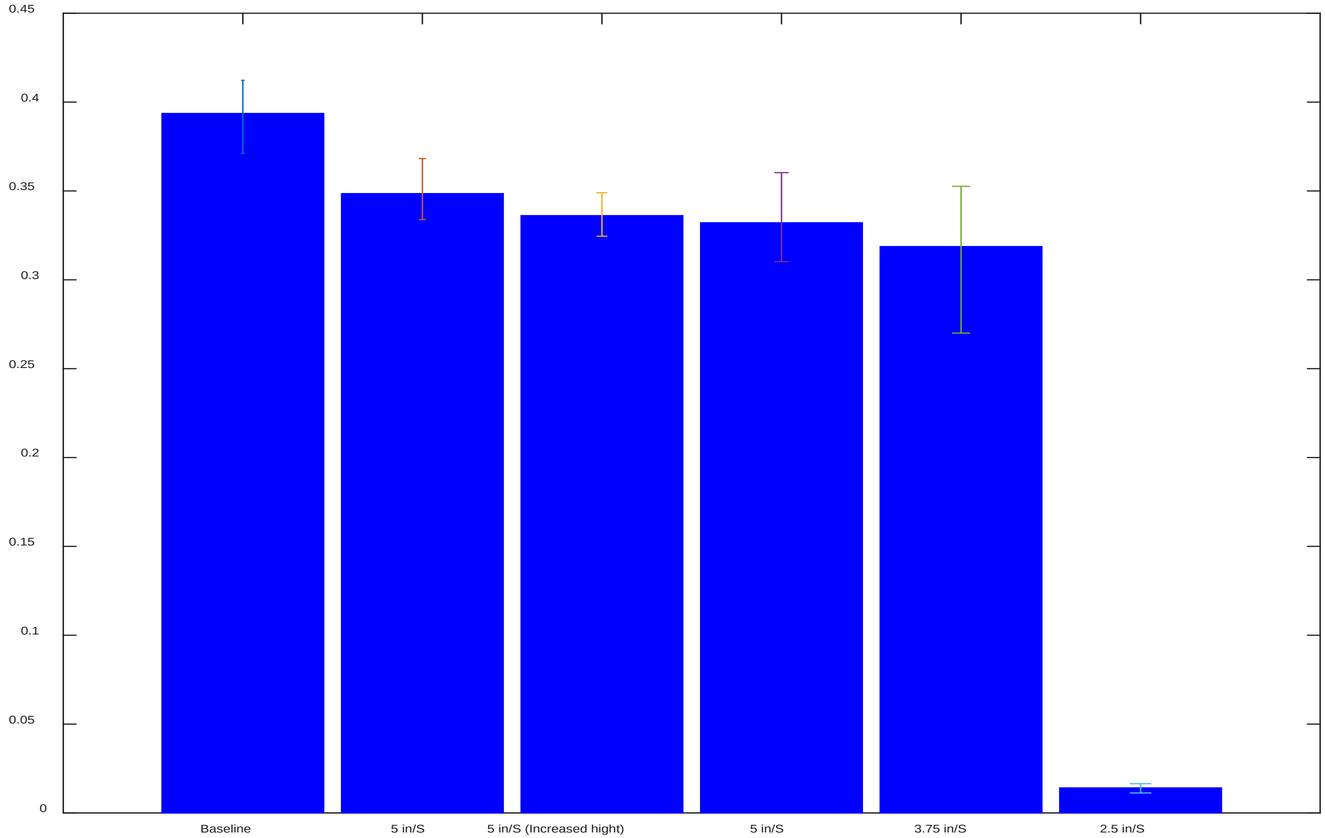
DCB results



Hexane	Baseline	Sprayed 2.5 in/s at 1 in height	Sprayed 3.75 in/s at 1 in height	Sprayed 5 in/s at 1 in height	Sprayed 5 in/s at 1 in height	Sprayed 5 in/s at 2 in height
Ave G1C	0.3935	0.0141	0.3187	0.3321	0.3485	0.3361

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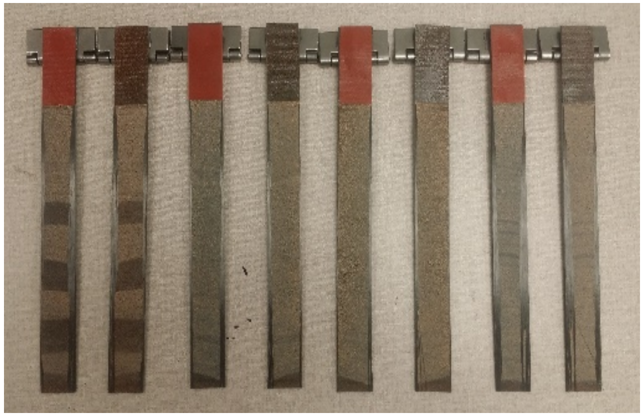
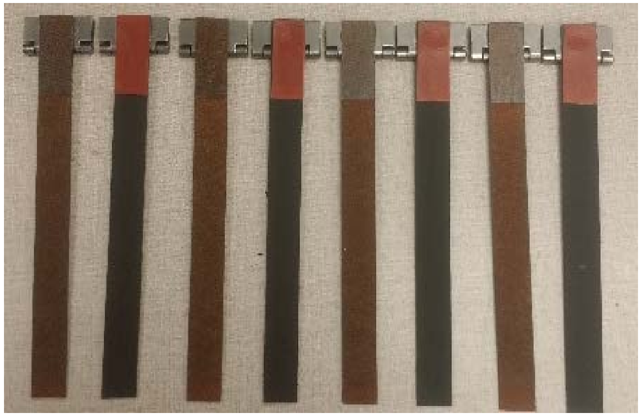
Weight Measurement

Weight of 1 x 1 in Composite Panel (mg)			
Spraying Speed (in/s)	**Baseline (mg)**	**Sprayed 100% Frekote (mg)**	**Contamination Weight (mg)**
2.5	1662.040	1662.265	0.225
3.75	1621.215	1621.348	0.133
5	1695.610	1695.734	0.124
5 (increased height)	1677.321	1677.431	0.110

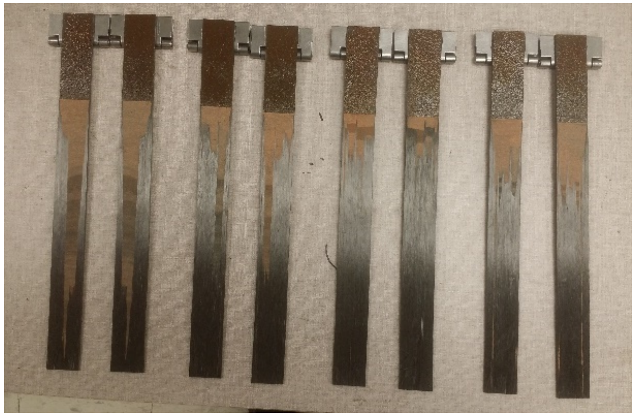
Modes of Failure



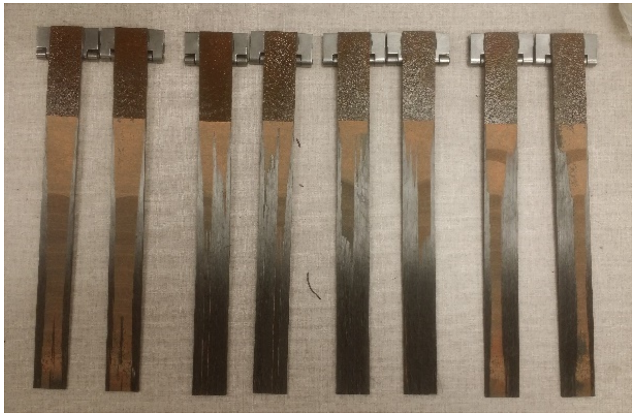
Baseline



2.5 in/s



3.75 in/s



5 in/s

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Summary

- 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 evaluation 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 (2.5 inch/sec) for contamination was established that provides a lower bound for bond strength (~4% of baseline strength).

- Additional speeds were run at 5 and 3.5 inch/sec providing bond strengths of ~84% and ~81% of the baseline strength, respectively. Repeated tests provided similar results.

- As expected, weight measurements of the contamination correlated well with the G_{1c}s.

Path Forward

- Testing will continue – varying the speed of the nozzle to change the level of contamination (between 3.75 and 2.5 in/s). The goal is to obtain repeatable data at 2 to 3 contamination levels between baseline and the 2.5 in/s rate.

- 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, solvent wiping and laser ablation (GLC). 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/Mini-DCB contaminated specimens will be manufactured and loaded in the SEM to evaluate the failure in real time.

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.

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CMH-17 Support

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

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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 …

Questions ?

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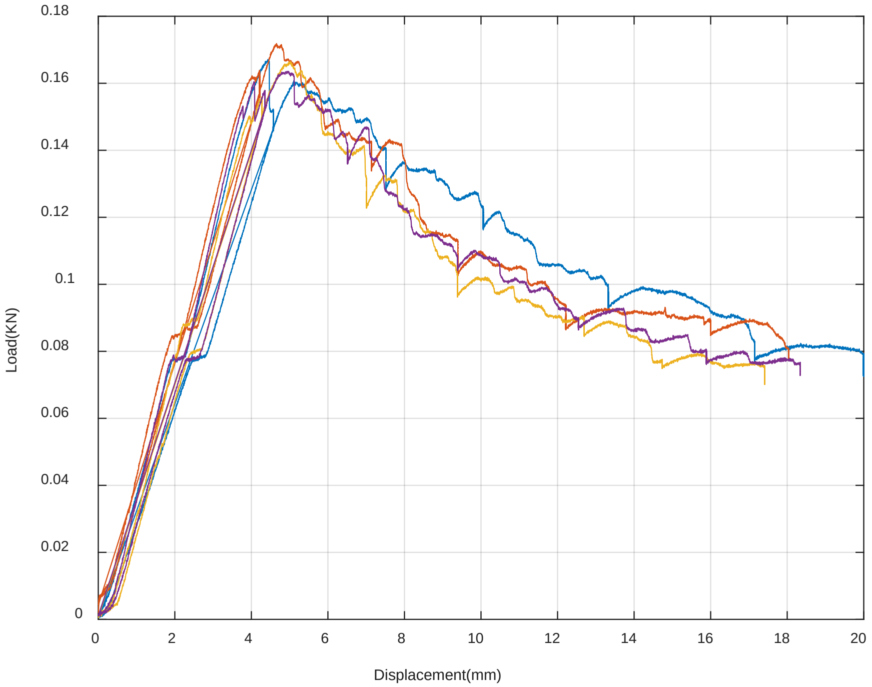
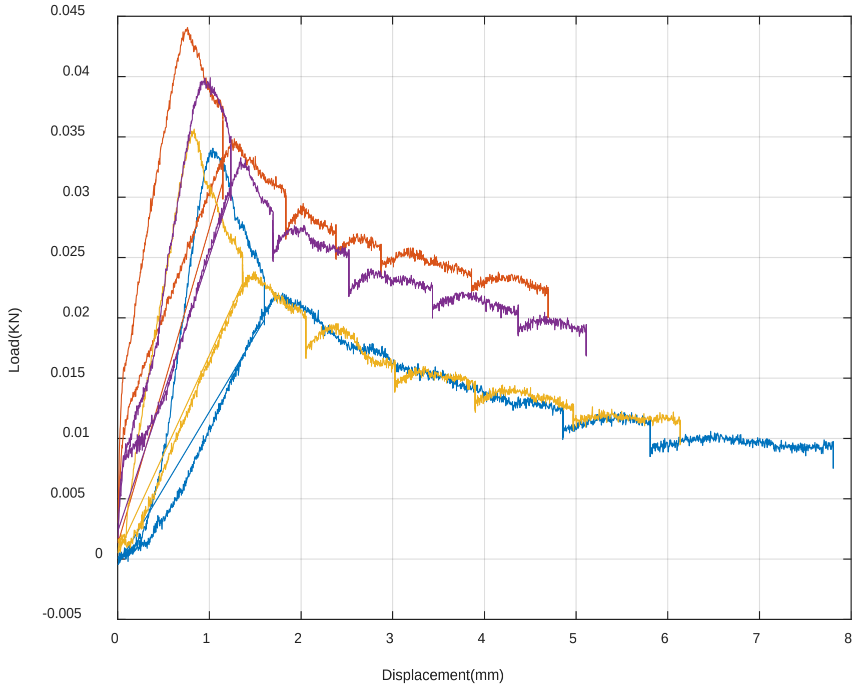
DCB results

Baseline



Sprayed 2.5 inch/Sec & 1 inch height



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