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AMTAS

Advanced Materials in Transport Aircraft Structures

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 and contamination mitigation 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**
  - Gabriela Gutierrez-Duran, Daniella Gil
- **FAA Technical Monitor**
  - Ahmet Oztekin
- **Industry Participation**
  - Exponent, 3M, Embraer, Boeing

Bonding/Contamination
Materials and Test Procedures

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
- Frekote 700-NC from Henkel Corporation

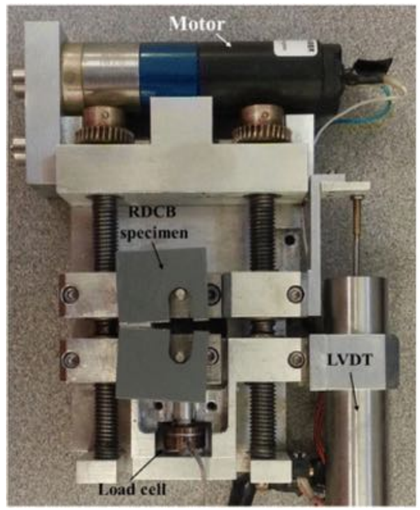
Bond Quality Evaluation

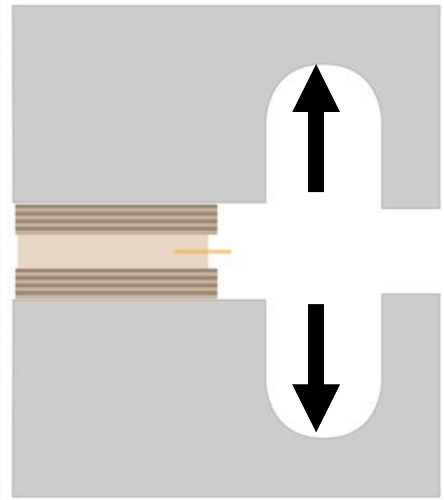
- Dual Cantilever Beam Testing – Macroscale
- μDCB and μENF – Microscale, In situ Electron Microscopy

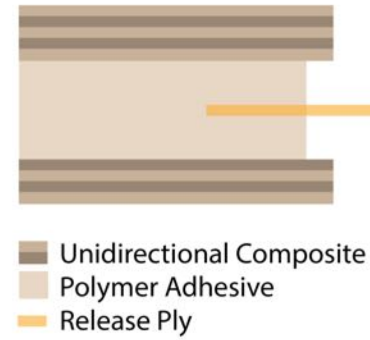
In-situ Testing
Load Frame and Electron Microscopy

Test Development

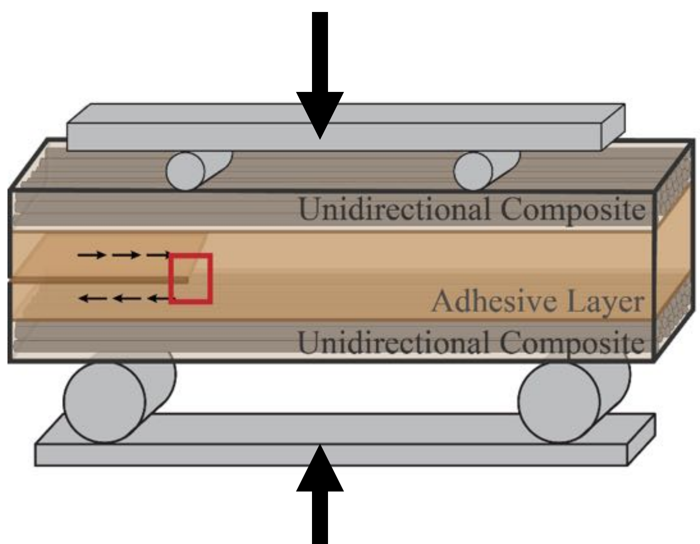
μDCB (Dual Cantilever Beam)
Assess the mechanisms of mode I fracture. Fixture was designed based on literature of metal-adhesive bond testing.

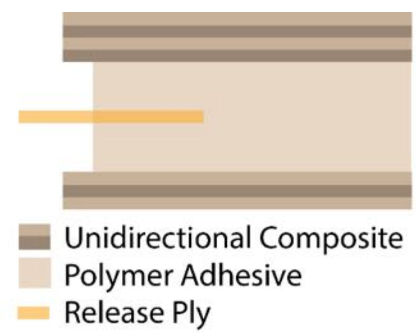






μENF (End Notch Flexure)
Assesses the mechanisms of mode II fracture. Fixture was designed based of traditional ENF testing of composite bonds





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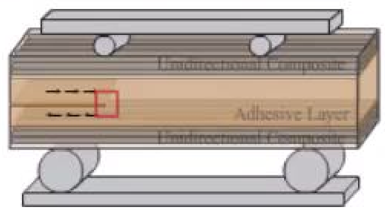
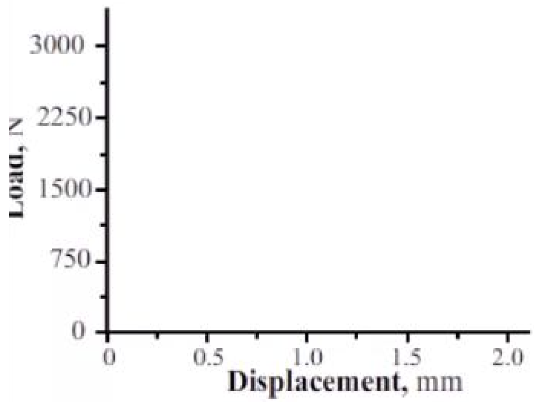
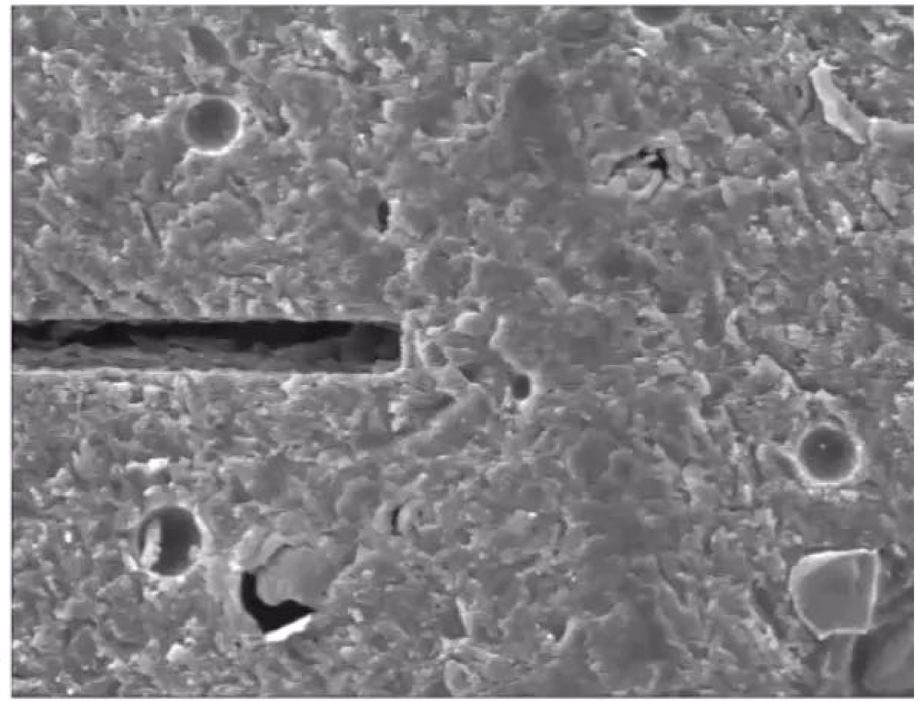
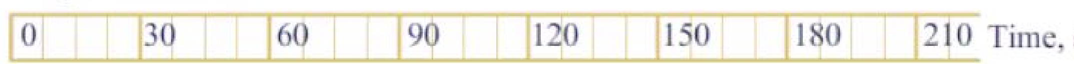
In-situ Testing
Load Frame and Electron Microscopy

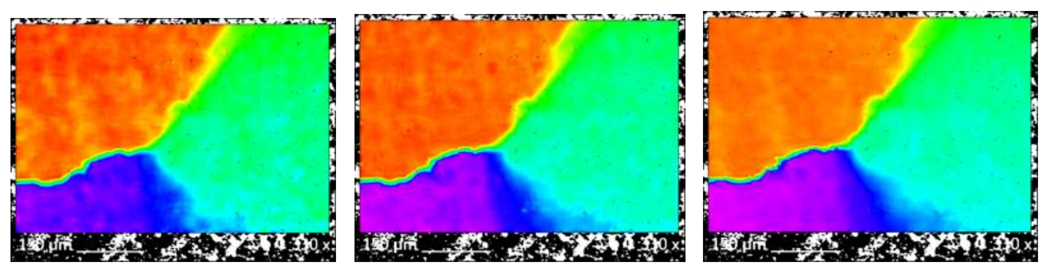
Testing Details: End Notch Flexure
Displacement Control: 0.5 mm/min
Sample: Adhesive-MENs (1 vol. %)







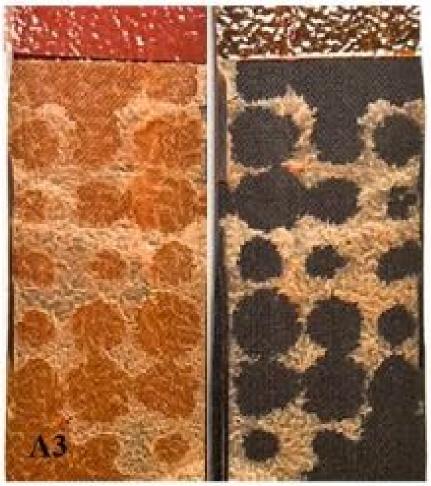




Advantages of Testing
Combined load-displacement with high magnification imaging can reveal the mechanisms of fracture. DIC can capture quantitative information (strain) as a function of processing.

Road Map of Contamination Studies

Discrete vs Continuous Approaches

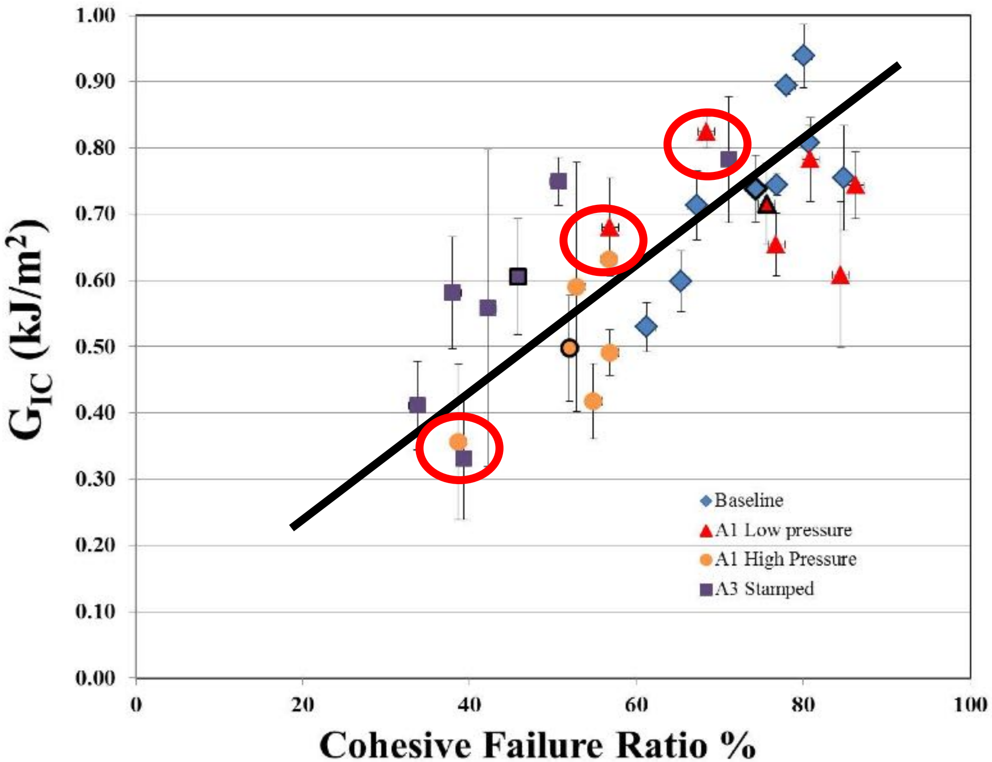


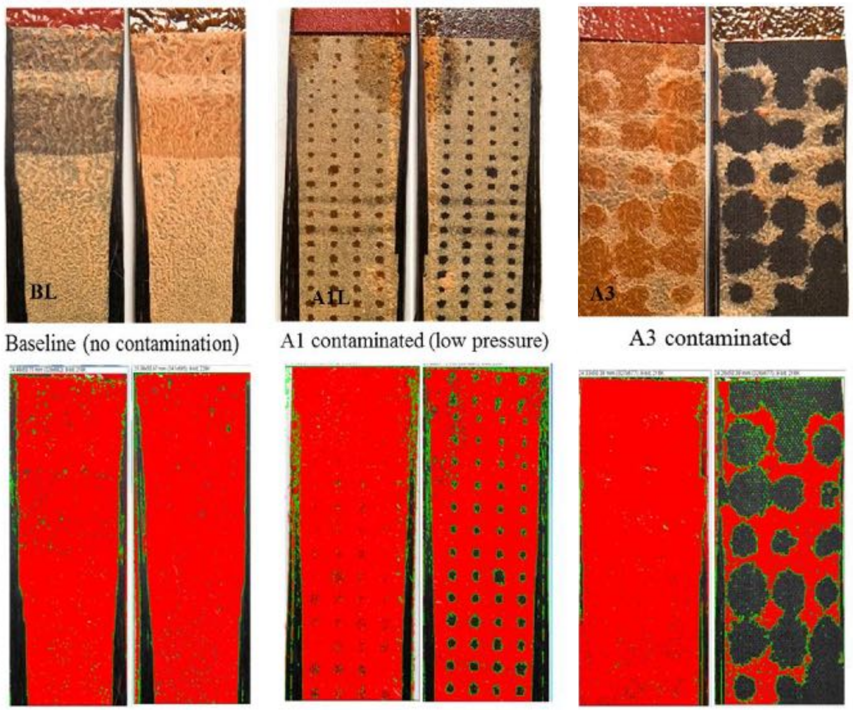
Discrete Method	Static	Exposed	Fatigue	Exposed & Fatigue
Baseline	DCB, μENF	DCB, μENF	DCB	DCB
1 mm (0 kg)	DCB	DCB	DCB	DCB
1 mm (22 kg)	DCB	DCB	DCB	DCB
3 mm (0 kg)	DCB, μENF	DCB, μENF	DCB	DCB

Continuous Method	Static	Exposed	Fatigue	Exposed & Fatigue
Baseline	DCB, μDCB	DCB, μDCB	DCB, μDCB	
~10%	DCB, μDCB	DCB	DCB	
~50%	DCB, μDCB	DCB, μDCB	DCB, μDCB	
~75%	DCB, μDCB	DCB	DCB	

Previous Contamination Efforts
Discrete Methods – DCB Testing

Create scaled bond strength – vary contamination size and area



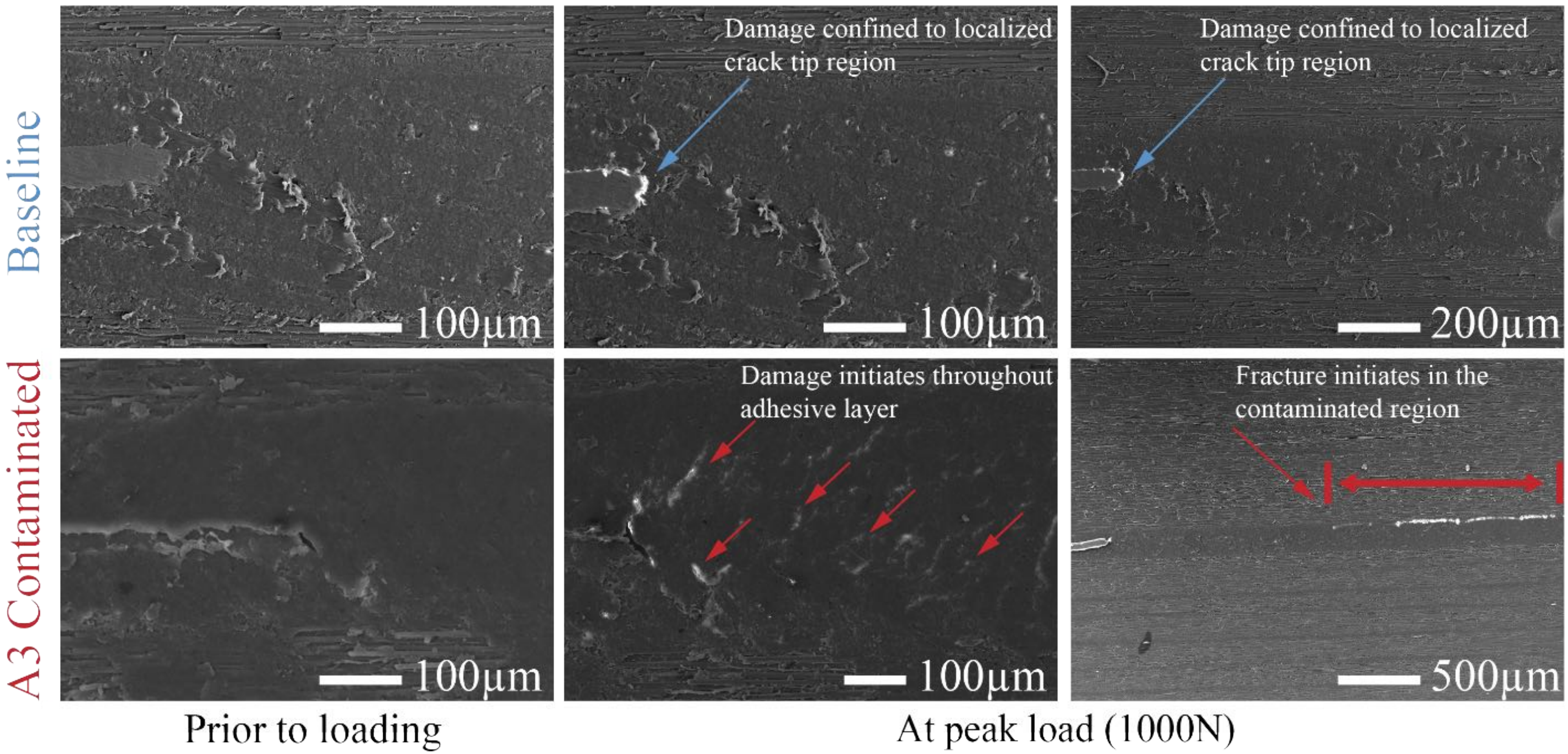


18% Drop in G_{IC} per 10% decrease in cohesive ratio

Contamination was modeled as circular cracks

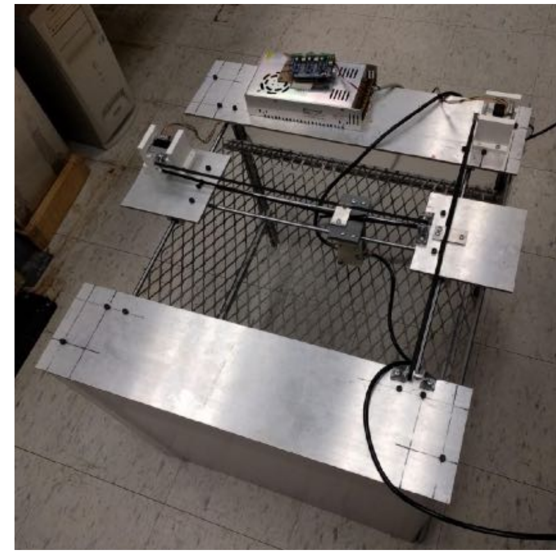
No significant effects of contaminate sizing, area was the dominate parameter.

Previous Contamination Efforts
Discrete Methods – μENF Testing



Initial Continuous Contamination
Spray Methods – Combine Hexane & Frekote

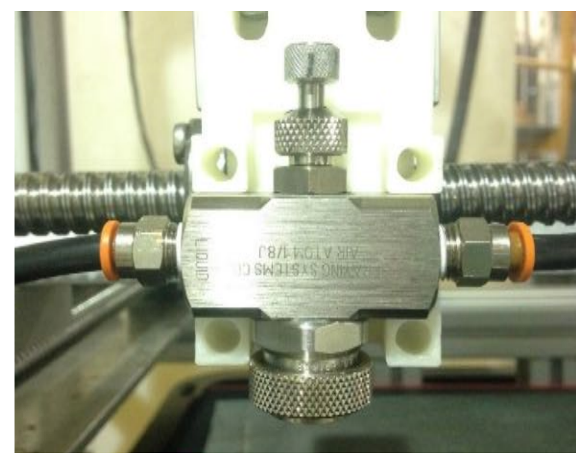
Feedback – interested in larger scale contamination areas
– uniformly distributed contamination to create weak bonds



Initial Uniform Contamination Approach

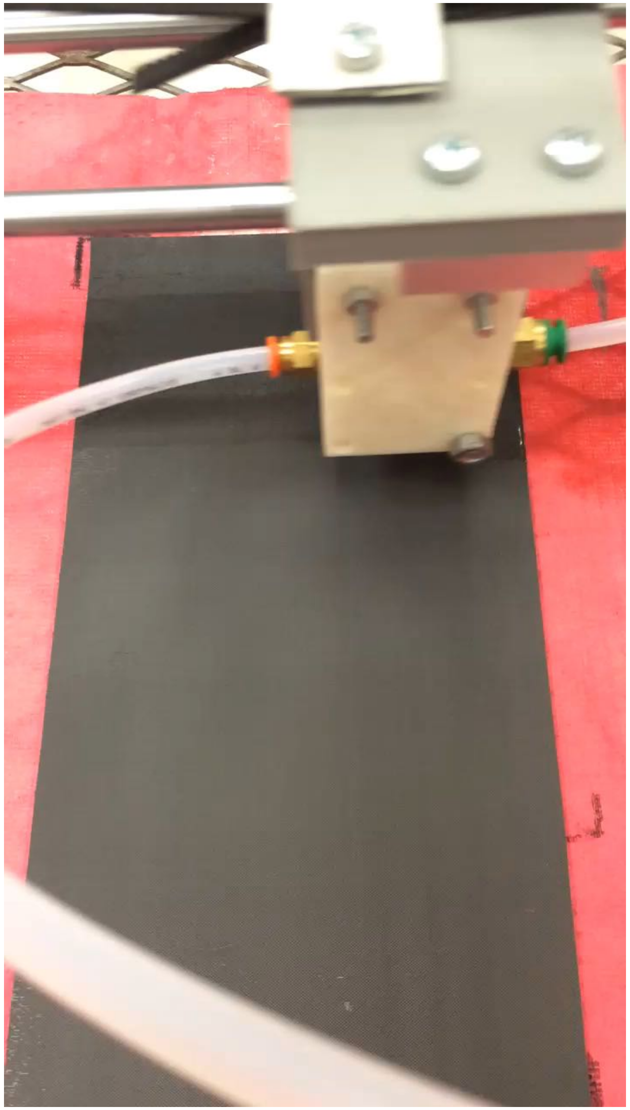
Contaminant –Frekote release agent (Siloxane)

- 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

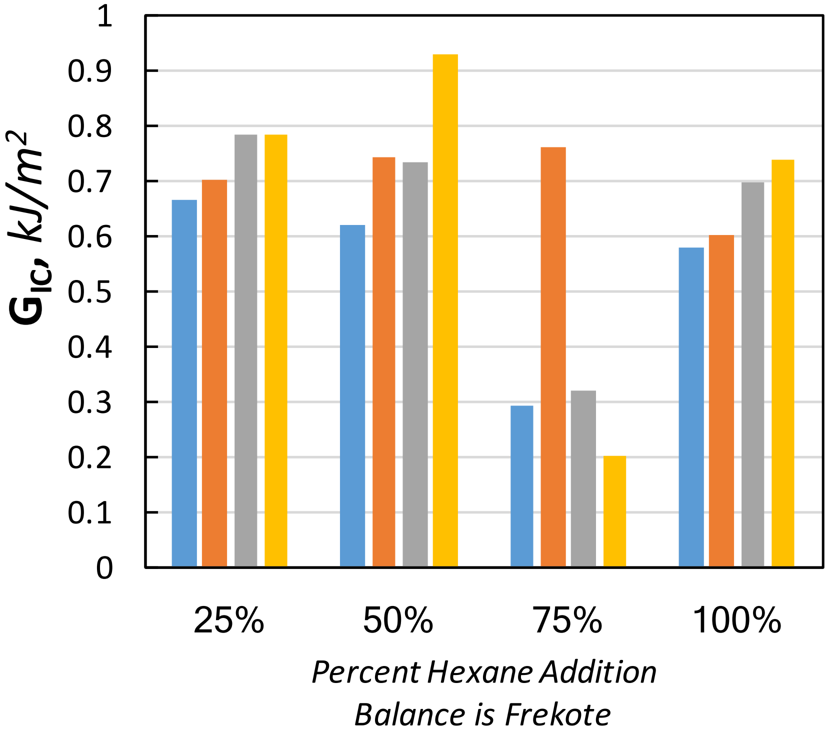


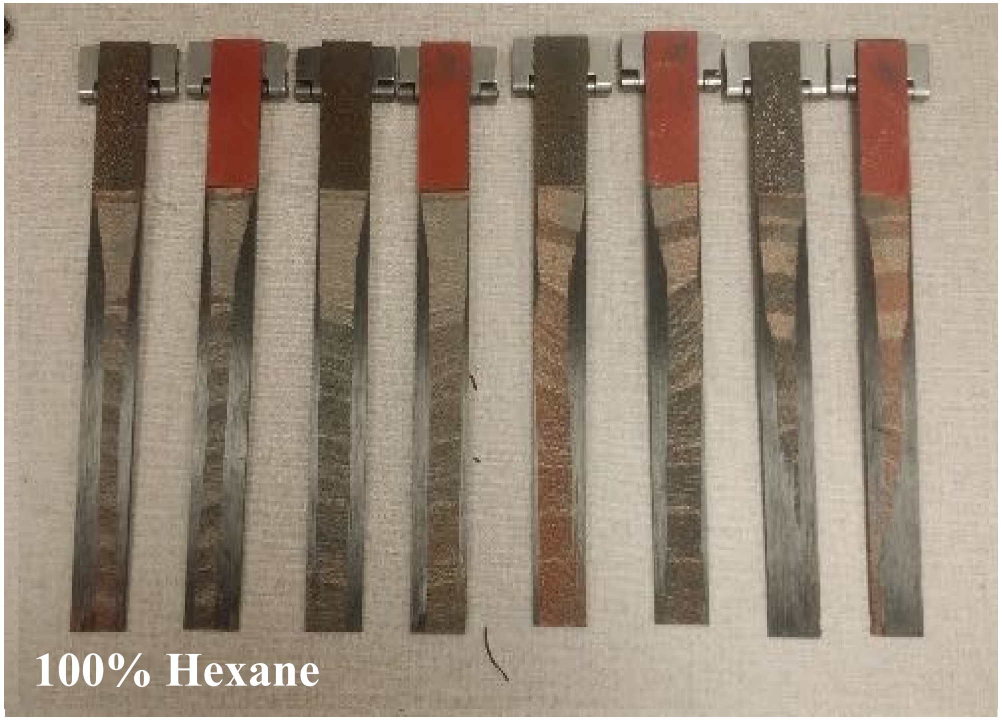
Initial Uniform Contamination

Example of Spray Methods



Initial Uniform Contamination
Combined Hexane & Frekote





Inconsistent and variable from spray to spray

Failure surfaces showed significant interlaminar failure, poor bond quality

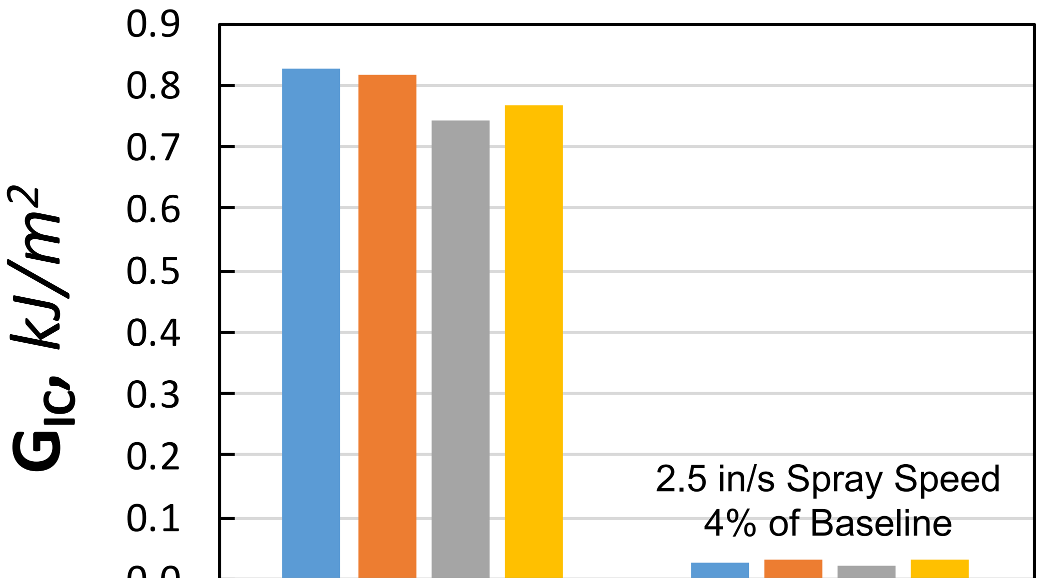
Alternative Uniform Contamination
Use only Frekote, Vary Spray Parameters

Direct Contamination Approach

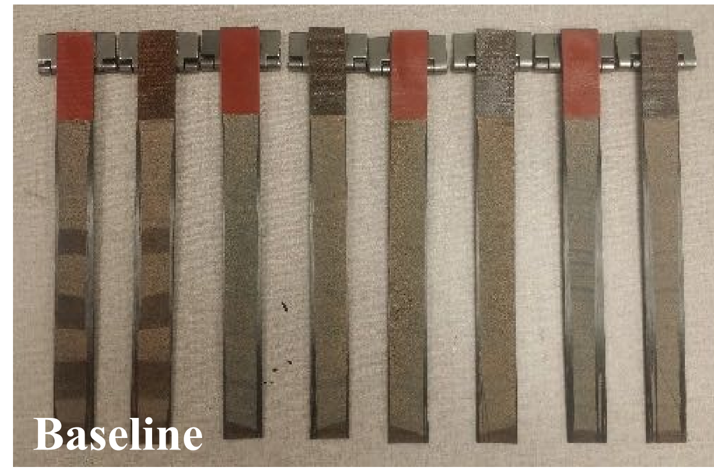
- Due to mixed results and effects of Hexane – sought an alternative approach
- Contaminate panels directly, to determine if amount of Frekote can be varied/controlled to reduce bond strength
- Vary spray head speed to control amount of Frekote
    - Initially used slower speeds to find a lower level of bond strength
    - Subsequent trials use faster head speeds
- Spray 1x1 inch coupon and compare mass changes
- Try to obtain consistent and repeatable levels of contamination at approximately at 3 different levels (i.e. 10, 50 and 75%) of the original bond strength

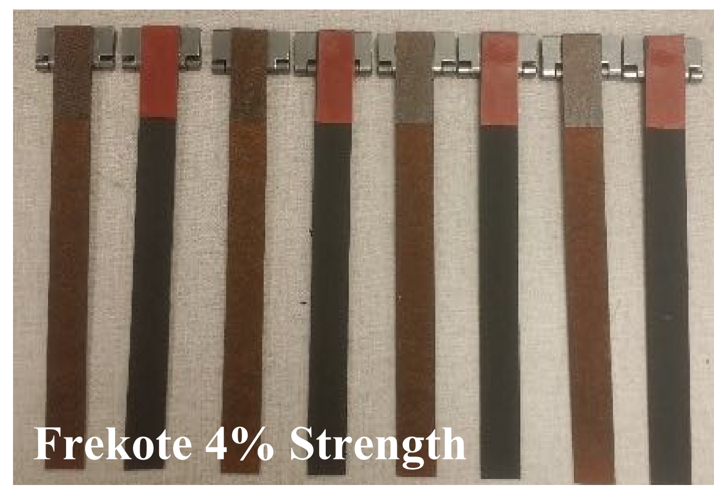
Continuous Contamination

Initial Bond Quality Assessment (DCB)



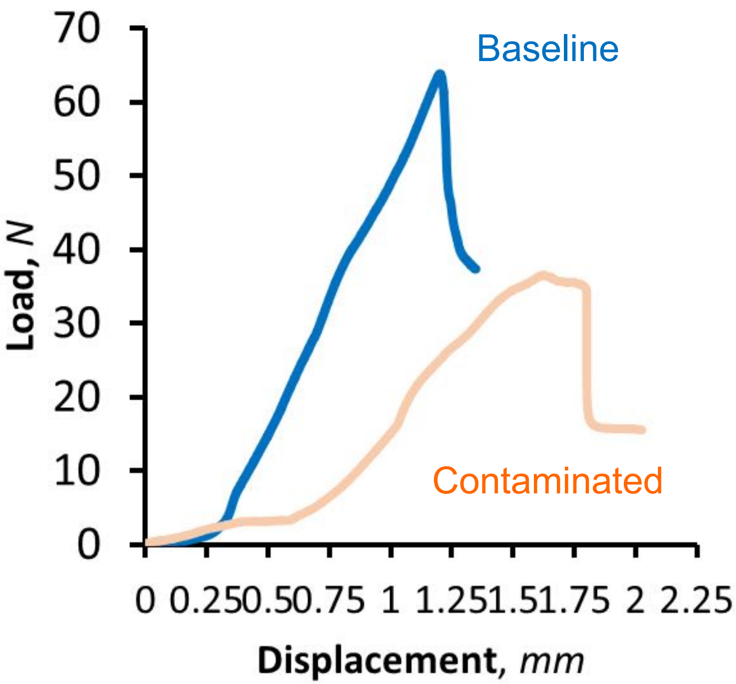
	Baseline	As Sprayed
1	0.824	0.027
2	0.814	0.031
3	0.742	0.022
4	0.768	0.033



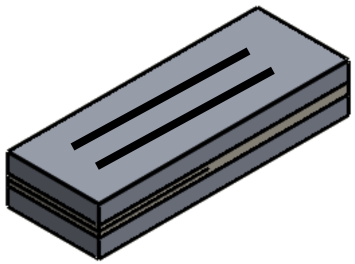


In-situ Testing

Baseline: µDCB Testing



Specimen Details



Baseline

L/W: 40mm x 10mm

thickness: 5.2 mm

Pre-crack: 8 mm

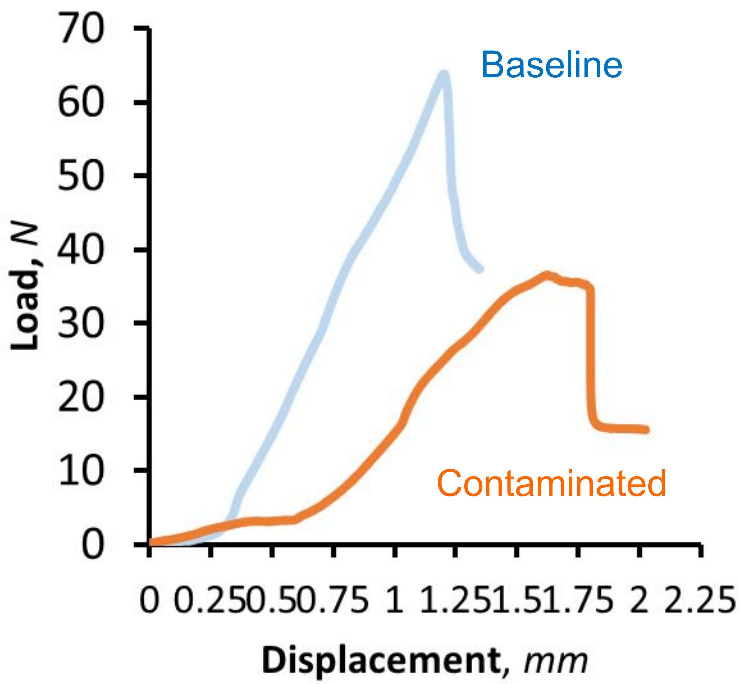
10 layer unidirectional composite panels

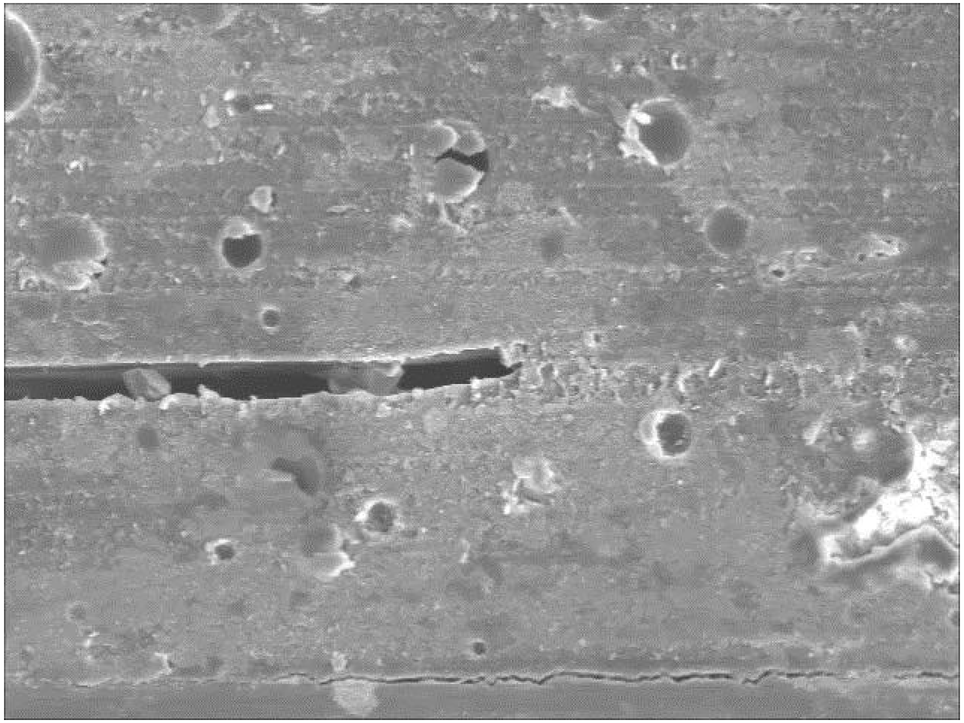
Observations

- 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

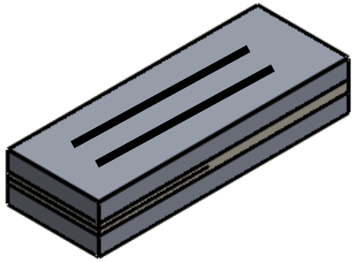
In-situ Testing

4% Contamination: μDCB Testing





Specimen Details



Contaminated

L/W: 40mm x 10mm

thickness: 5.2 mm

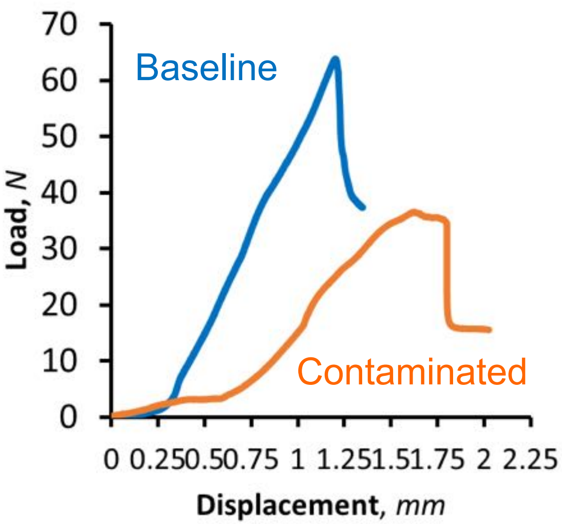
Pre-crack: 8 mm

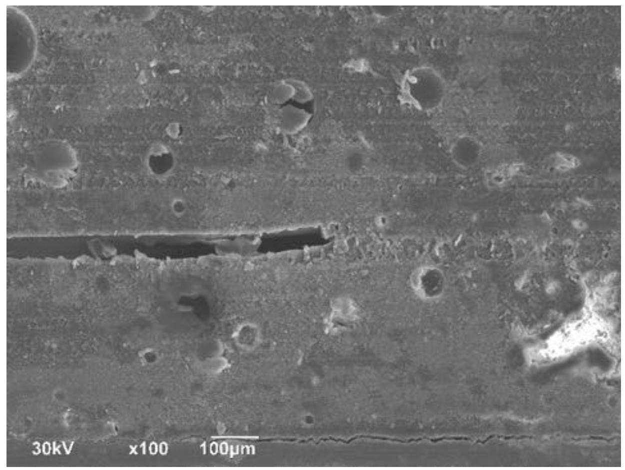
4% contamination procedure was used at the interface

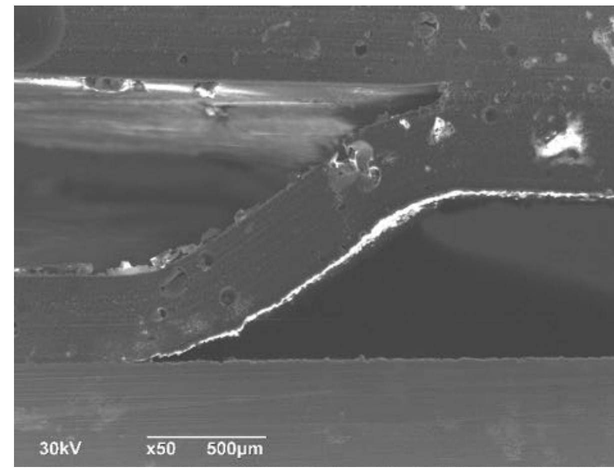
Observations

- 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

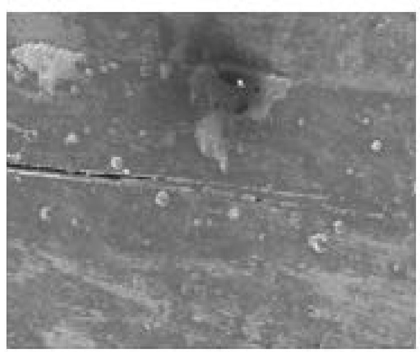
In-situ Testing
Summary of Mechanisms

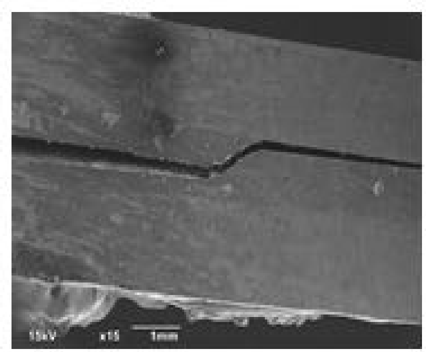


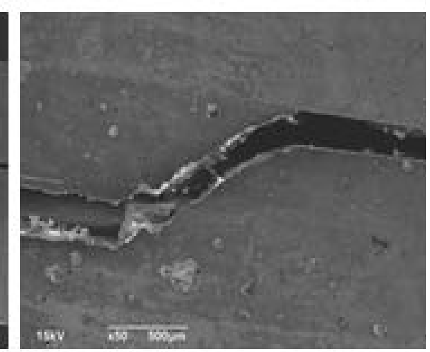




Advantages of Testing
Combined load-displacement with high magnification imaging can reveal the mechanisms of fracture. DIC can capture quantitative information (strain) as a function of processing.

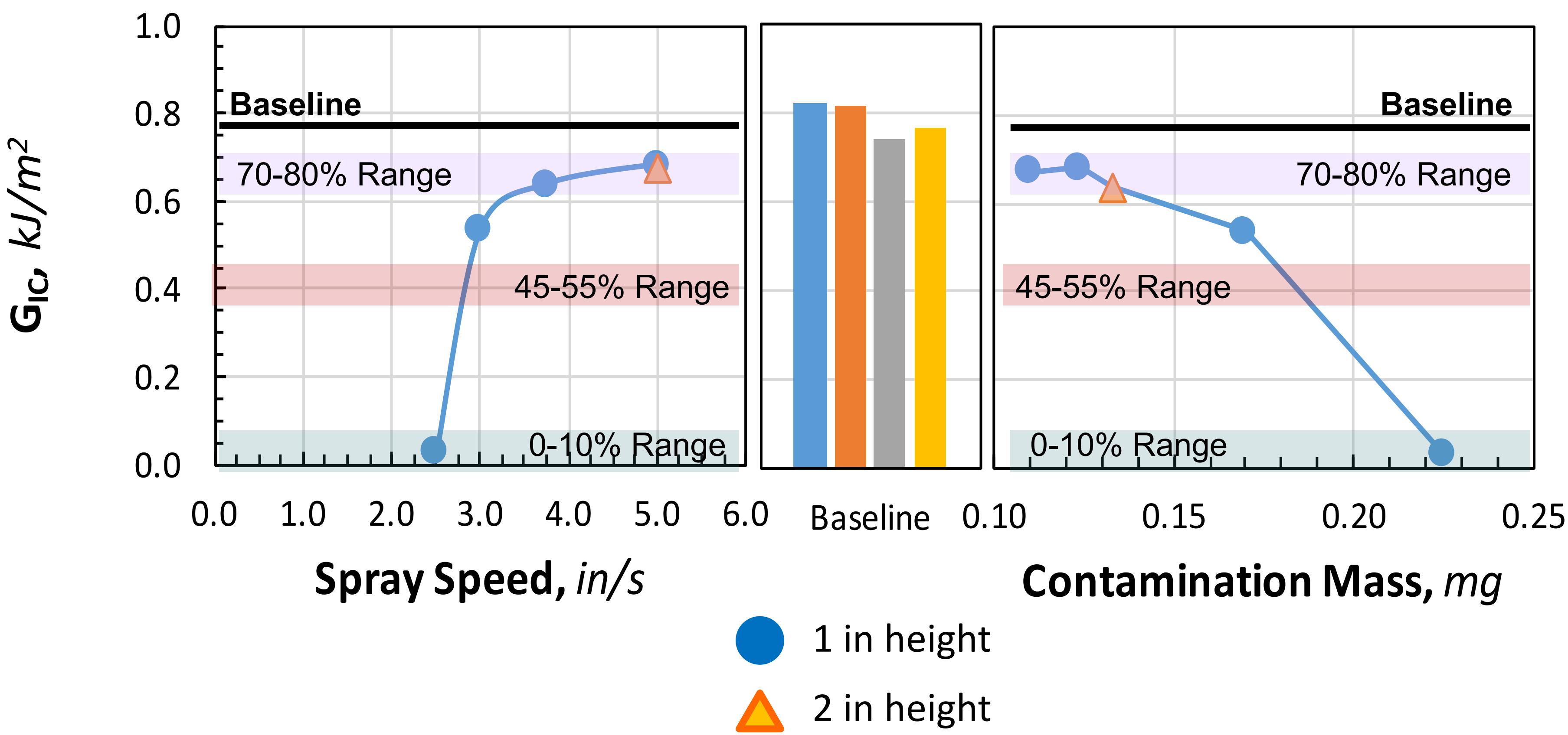




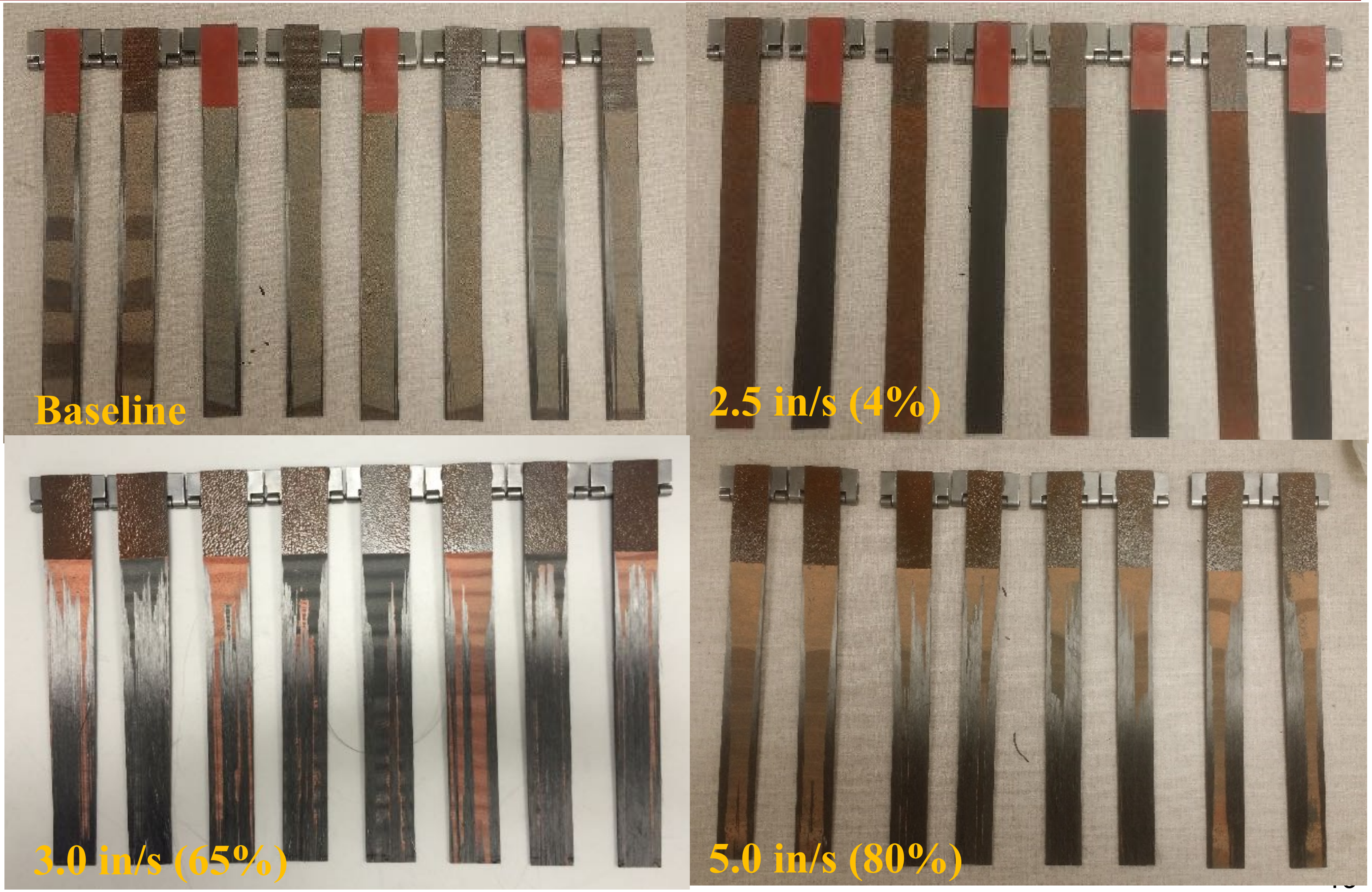


Continuous Contamination

Varying Spray Speed and Contamination Mass



Continuous Contamination
Modes of Failure



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Contamination Mitigation

Preliminary Data for MPK Wipes

- Initial strategy: solvent wipes (Methyl Propyl Ketone)
- After contamination, panels were wiped with MPK wipe and then with a dry lint free wipe. Process was then repeated.
- 4 DCB coupons were manufactured with contamination on one interface and cleaned
- Average bondline thickness: 236 μm

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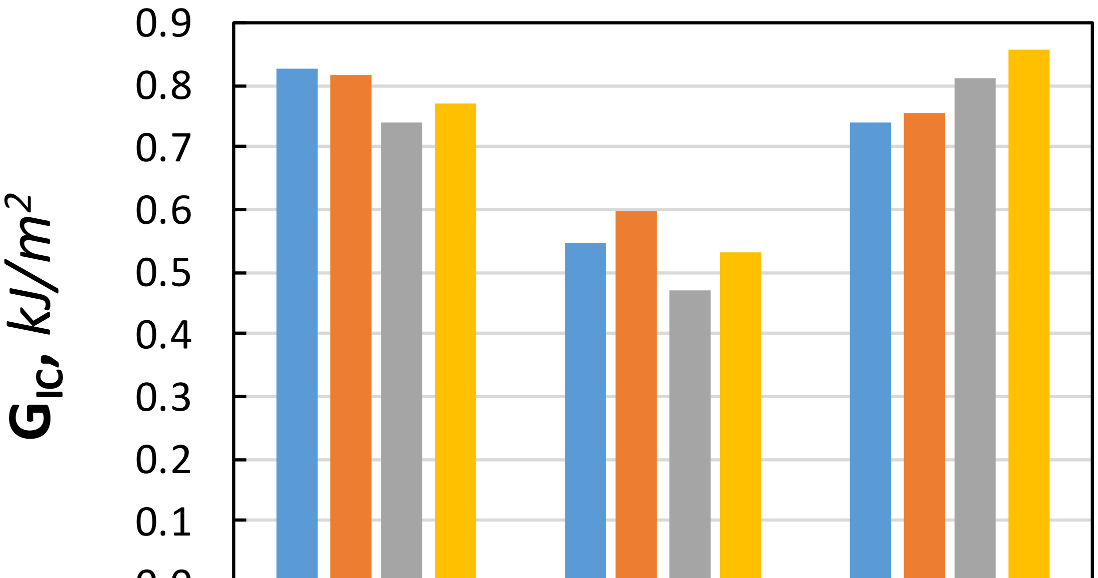
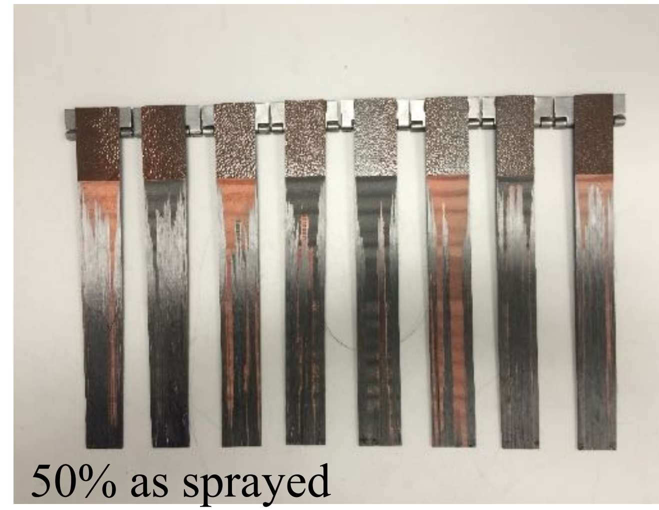
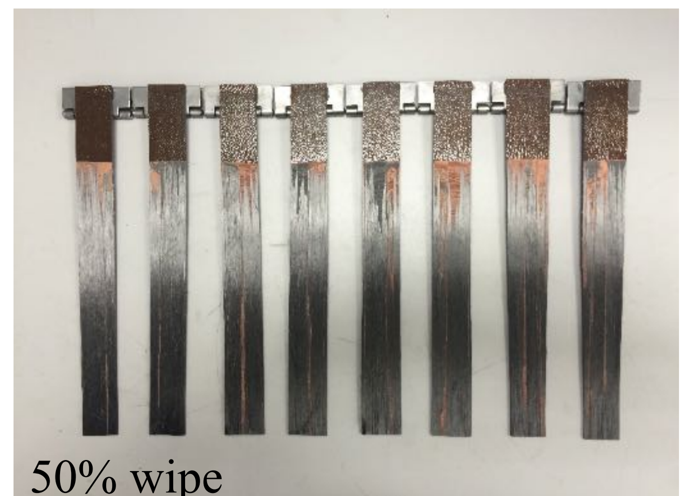
Contamination Mitigation
Preliminary Data for MPK Wipes



	Baseline	65%%	65% Wipe
1	0.824	0.548	0.738
2	0.814	0.598	0.754
3	0.742	0.468	0.812
4	0.768	0.532	0.854


50% as sprayed


50% wipe

Key Contributions/Summary

- Development of a procedure to produce scalable and repeatable weak bonds by contaminating one of the bond surfaces
  - Manufactured a cost effective contamination system that scales the bond strength by varying the speed of the spray nozzle
  - Bond strengths of ~80%, 65%, 4% were obtained (repeat 50% procedure)
- Incorporate strategies to mitigate the contamination – current approach includes solvent wiping (next steps will include sanding)
  - Solvent wiping provided improvement of fracture toughness data, but fracture mechanisms were inconclusive.
- Evaluate both contaminated and treated samples using multi-scale approach – includes typical DCB testing and micro DCB testing that can provide real time monitoring of the crack propagation under load
  - Micro DCB testing utilizes a load frame sized to fit within an SEM. Small DCB coupons can be used to investigate mechanisms of failure
- Evaluate both the short term bond quality and long term durability with exposure in an environmental chamber (next steps)

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Path Forward
Durability and Mitigation Assessment

Continuous Method	Static	Exposed	Fatigue	Exposed & Fatigue
Baseline	DCB, µDCB	DCB, µDCB	DCB, µDCB	
~10%	DCB, µDCB	DCB	DCB	
~50%	DCB, µDCB	DCB, µDCB	DCB, µDCB	
~75%	DCB, µDCB	DCB	DCB	

- Intermediate bond level will be repeated to obtain three levels of weak bonds
- DCB and micro DCB coupons will be manufactured and tested with the three levels of contamination and will be treated with both solvent wipes and sanding (Merit ALO Resin Bond 180 grit)
- Durability analysis will be conducted for the same DCB and µDCB coupons to assess the effects of water ingression and elevated temperature (exposure in an environmental chamber for two months) on the bond strength

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 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

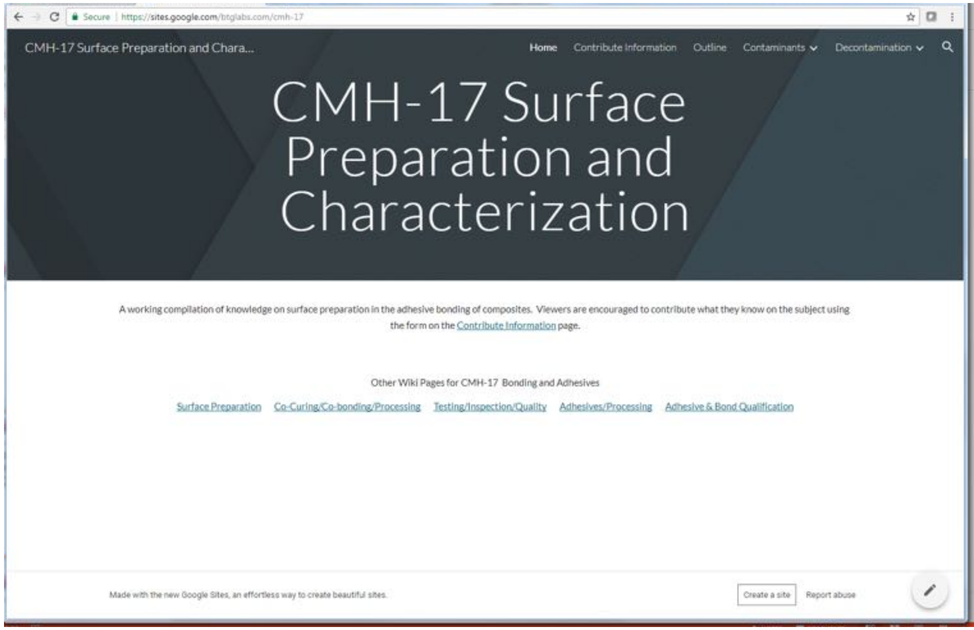
1. General Considerations — Creel, 3M
2. Surfaces — Faria, Embraer
3. Adhesives and Processing — Creel, 3M
4. Inspection, Testing, Quality — McDaniel, FIU
5. Co-cure, Co-bond, Multi-stage — TBD

Volunteers for Bonded Joint Working Groups needed!

Also: Leadership for Bolted Joint Content

CMH-17 Support

- The BAT groups has monthly calls to discuss progress.
- Map of existing (limited) content bonding content was provided identifying relevant sections in the current handbook.
- Initial efforts on updating Ch 3 Section 5.9 - path forward will depend on the amount of content developed.
- Initial outline for 5.9 has been expanded and sections have been assigned to each team.
- Focus will initially be on developing content for the Surface Prep Team.
- Tim Barry (BTG Labs) has been leading the effort to create Wiki links that will facilitate the accumulation of content.
- Links have been created for each of the five sections.
- Integration of JAMS content and DARPA TRUST
- Preparing to present an update at the upcoming CMH-17 in November



Questions?