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

- There is significant interest in assessing the durability of composite bonded joints and how durability is affected by contamination.
- Need to study mechanisms of failure to understand property influences and possibly predict bond failure
- Past research has focused on determining/understanding acceptable performance criteria using the initial bond strength of composite bonded systems.

Objective

- Formulate a methodology to create **undesirable bonding conditions** in a scalable and repeatable manner.
- Evaluate methods to mitigate the undesirable conditions via surface preparation methods.
- Investigate the effect of harsh environmental conditions on adhesive bonds
- Understand the mechanisms of fracture and failure of these bonds to further improve predictive abilities.
- Support the CMH-17 Handbook

Effect of Surface Contamination on Composite Bond Integrity and Durability

- **Principal Investigators**
  - Dwayne McDaniel, Ben Boesl

- **Students**
  - Brian Hernandez, Gabriela Gutierrez-Duran, Fernando Rojas, Mauricio Pajon

- **FAA Technical Monitor**
  - Ahmet Oztekin

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

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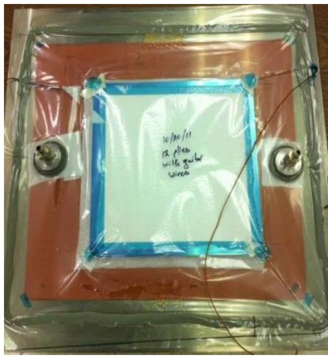
Manufacturing of Bonded Systems

Fabrication of Laminates



Laminate Cure

(Cure Cycle @350F)

Bonding of Laminates

Adhesive Cure

(Cure Cycle @350F)

Preparing/Cutting Samples

Adhesive Bond Strength Testing

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.

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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 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

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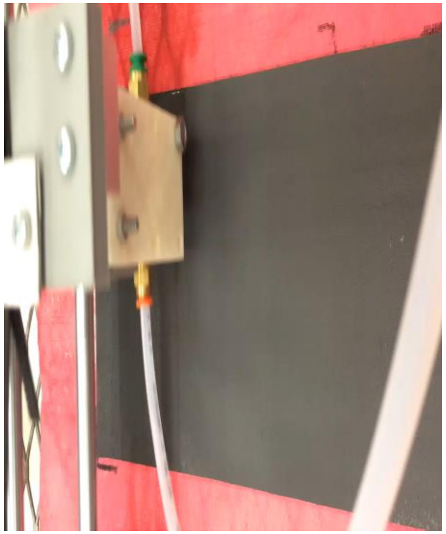
Contamination Approach

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 volume of Frekote
- Total amount of contaminate applied is measured using an analysis of pre- and post- weight measurement.



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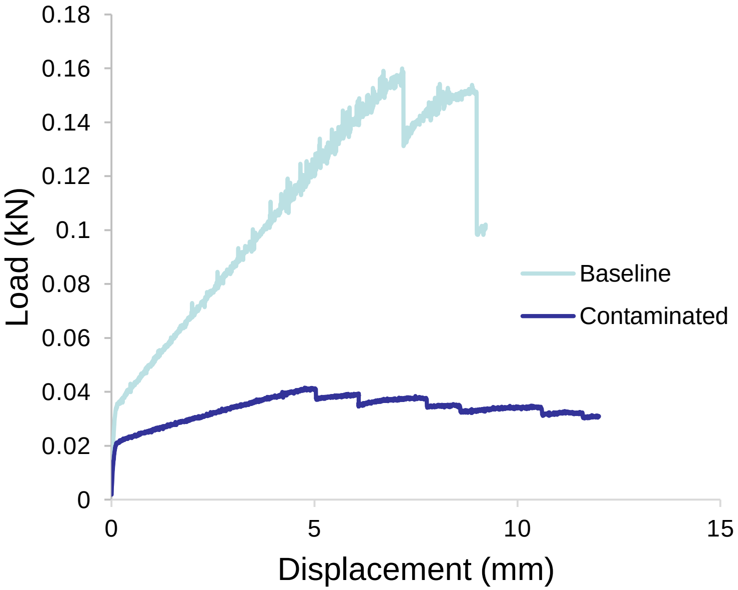
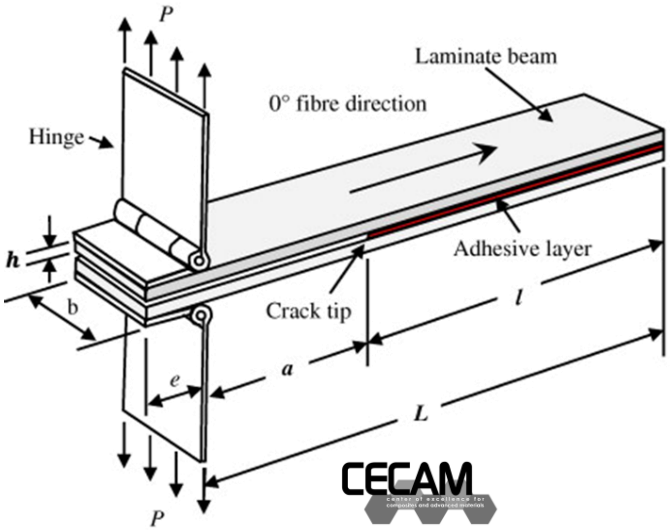
Calibration of Contamination Levels

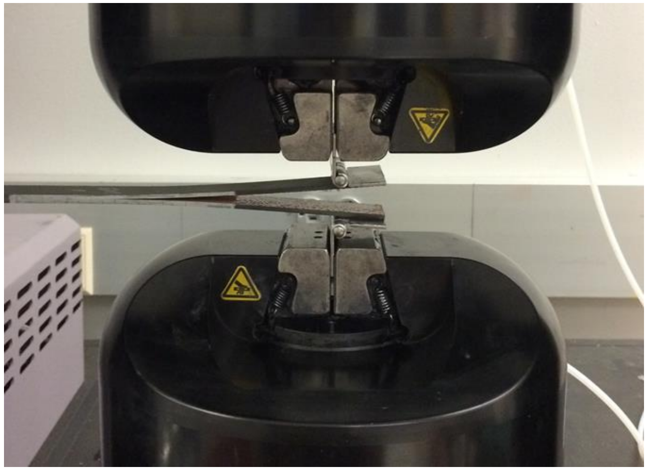
- Calibration of the contamination levels is important in order to be able to trace back the amount of contaminant used and relate that amount to the strength of the weak bond created
  - This enables us to determine the different bond strengths that can be created from different amounts of contaminant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

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







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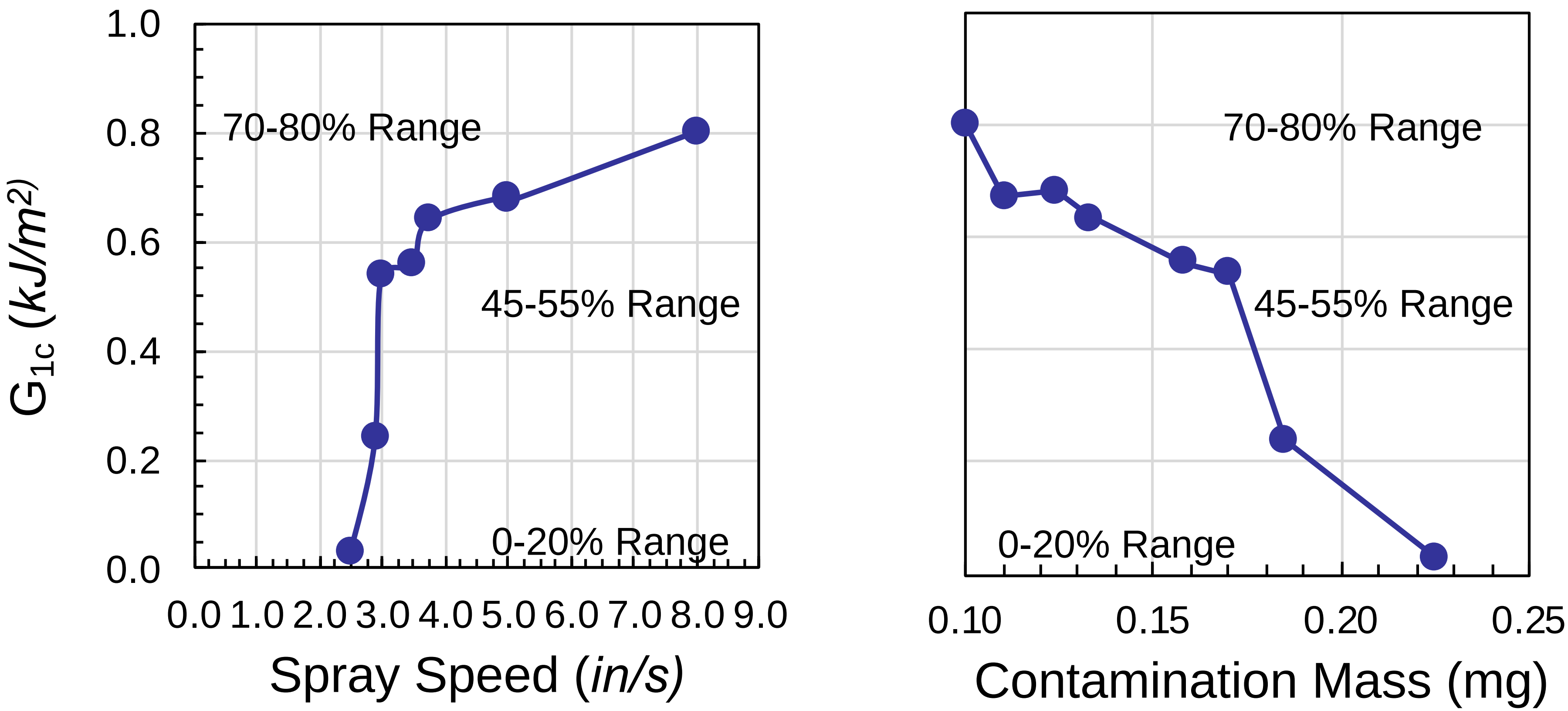
Contamination Results



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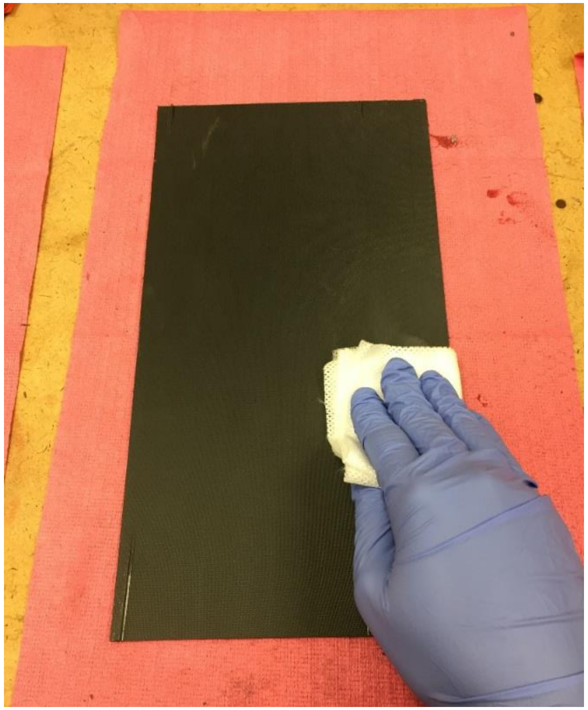
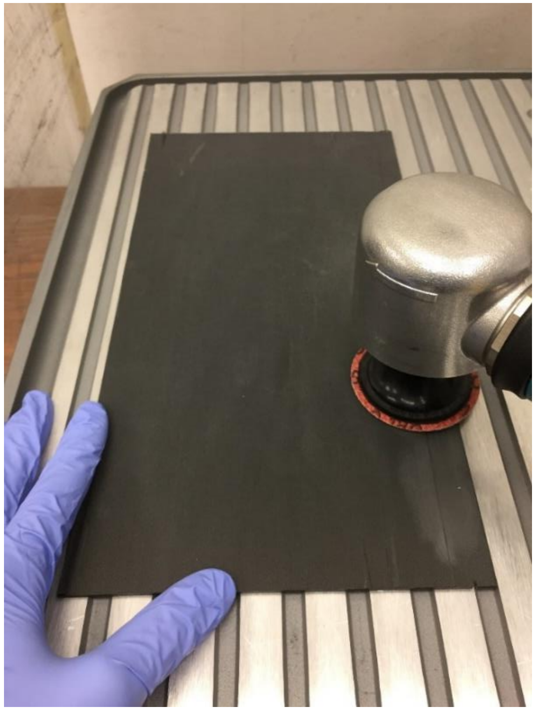
Mitigation Procedures

- **GOAL –** Evaluate mitigation processes that are designed to remove 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





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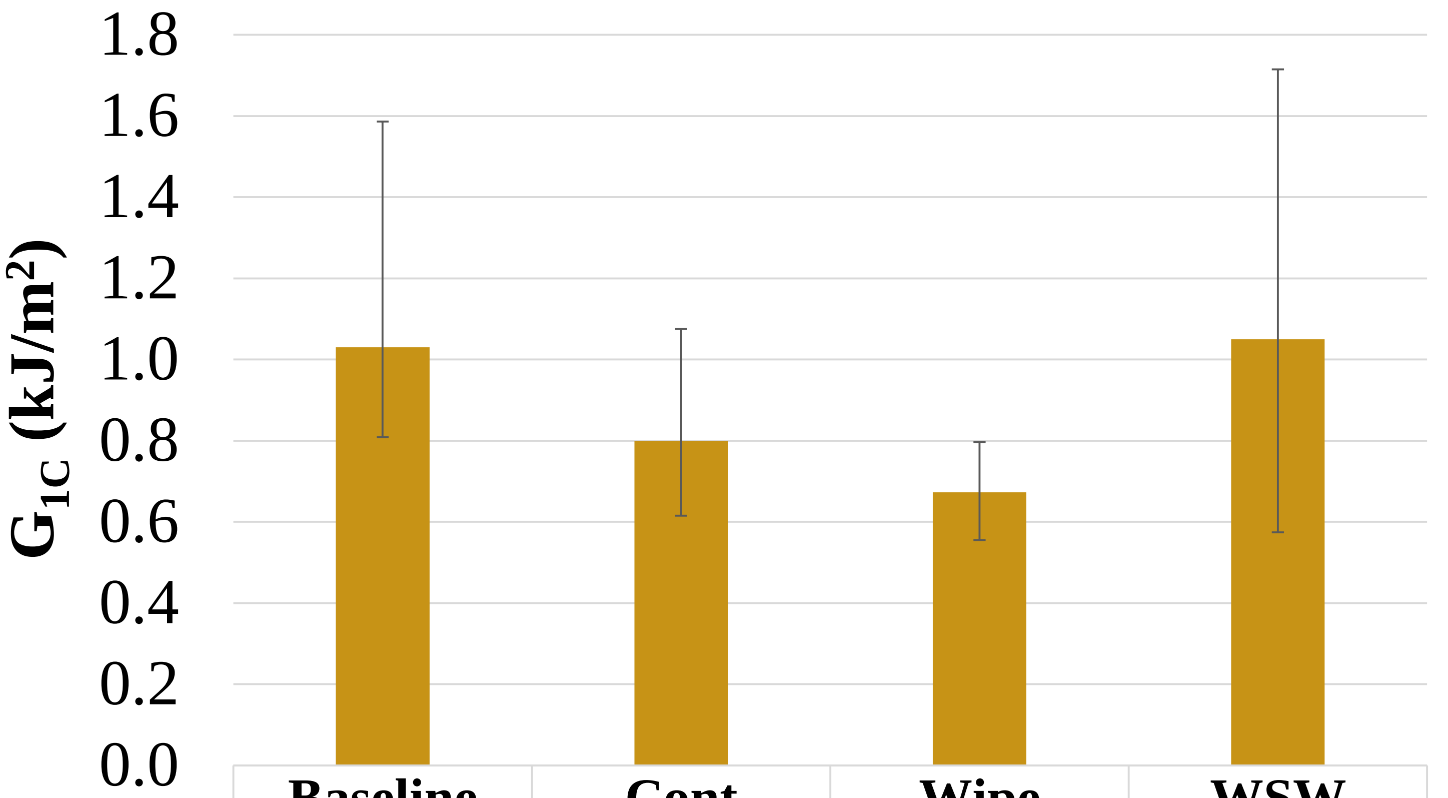
Results of Mitigation Approaches

70-80% of Baseline



	Baseline	Cont	Wipe	WSW
78%	1.030	0.800	0.673	1.050

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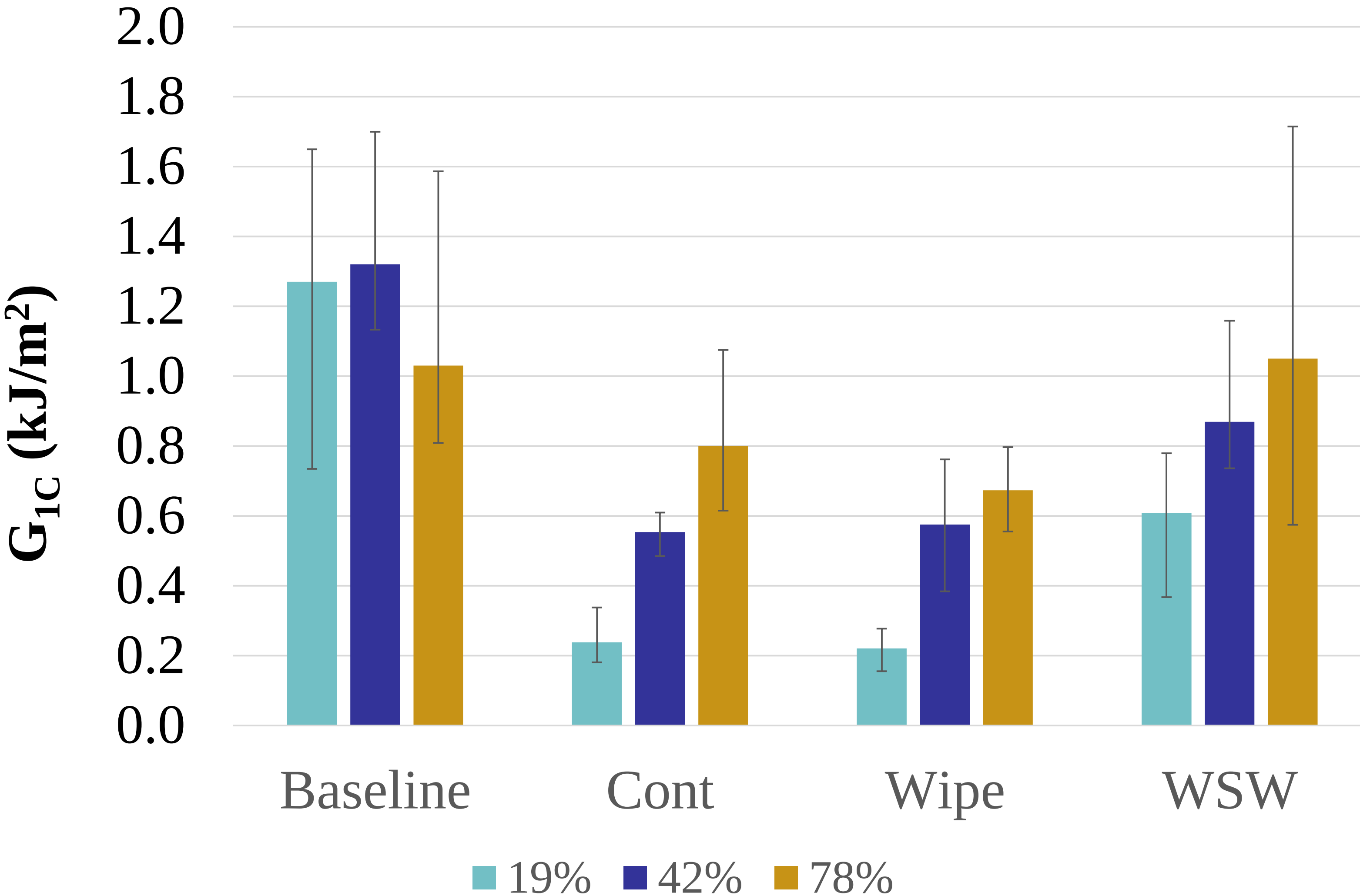
Mitigation Results



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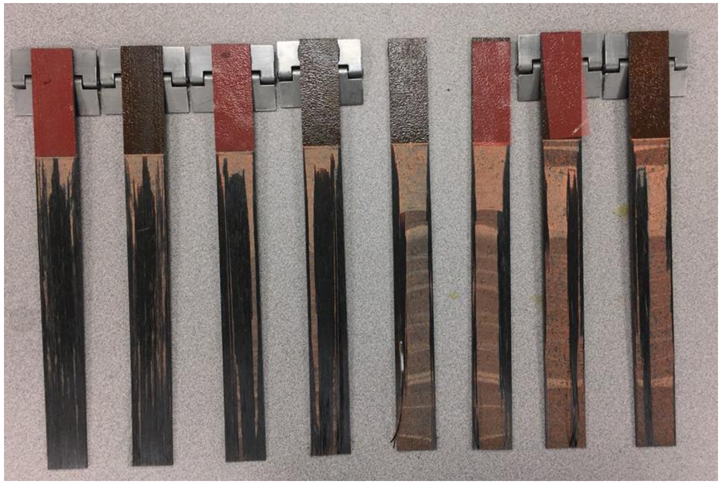
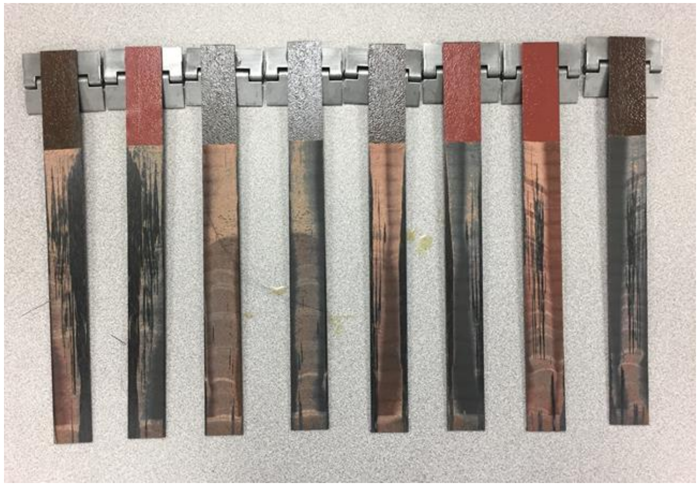
Failure Modes – 0-20% of Baseline

Mixed-mode failure

Variable combination of interlaminer and cohesion



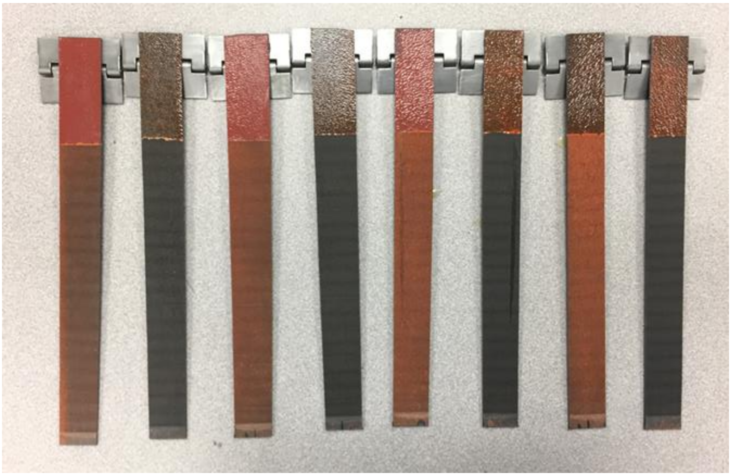
Baseline



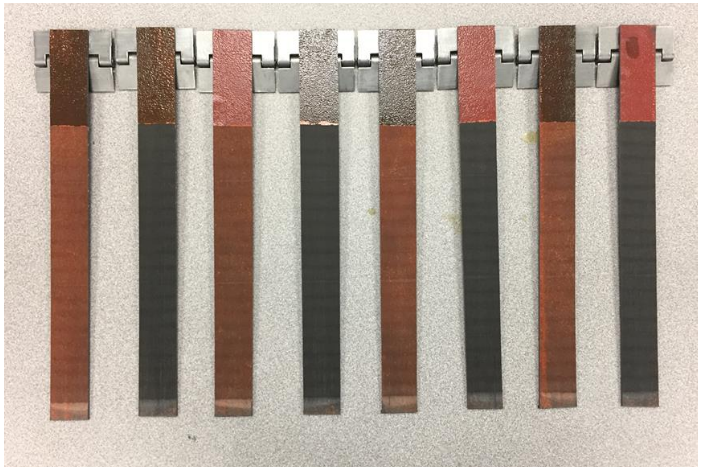
19% Wipe/Sand/Wipe

Adhesion failure

Separates from the surface of adherent



19% Only



19% Wipe

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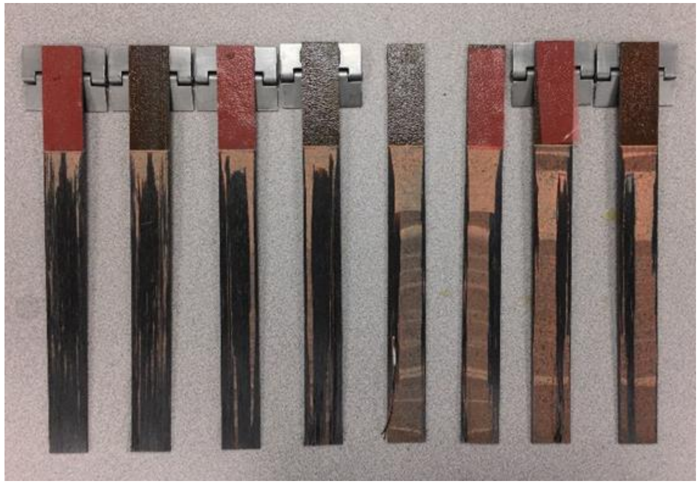
Failure Modes – 45-55% of Baseline

Mixed-mode failure

Variable combination of interlaminer and cohesion

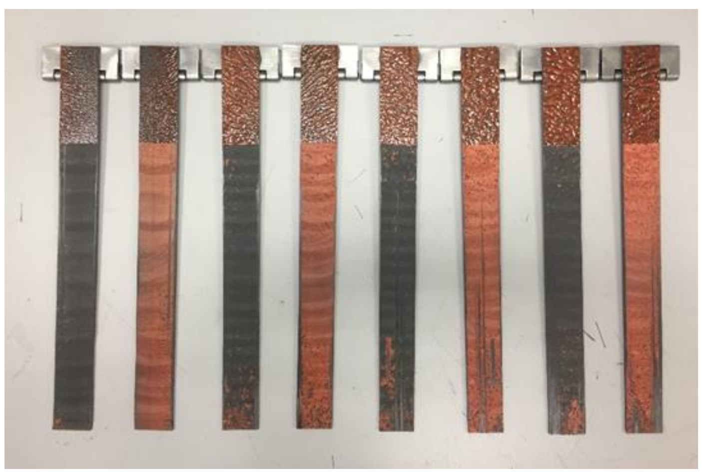


Baseline

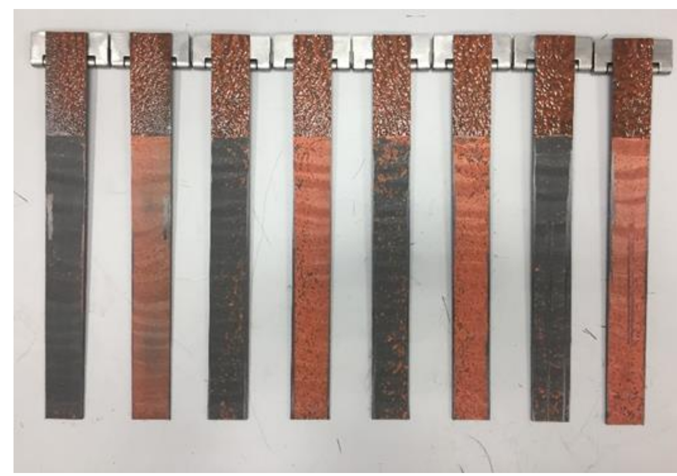
42% Wipe/Sand/Wipe

Adhesion failure

Separates from the surface of adherent



42% Only



42% Wipe

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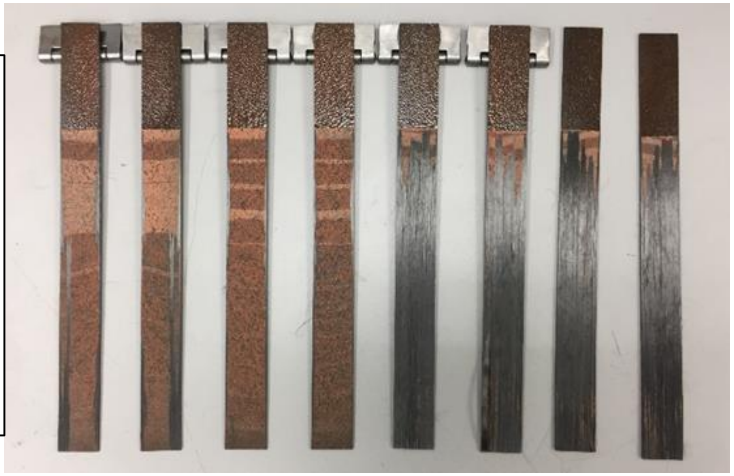
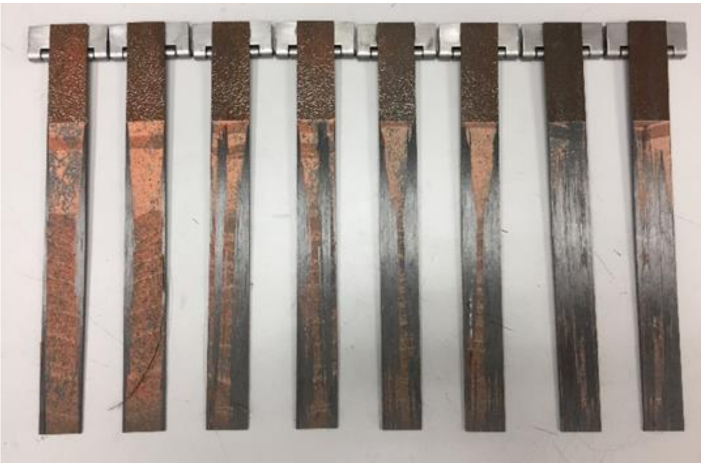
Failure Modes – 70-80% of Baseline

Mixed-mode failure

Variable combination of interlaminer and cohesion



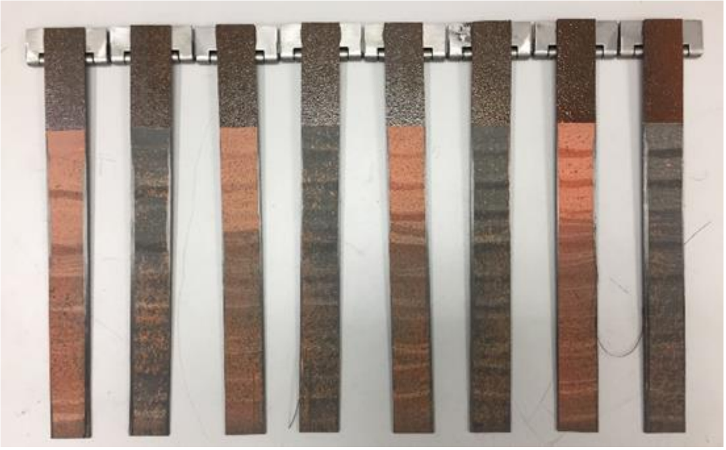
Baseline



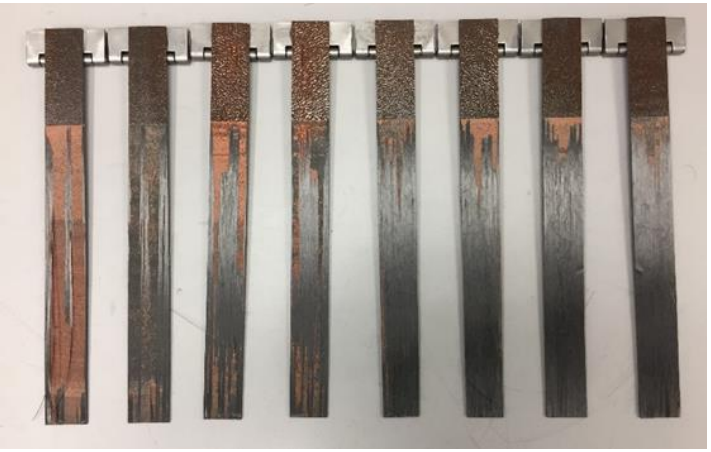
78% Wipe/Sand/Wipe

Adhesion failure

Separates from the surface of adherent



78% Only

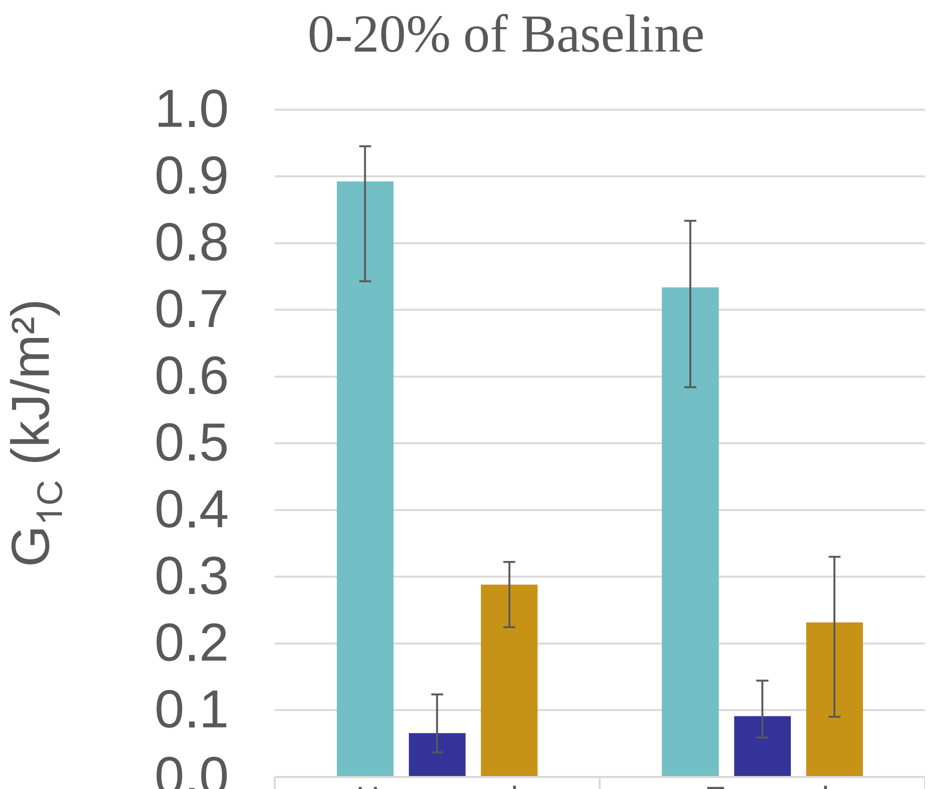


78% Wipe

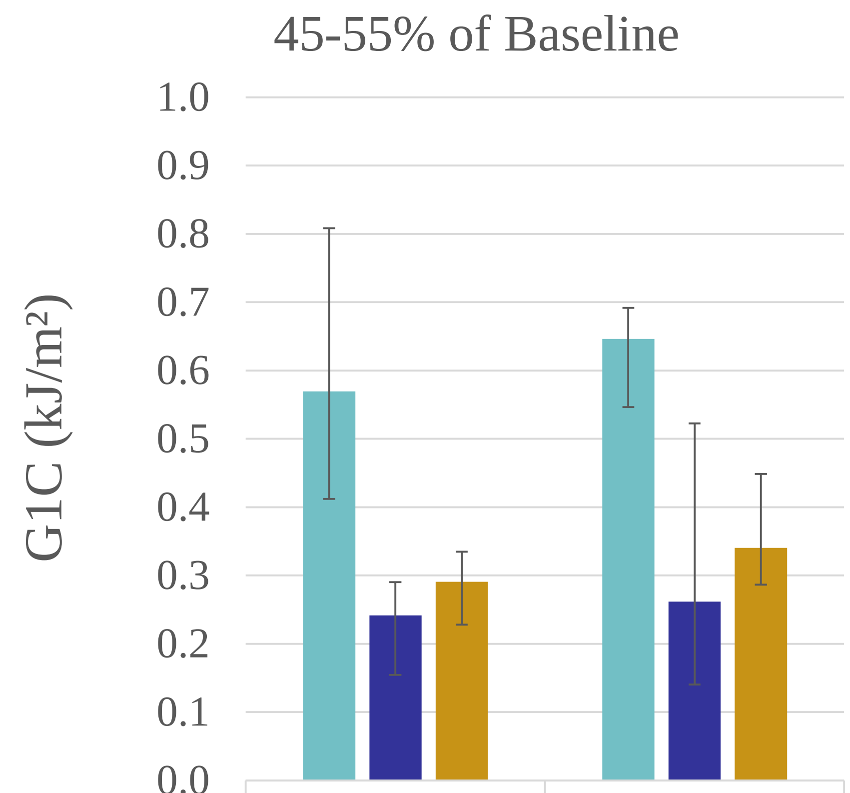
Environmental Aging

- Coupons were exposed to 70°C and 95% rel. humidity
- 8 coupons were manufactured for each set: baseline, contaminated, and wipe/sand/wipe
- 4 coupons from each set were exposed in the environmental chamber and the remaining 4 coupons served as the unexposed set
- After 4 weeks in the environmental chamber, the exposed samples were removed from the chamber and DCB tests were performed.

Results - Post Environmental Exposure



	Unexposed	Exposed
Baseline	0.892	0.734
Contaminated	0.065	0.091
WSW	0.288	0.232



	Unexposed	Exposed
Baseline	0.57	0.65
Contaminated	0.24	0.26
WSW	0.29	0.34

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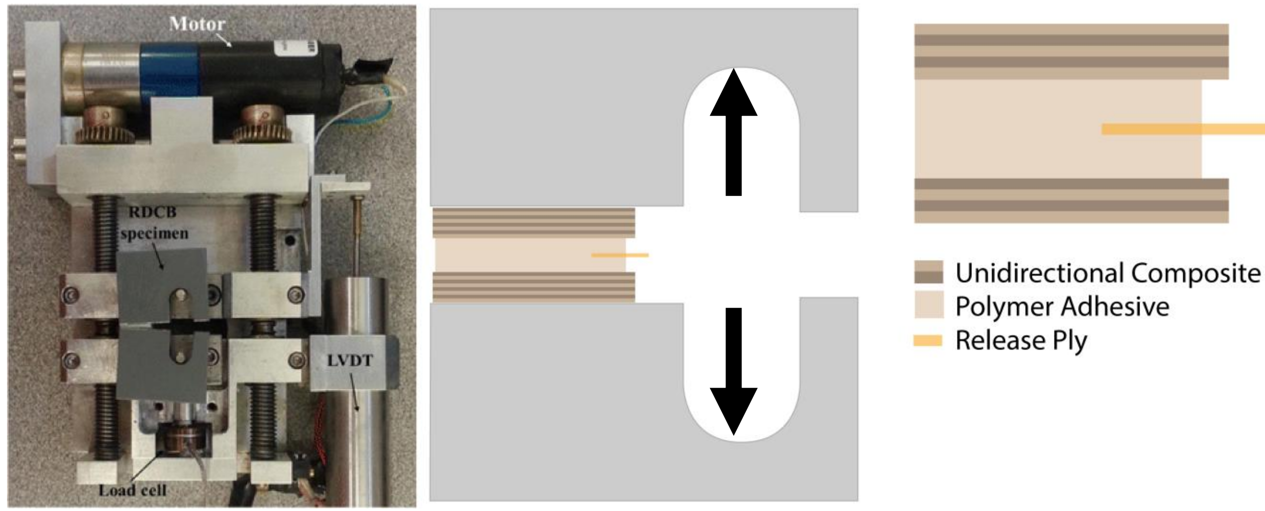
In-situ Testing

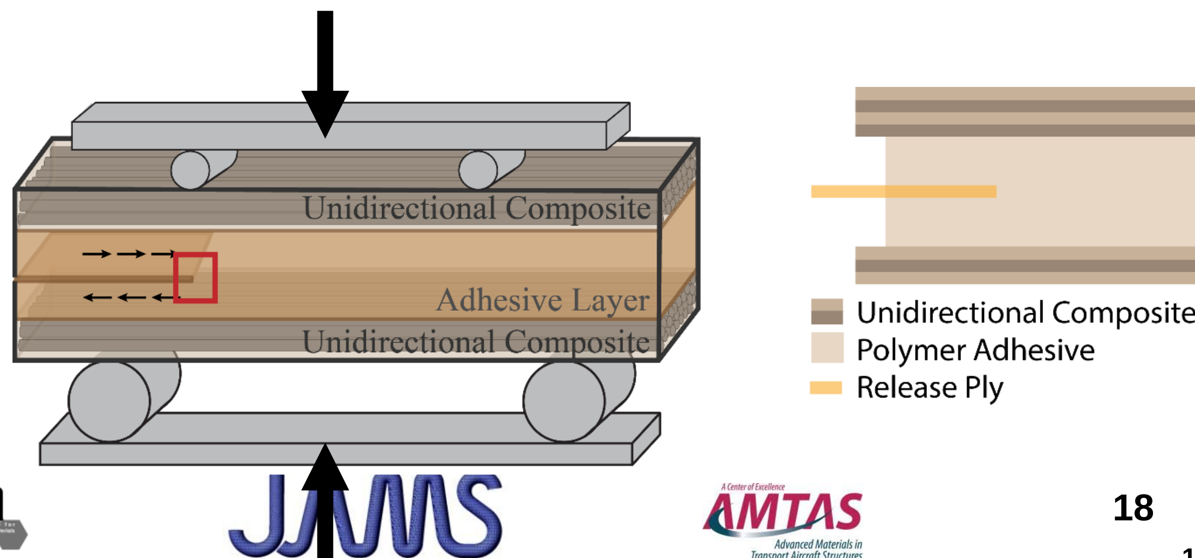
Combined Load Frame and Electron Microscopy

Test Development

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

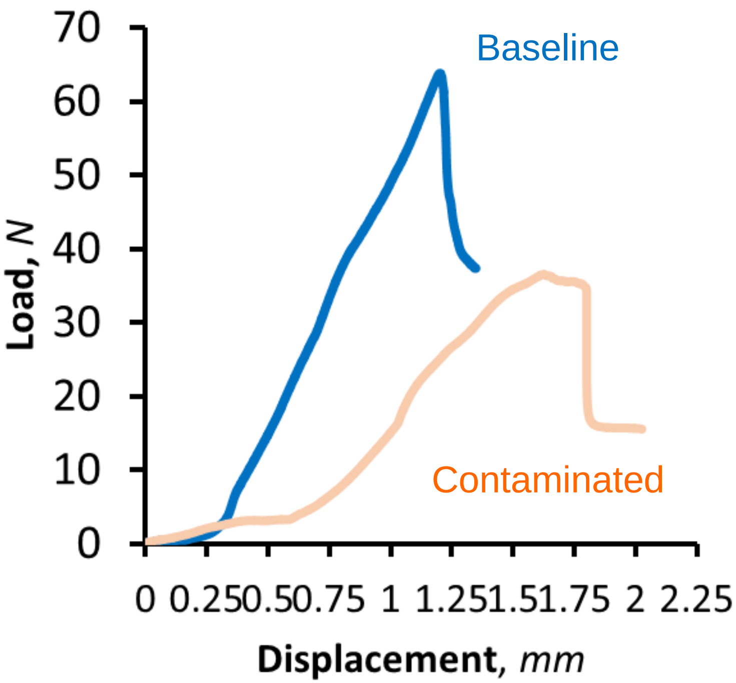


µENF (End Notch Flexure)
Assesses the mechanisms of mode II fracture. Fixture was designed based of traditional ENF testing of composite bonds

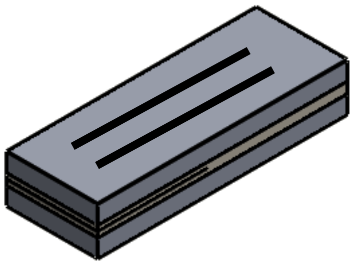


In-situ Testing

Combined Load Frame and Electron Microscopy



Specimen Details



Baseline

L/W: 40mm x 10mm

thickness: 5.2 mm

Pre-crack: 8 mm

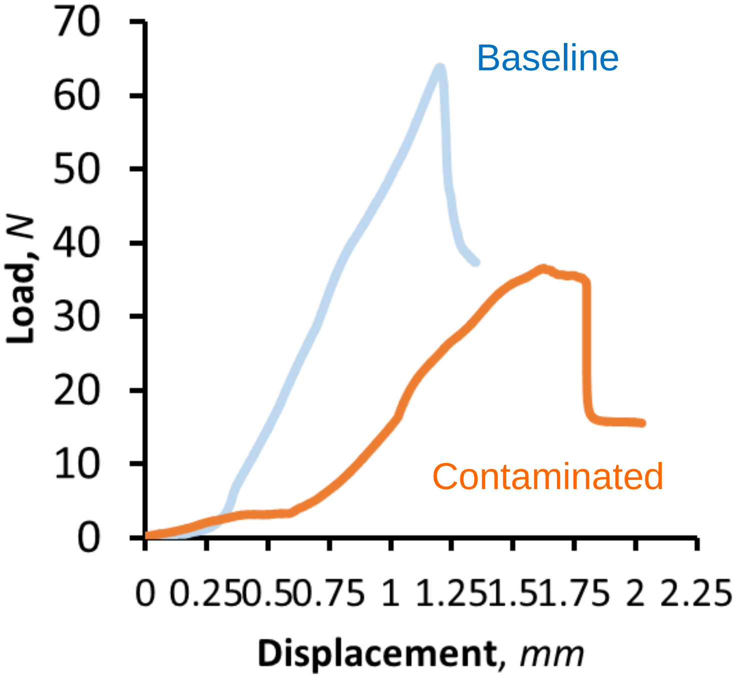
10 layer unidirectional composite panels

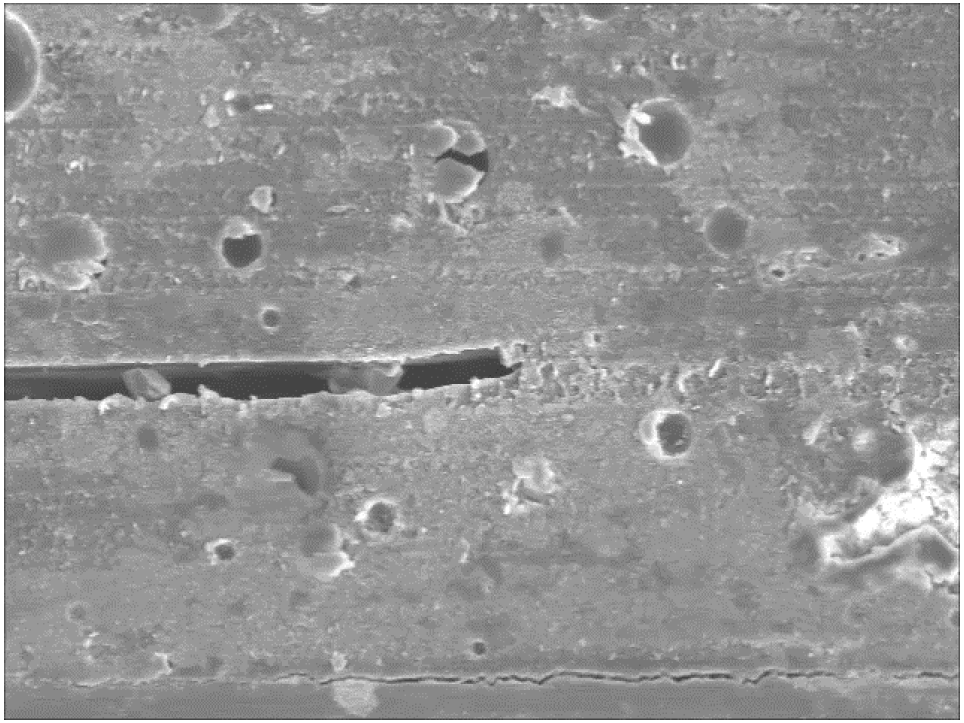
Observations

- 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

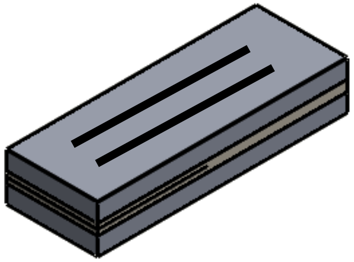
In-situ Testing

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

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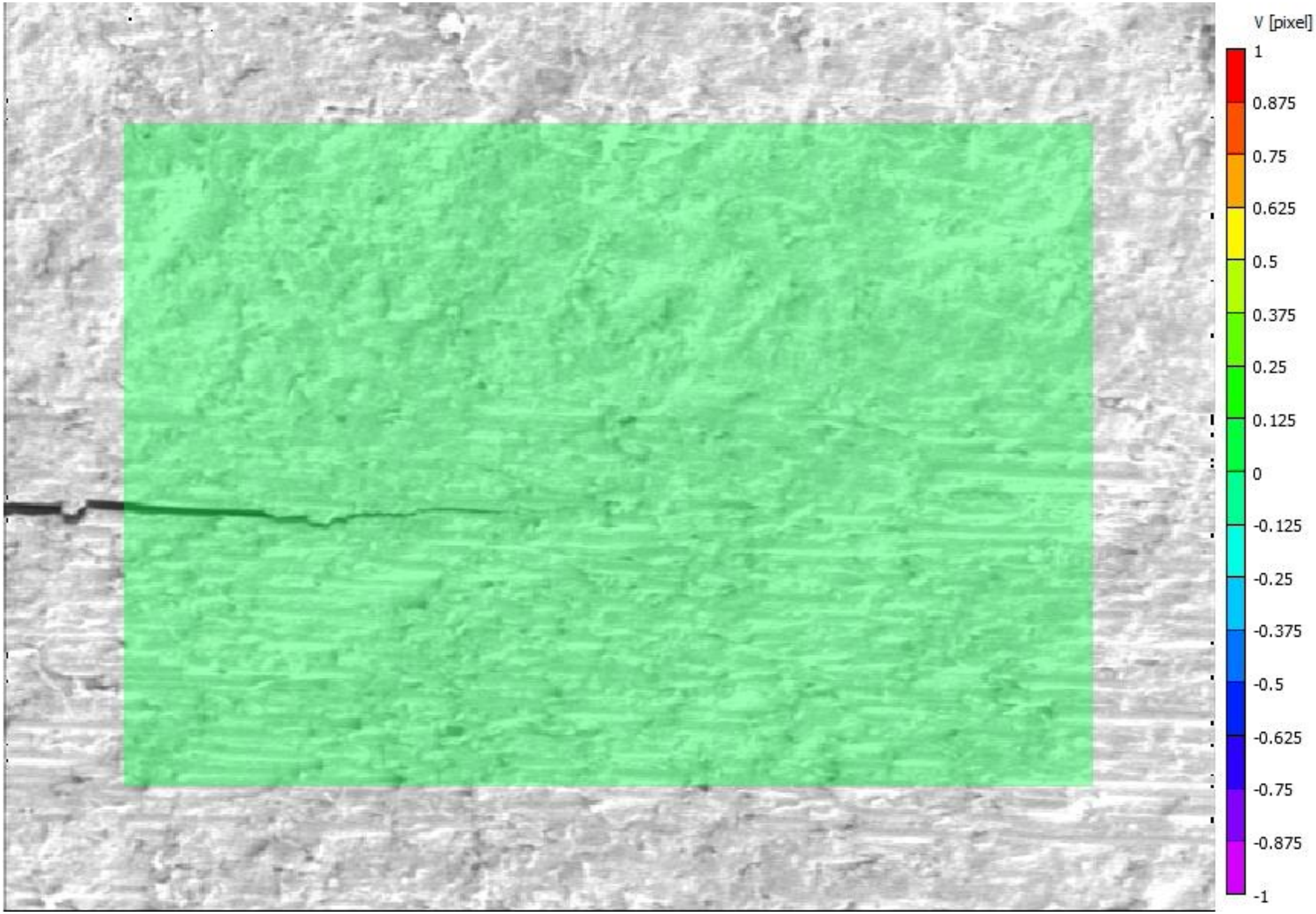


V [pixel]
1
0.875
0.75
0.625
0.5
0.375
0.25
0.125
0
-0.125
-0.25
-0.375
-0.5
-0.625
-0.75
-0.875
-1

In-situ Testing

Combined Load Frame and Electron Microscopy

Complications with in situ testing

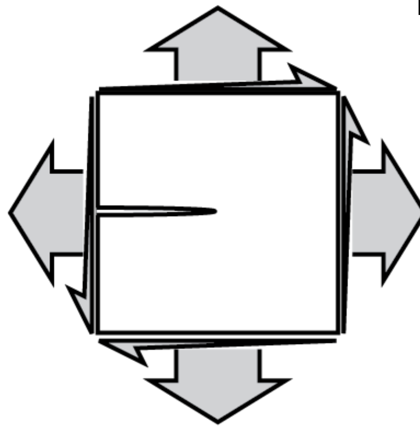
Small sample sizes and edge effects

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.

<u>Therefore if we know the displacements we can solve for the KI value.</u>

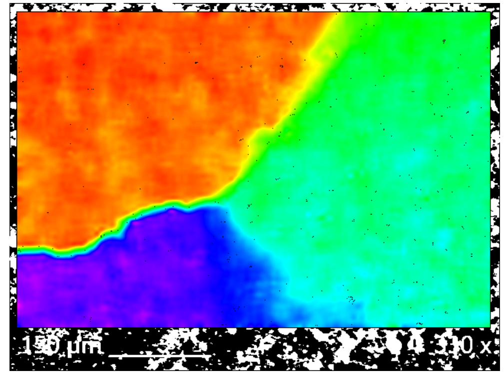


From LEFM

$$\sigma_{xx} = \frac{K_I}{\sqrt{2\pi r}} \cos\frac{1}{2}\theta \left(1 - \sin\frac{1}{2}\theta \sin\frac{3}{2}\theta\right)$$

$$\sigma_{yy} = \frac{K_I}{\sqrt{2\pi r}} \cos\frac{1}{2}\theta \left(1 + \sin\frac{1}{2}\theta \sin\frac{3}{2}\theta\right)$$

$$\sigma_{xy} = \frac{K_I}{\sqrt{2\pi r}} \sin\frac{1}{2}\theta \cos\frac{1}{2}\theta \cos\frac{3}{2}\theta$$



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

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

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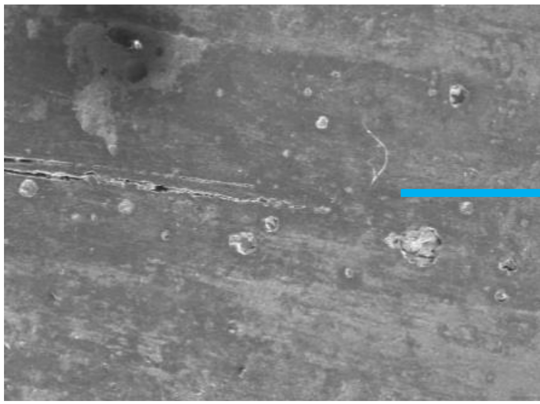
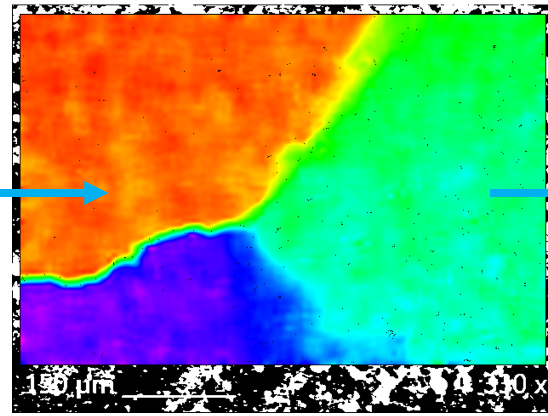
In-situ Testing

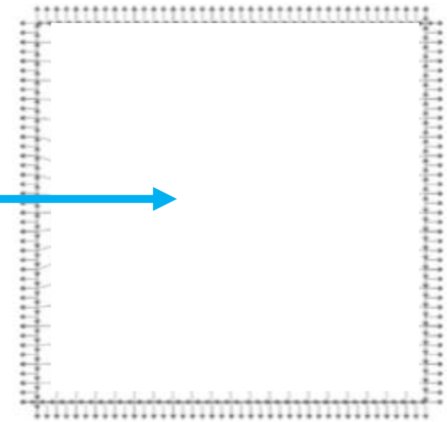
Combined Load Frame and Electron Microscopy



In situ Microscopy



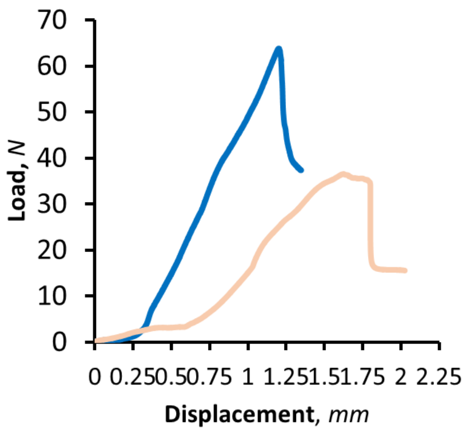
Digital Image Correlation



Digitized Displacements

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

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



Summary

- A contamination procedure was developed using a customized contamination rig with Frekote to create a scalable weak bond. The weak bonds can be used to evaluate surface prep techniques and potentially NDI methods using three levels of contamination.
- Mitigation approaches included solvent wiping and solvent wiping/sanding/solvent wiping. Results from these tests indicated that wiping alone did not improve the bond strength, however, there was significant improvement with the wiping/sanding/solvent wiping.
- A platform for testing mini-DCBs has been established with the intent of quantifying fracture toughness via DIC strain mapping within an SEM. In addition, crack-tip propagation phenomena can be investigated to gain better understanding of the failure modes.

Path Forward

- 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.

- SEM image stabilization will be sought to improve accuracy of DIC techniques for fracture toughness quantification. Pre-crack propagation will be studied for detectable patterns in baseline and contaminated specimen.

Test SAMPLES

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- 10 layers – Unidirectional Prepreg carbon fiber (Toray)
- 3M AF555M film adhesive:
  - 2 Layers
  - 2 Layers contaminated
  - 4 Layers
- 7mm by 70mm mini DCBs with 20mm pre-crack
- Frekote Mold Release Agent (contaminant)

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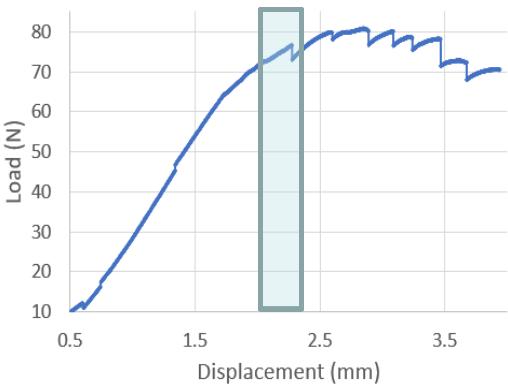
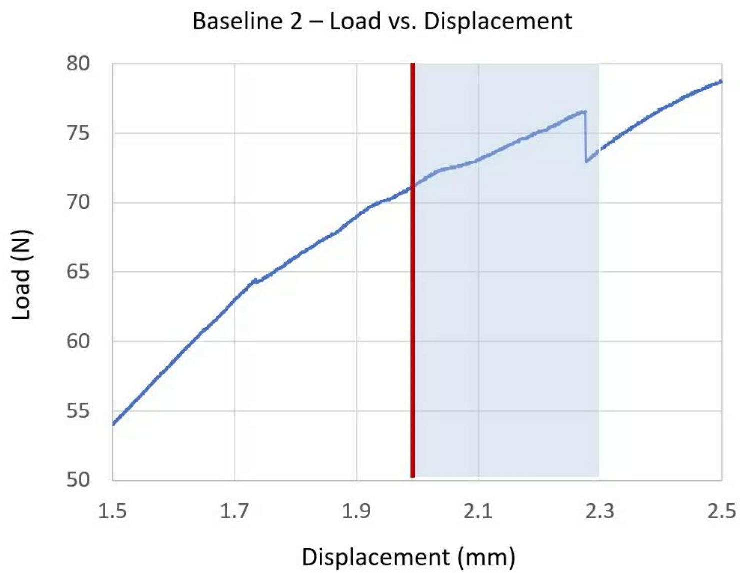
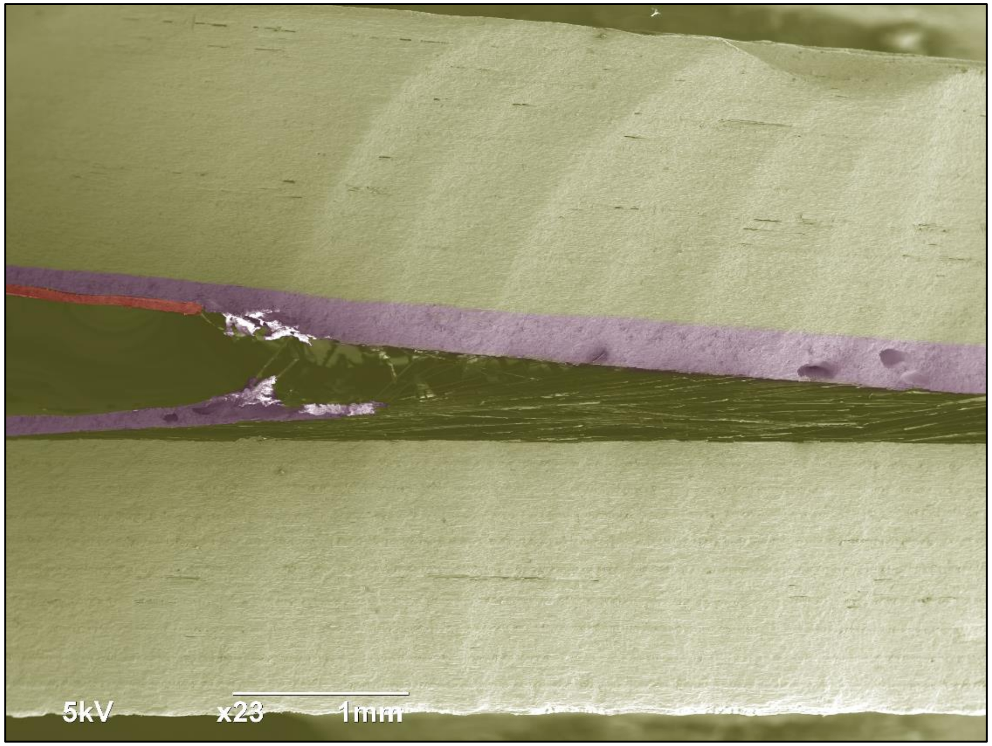
Baseline 2 layers

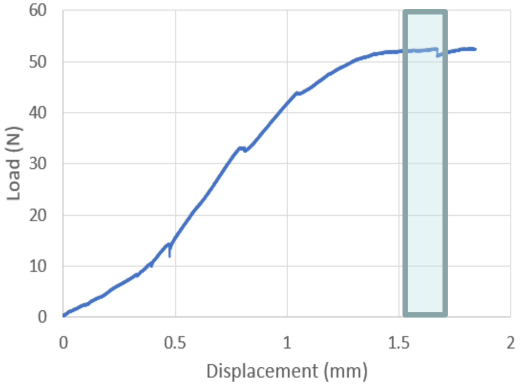


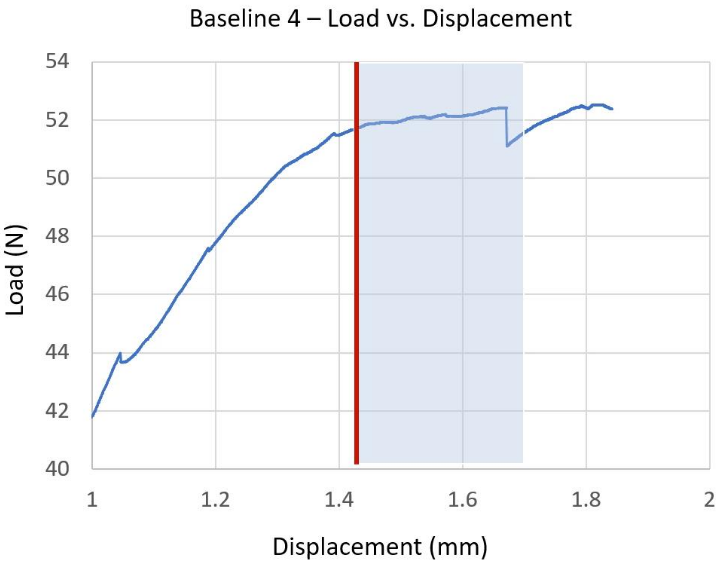


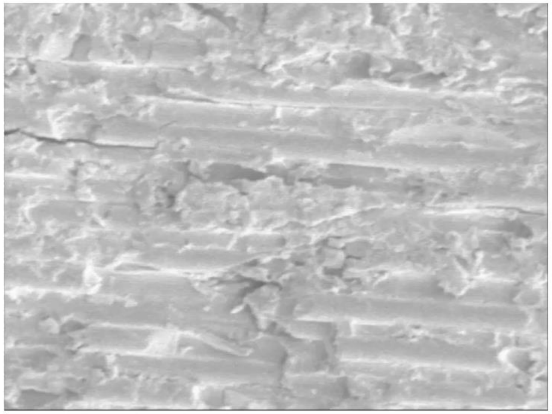
Baseline 2 layers



Baseline 4 layers







Baseline 4 layers

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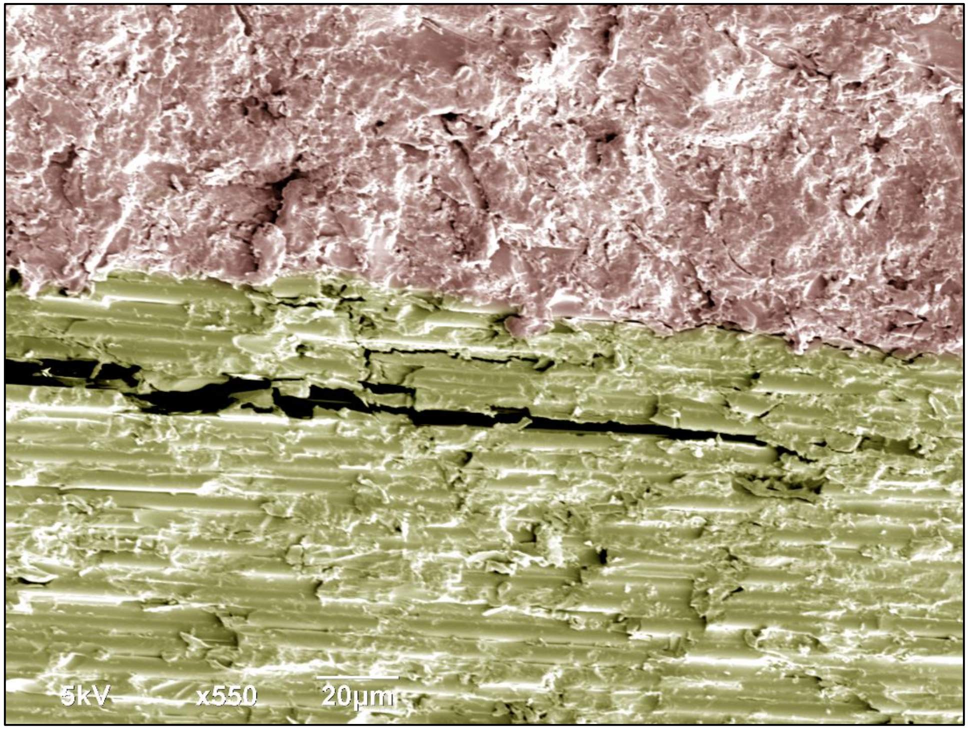

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Baseline 2 layers (contaminated)

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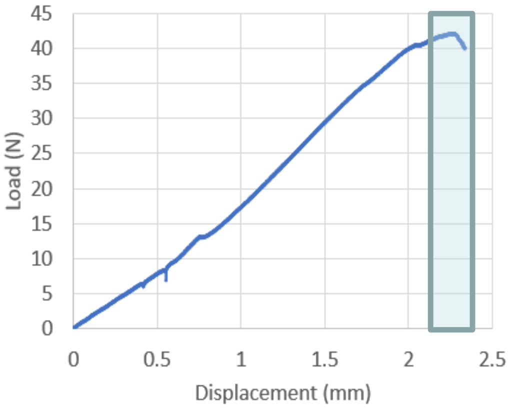

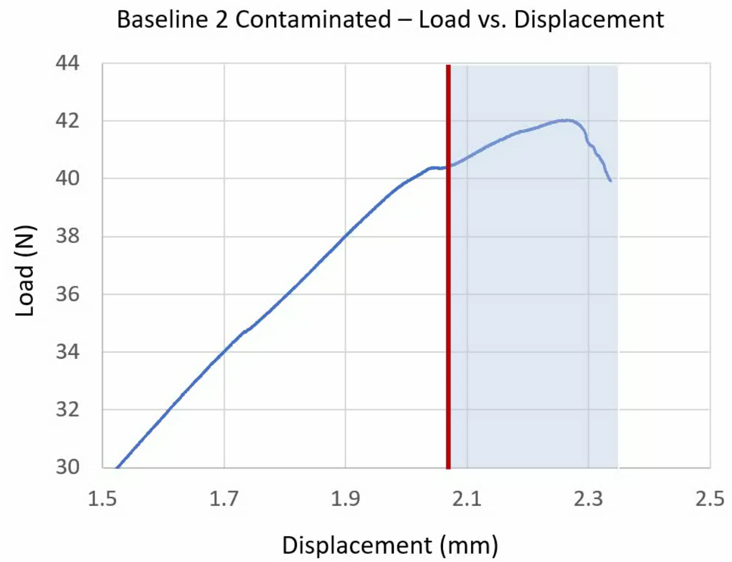

Baseline 2 layers (contaminated)

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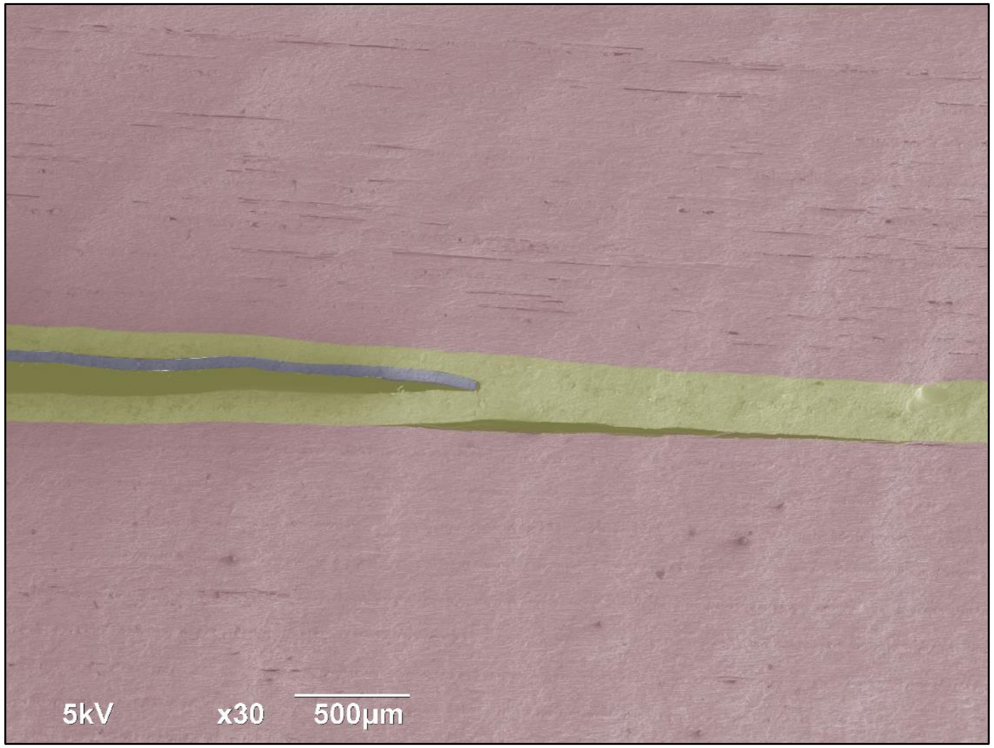

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CMH-17 Support

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

Advantages	Disadvantages
Bonded Joints	
Small stress concentration in adherends; stiff connection; Excellent fatigue properties; No fretting problems; Sealed against corrosion; Smooth surface contour; Relatively lightweight; Damage tolerant	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
Bolted Joints	
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	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

CMH-17 Bonding Process Task Group

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 Sponsor

Margaret Roylance – M&P

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

CMH-17 Bonding Process Task Group

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.

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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

CMH-17 Support

Using online forums to organize CMH-17 content