



# Effect of Surface Contamination on Composite Bond Integrity and Durability

Gabriela Gutierrez-Duran & Dr. Benjamin Boesl  
Florida International University



Contact: [mcdaniel@fiu.edu](mailto:mcdaniel@fiu.edu) Ph: (305) 348-6554

# 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

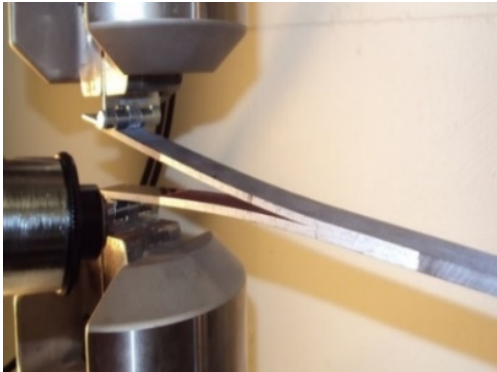
- **FAA Technical Monitor**

- Ahmet Oztekin

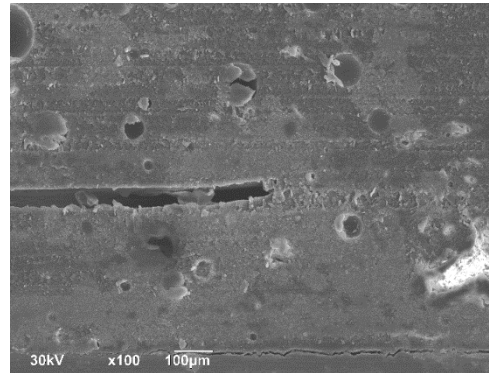
- **Industry Participation**

- Exponent, 3M, Embraer, BTG Labs

# Research Tasks FY18-19



**Investigation of contaminated bondlines on the macro scale**



**Investigation of contaminated bondlines on the micro scale**

Chapter 5 Materials and Processes - The Effect of Variability on Composite Properties

1. Introduction
2. Purpose
3. Scope
4. Constituent Materials
5. Processing of Product Forms
6. Shipping and Storage Processes
7. Construction Processes
8. Cure and Consolidation Processes
9. Assembly Processes
10. Process Control
11. Preparing Material and Processing Specifications

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.

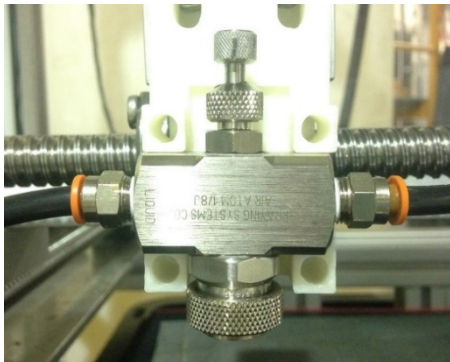
**Support of CMH-17 Handbook**

# Road Map of Contamination Studies

## Discrete vs Continuous Approaches



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

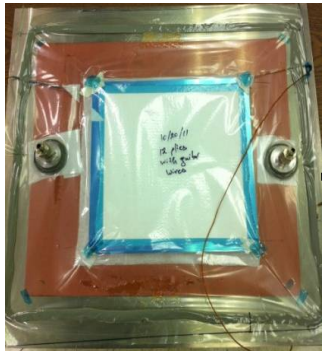


Continuous Method	Static	Exposed	Fatigue
Baseline	DCB	<b>DCB</b>	<b>DCB</b>
~10%	DCB	<b>DCB</b>	<b>DCB</b>
~50%	DCB	<b>DCB</b>	<b>DCB</b>
~75%	DCB	<b>DCB</b>	<b>DCB</b>

- **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

# Manufacturing of Bonded Systems

## Fabrication of Laminates

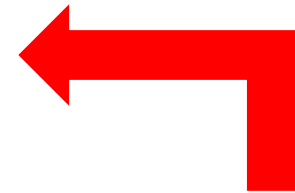


## Laminate Cure



(Cure Cycle @350F)

## Bonding of Laminates



### KEY QUESTION

What happens to bonded joint's strength when contamination occurs, if known can it be mitigated?

## Adhesive Bond Strength Testing



## Preparing/Cutting Samples



## Adhesive Cure



(Cure Cycle @350F)

### CAUSES

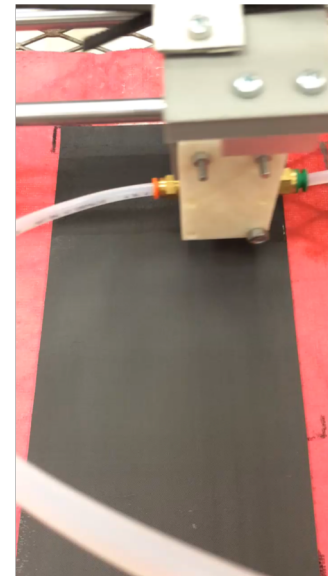
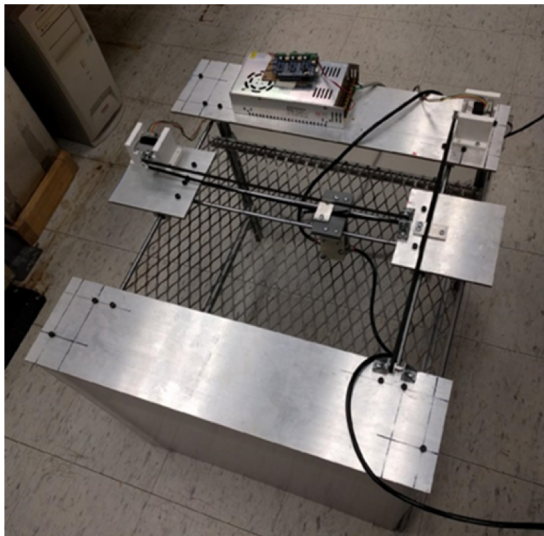
Contamination can occur in a manufacturing setting from oil on hands, mold release, leakage/spillage, etc.

## 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.





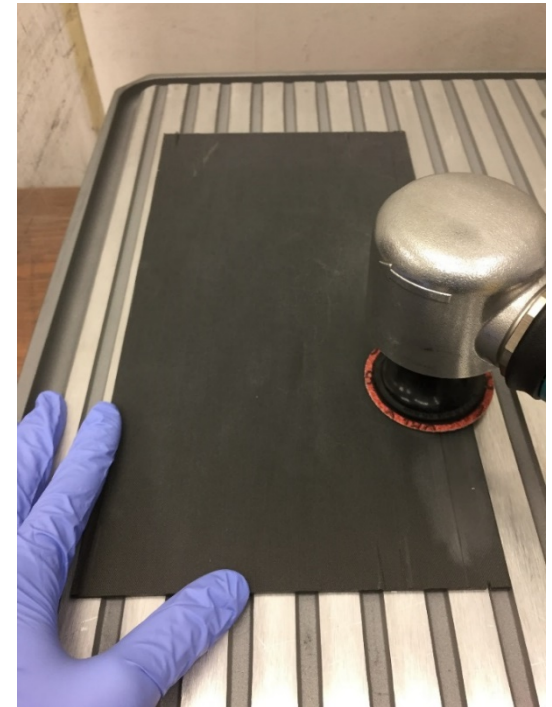
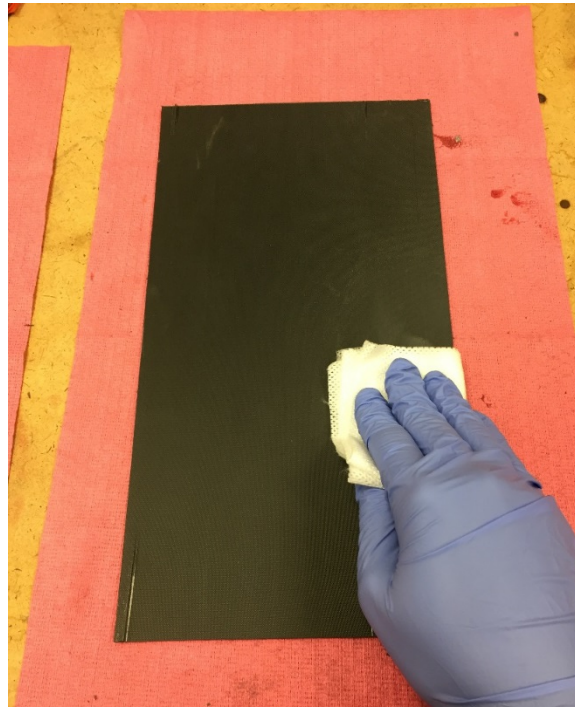
## Calibration of Contamination Levels

---

- 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

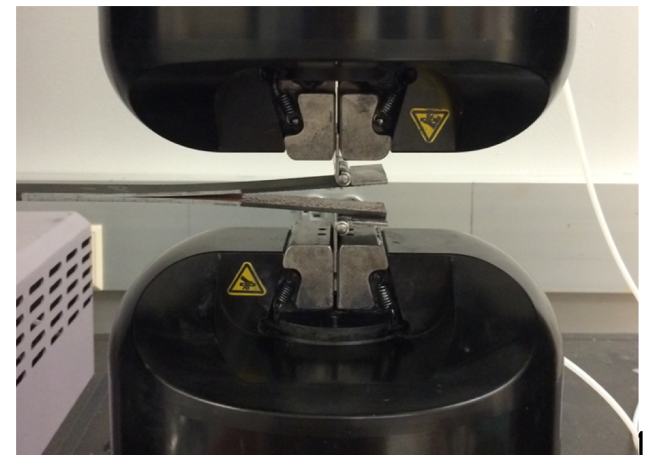
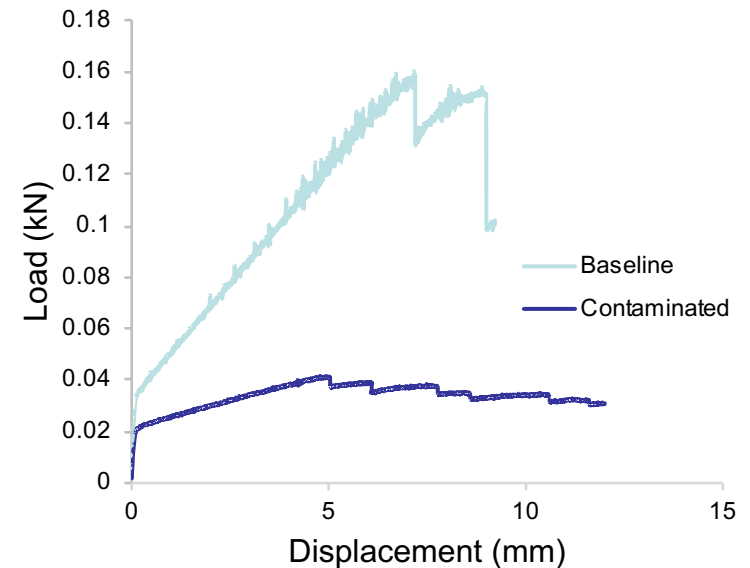
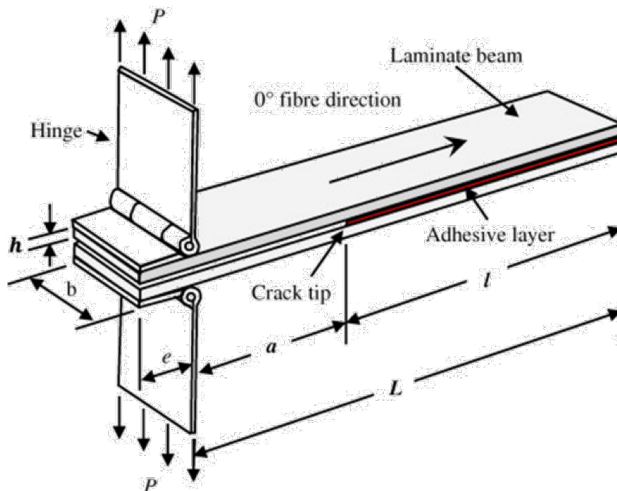


## Bond Quality Evaluation

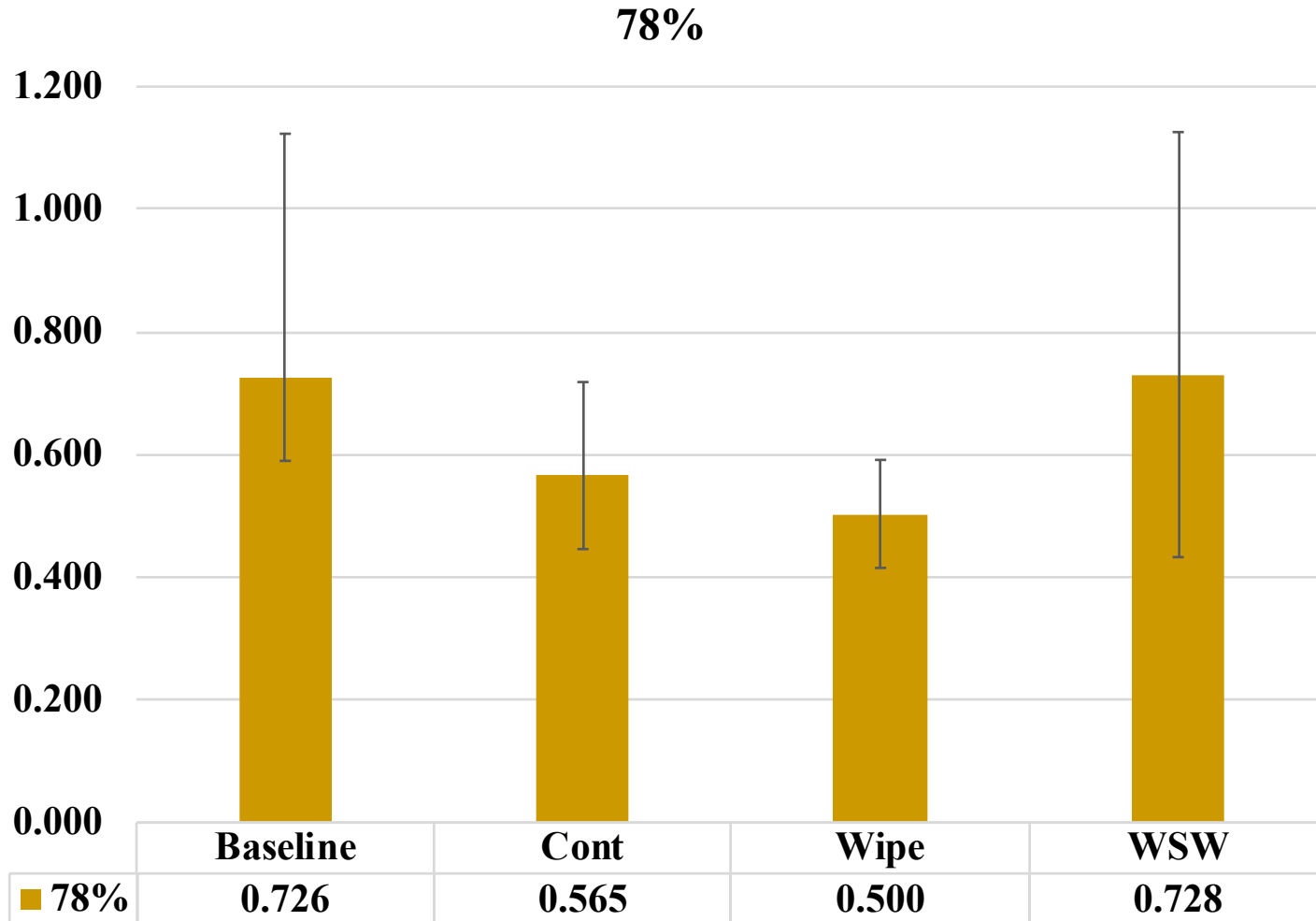
- Dual Cantilever Beam Testing
  - Measures interlaminar fracture toughness
- Fracture toughness provides a measure of composite strength
  - The critical energy a material may absorb before failure and resistance to delamination

$$- G_{1C} = \frac{3P\delta}{2b(a+|\Delta|)}$$

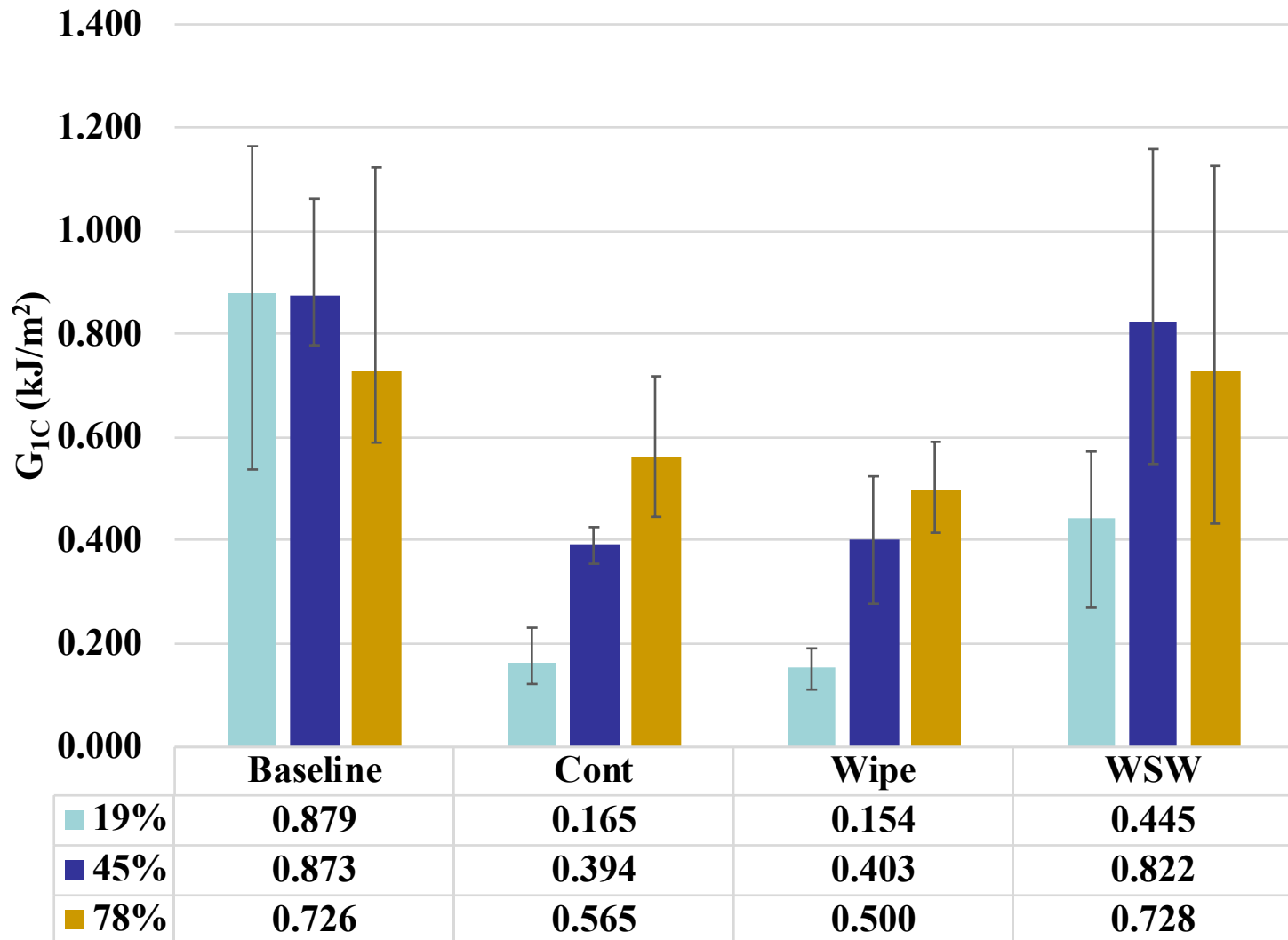
- Use of MTS machine to measure displacement



## Results of Mitigation Approaches



## Summary of Mitigation Results



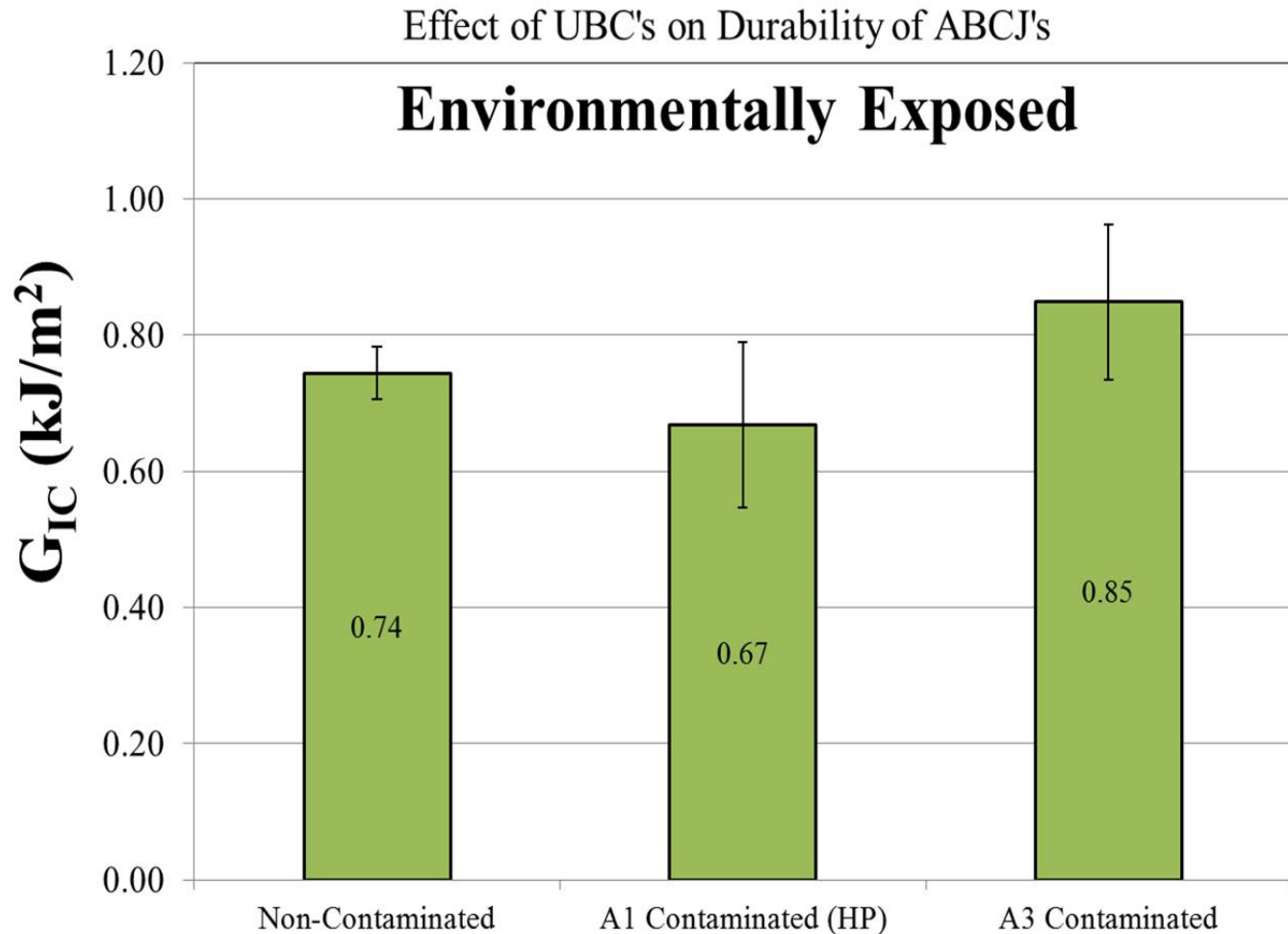
## Durability Characterization – Environmental Aging

- Why is it important?
  - Composite laminates not only go through contamination effects but are also exposed to other conditions when they've been placed on the plane
- Specimens are environmentally aged in a Thermotron 2800 Environmental chamber in an unstressed condition
  - Specimens are subjected to harsh environments i.e. controlled temperature and humidity conditions at 70°C and 95% RH
    - The fluid selected for the aging process is di-ionized water



## Durability Results - Stamp approach

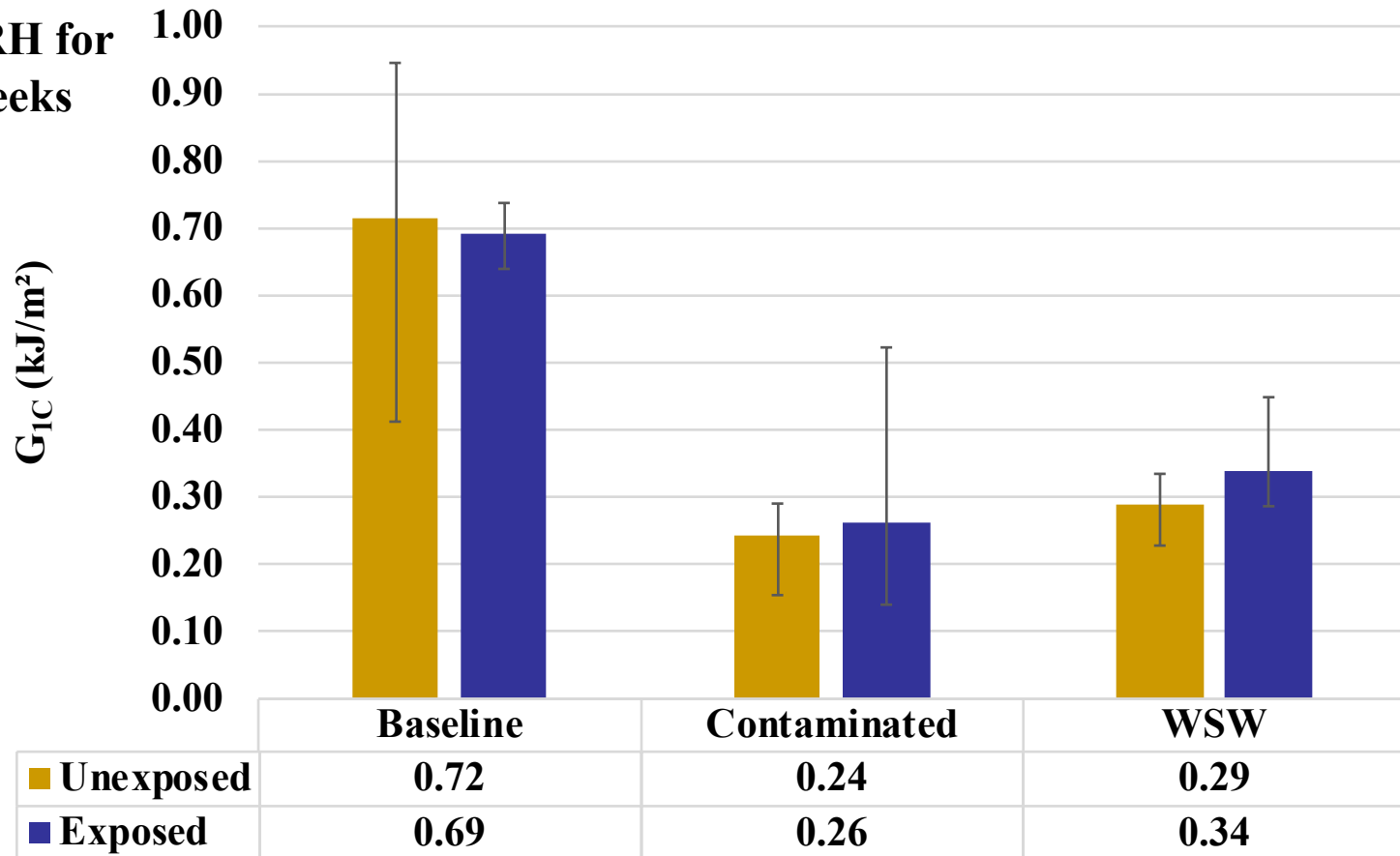
**70 C and  
95% RH for  
4 Weeks**



# Durability Results – Uniformly Sprayed Approach

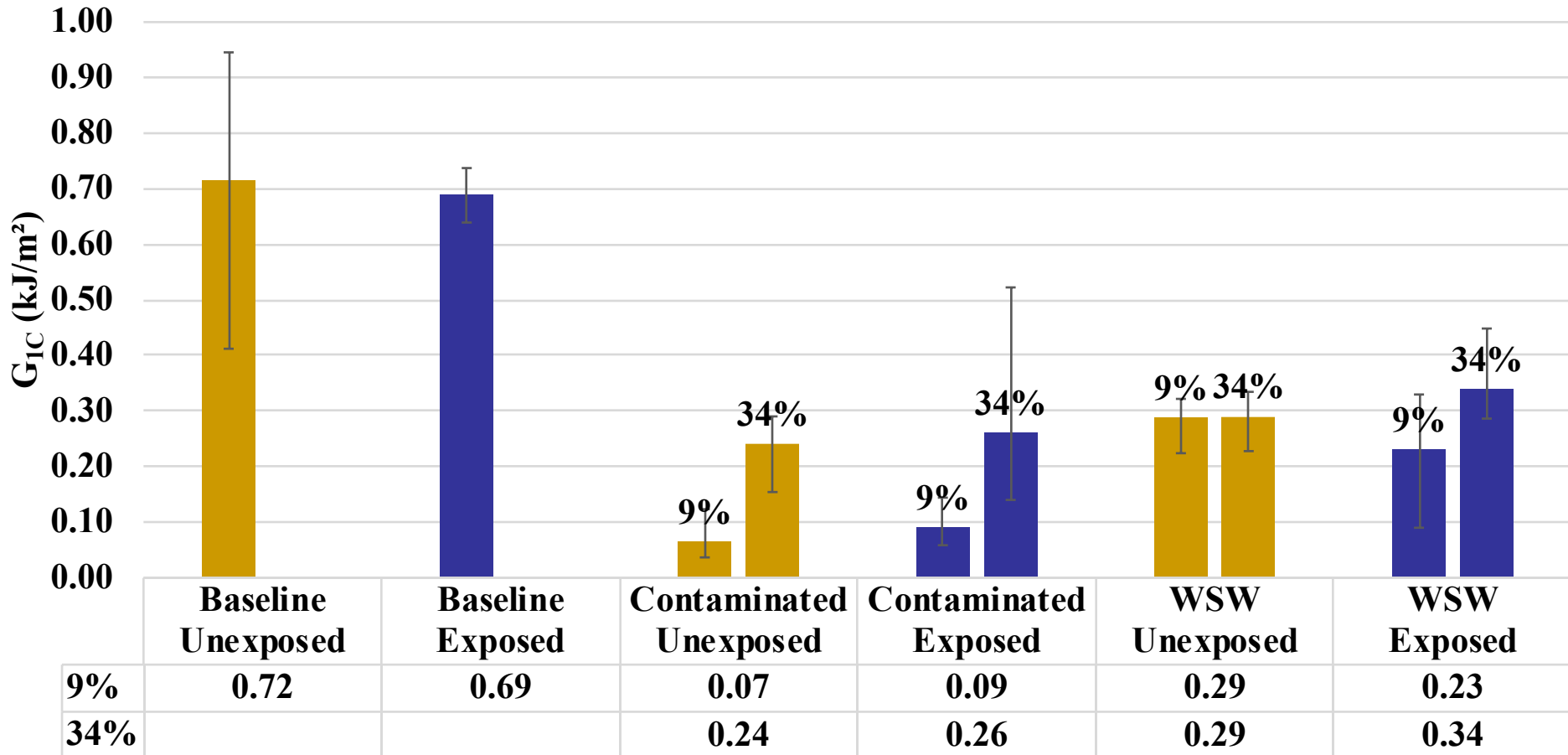
34%

70 C and  
 95% RH for  
 4 Weeks



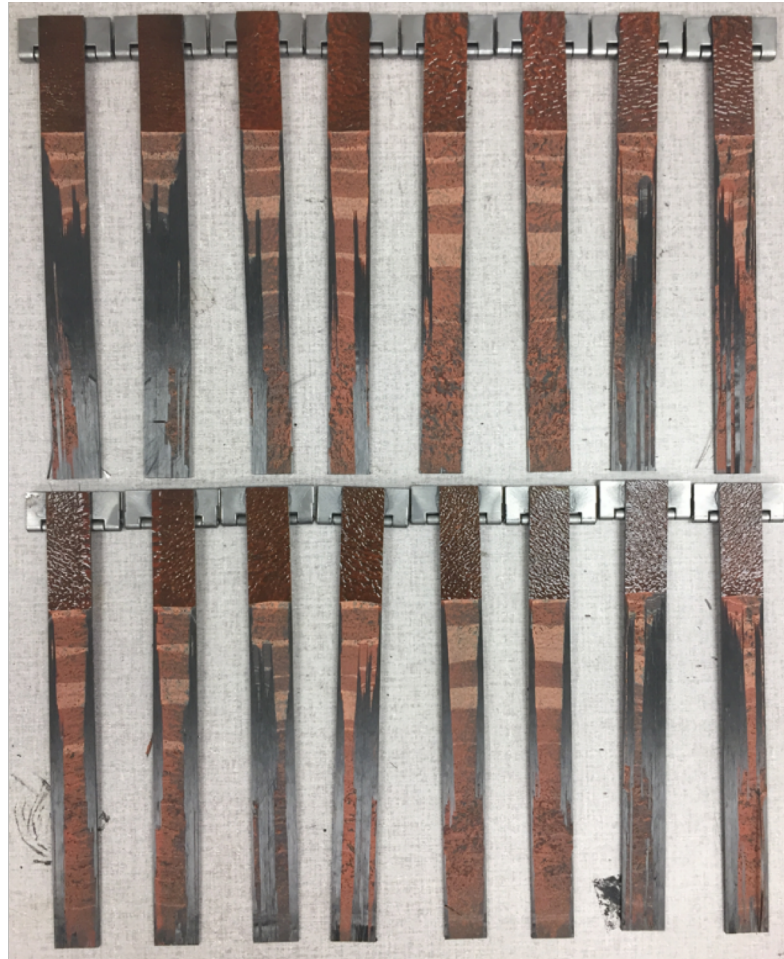


## Summary of Durability Results



## Failure Modes – Uniformly Sprayed Approach (Baseline)

**Unexposed**



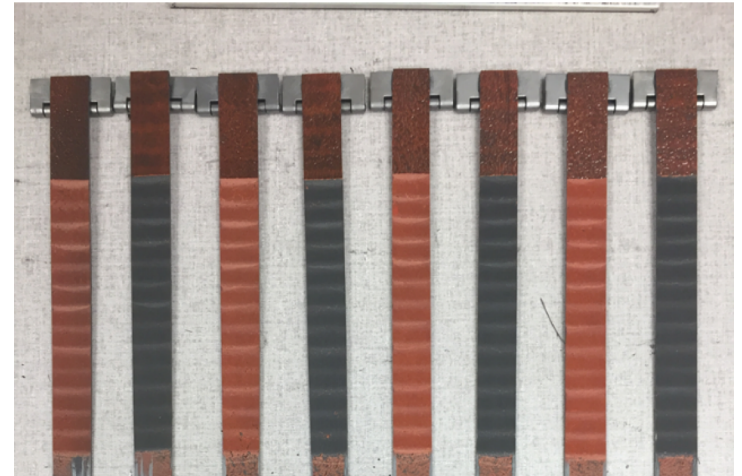
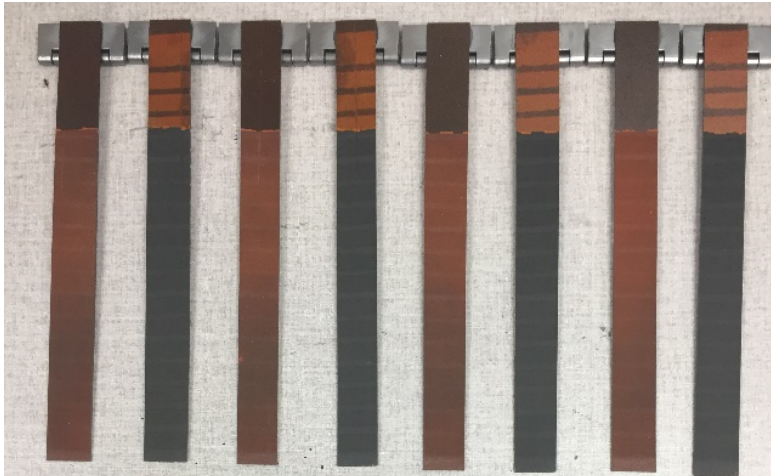
**Exposed**

# Failure Modes – Uniformly Sprayed Approach (Contaminated)

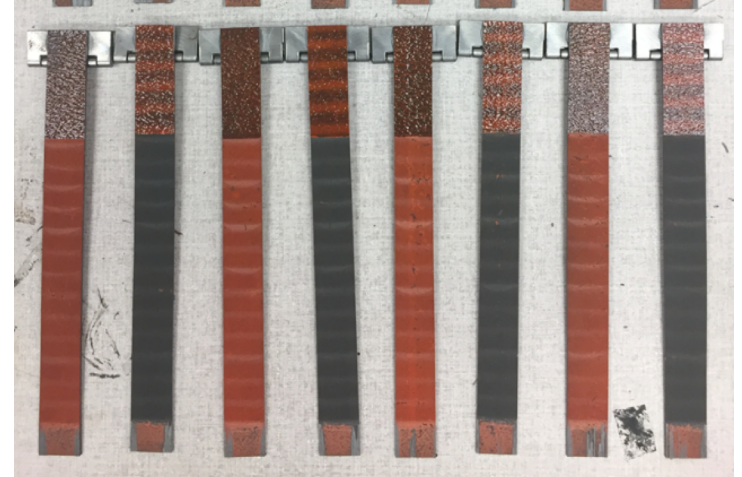
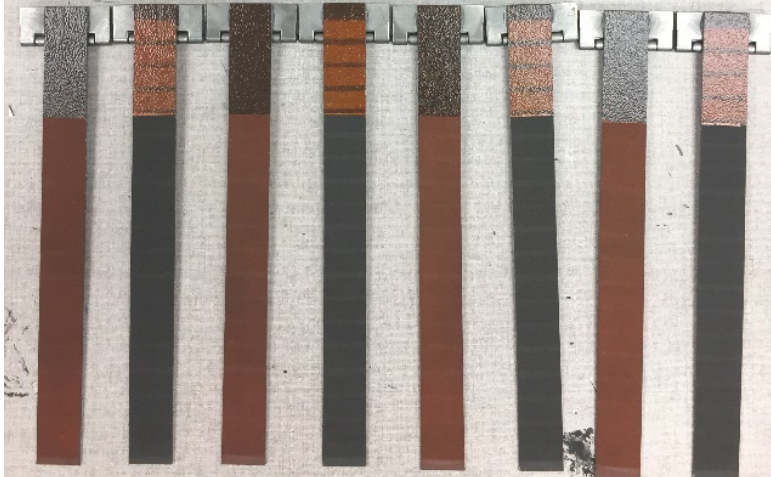
9%

34%

Unexposed



Exposed

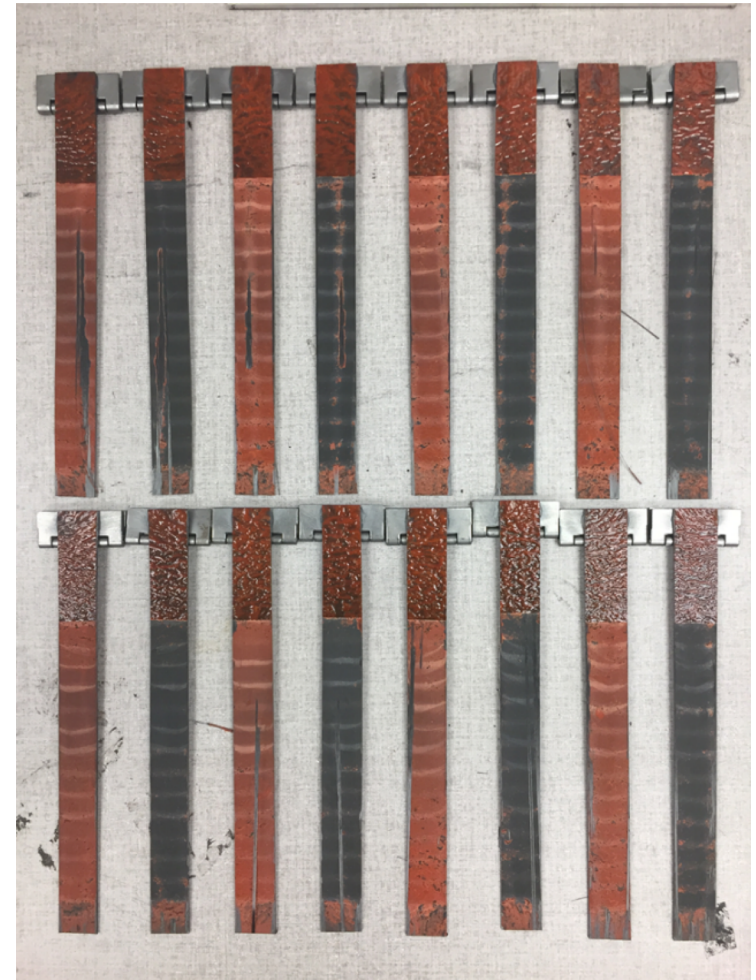
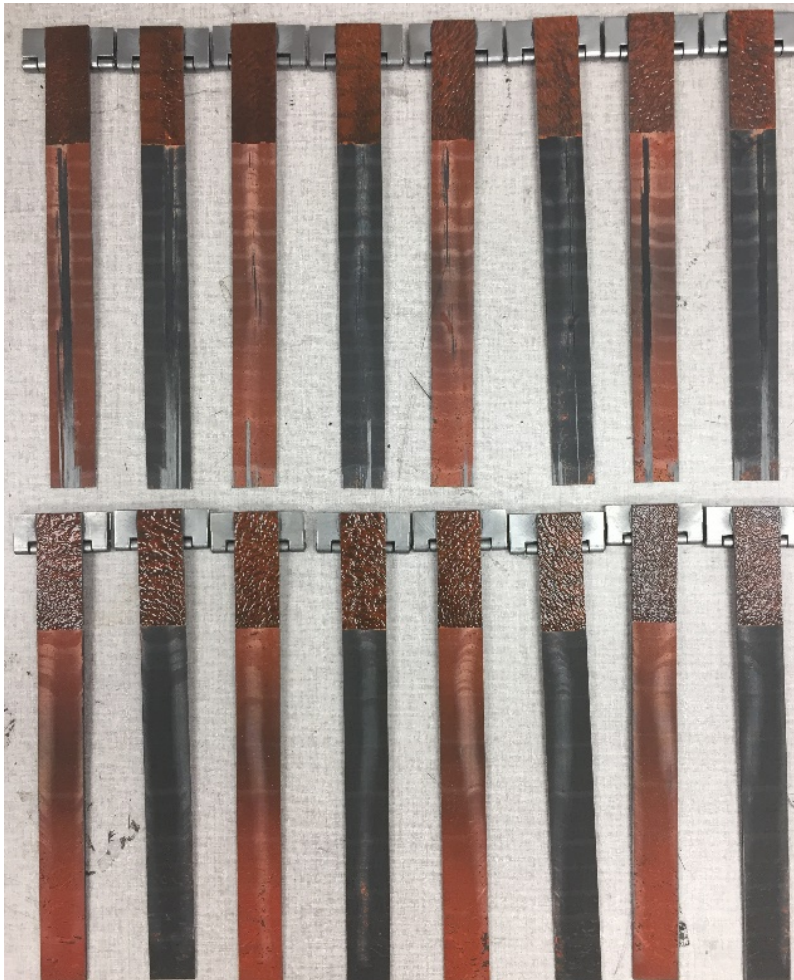


## Failure Modes – Uniformly Sprayed Approach (Wipe Sand Wipe)

9%

34%

Unexposed



Exposed

# Failure Modes – Uniformly Sprayed Approach

**Baseline**

**Contaminated**

**Wipe Sand Wipe**

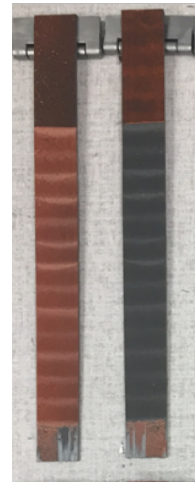
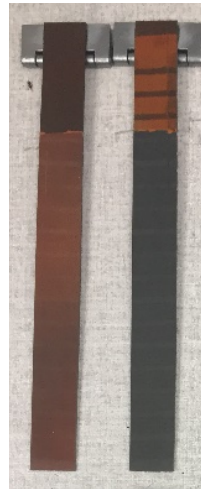
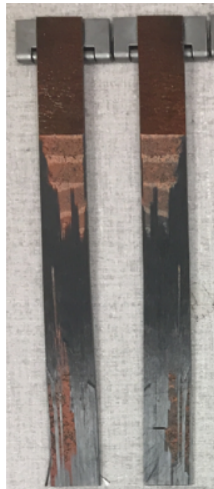
9%

34%

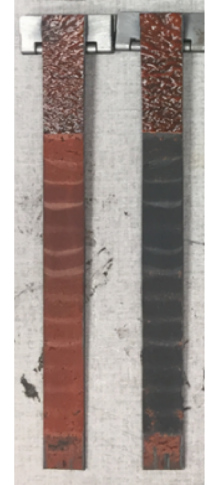
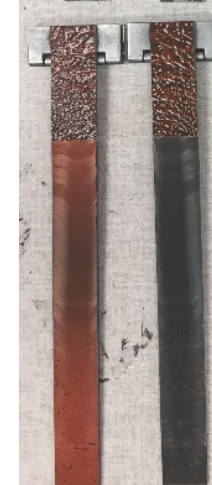
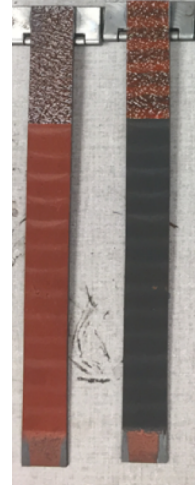
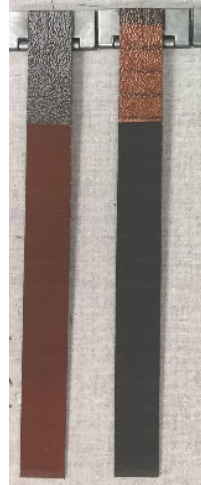
9%

34%

**Unexposed**



**Exposed**



## Summary

---

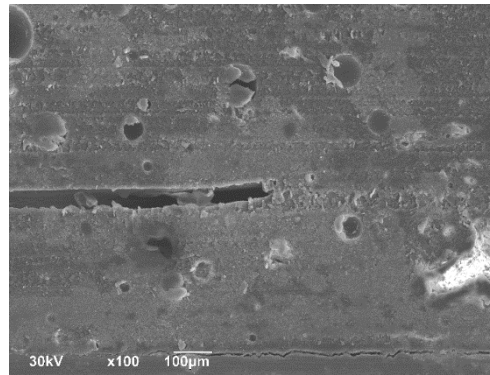
- A contamination procedure was developed using and Frekote to develop a scalable and repeatable weak bond. The weak bonds can be used to evaluate surface prep techniques and potentially NDI methods.
- Repeatable weakened bonds were obtained using a customized contamination rig for three levels of contamination (~10, ~50 and 75% bond strength).
- 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.
- Durability of both contaminated samples and contaminated with mitigation approaches were evaluated with both static loading and environmental exposure. There was no apparent combination effect from samples both contaminated and exposed.

# Research Tasks FY18-19



## Investigation of contaminated bondlines on the macro scale

Contaminated DCB coupons and coupons treated with the mitigation methods will be exposed to fatigue loading to determine effects on durability.



## Investigation of contaminated bondlines on the micro scale

In-situ testing will be used to evaluate aspects of failure that include, environmental exposure, contamination and bondline thickness. Efforts will also be made to quantify the fracture toughness using DIC to estimate the strain field around the crack tip.

Chapter 5 Materials and Processes - The Effect of Variability on Composite Properties

1. Introduction
2. Purpose
3. Scope
4. Constituent Materials
5. Processing of Product Forms
6. Shipping and Storage Processes
7. Construction Processes
8. Cure and Consolidation Processes
9. Assembly Processes
10. Process Control
11. Preparing Material and Processing Specifications

- 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.

## Support of CMH-17 Handbook

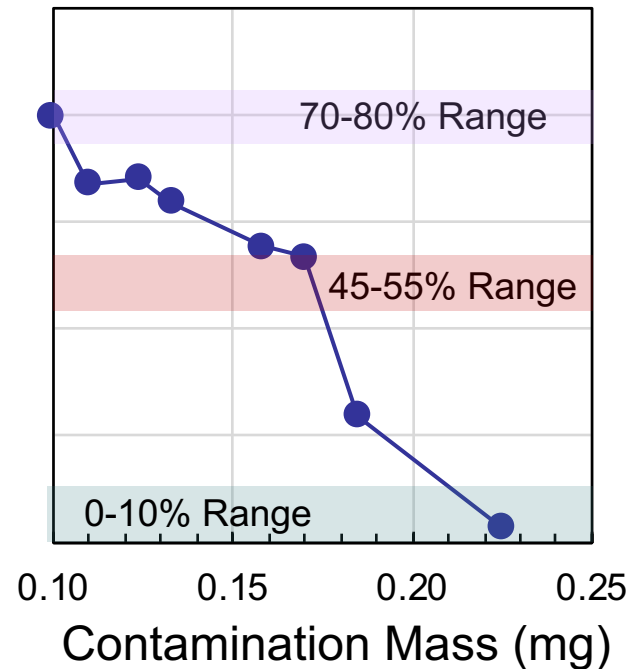
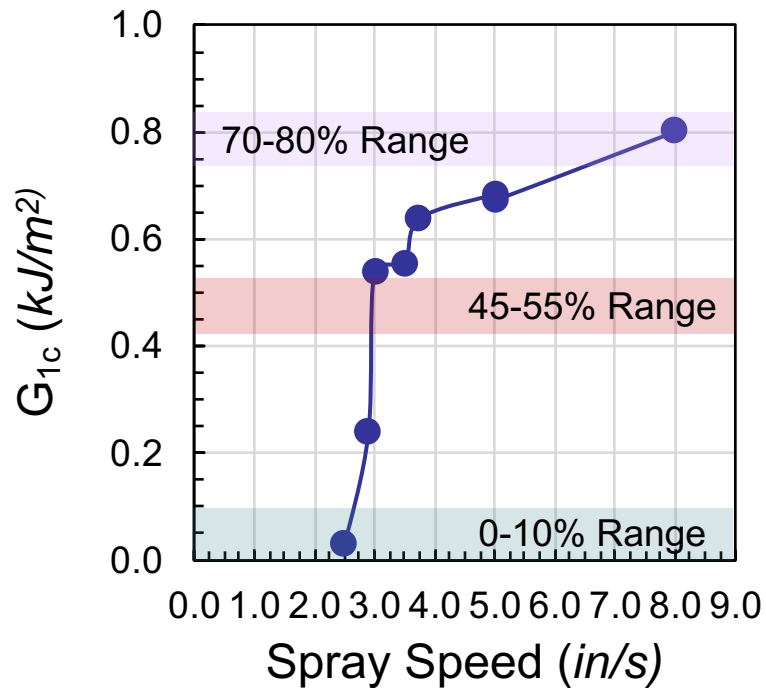
Content on bond testing and quality as well as materials will be assembled, organized and submitted for review for CMH-17.

# **Effect of Surface Contamination on Composite Bond Integrity and Durability**

**Questions?**



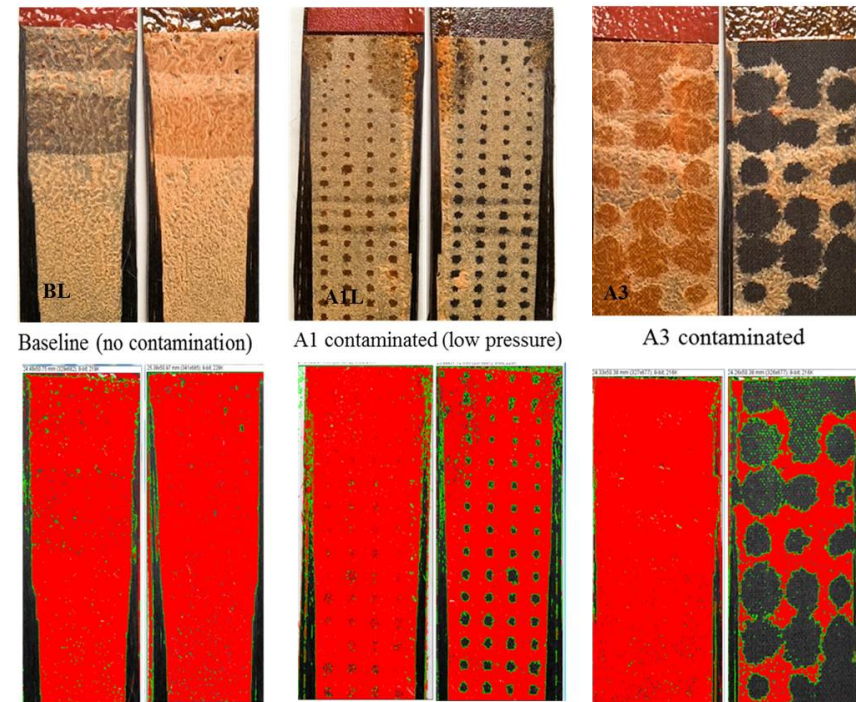
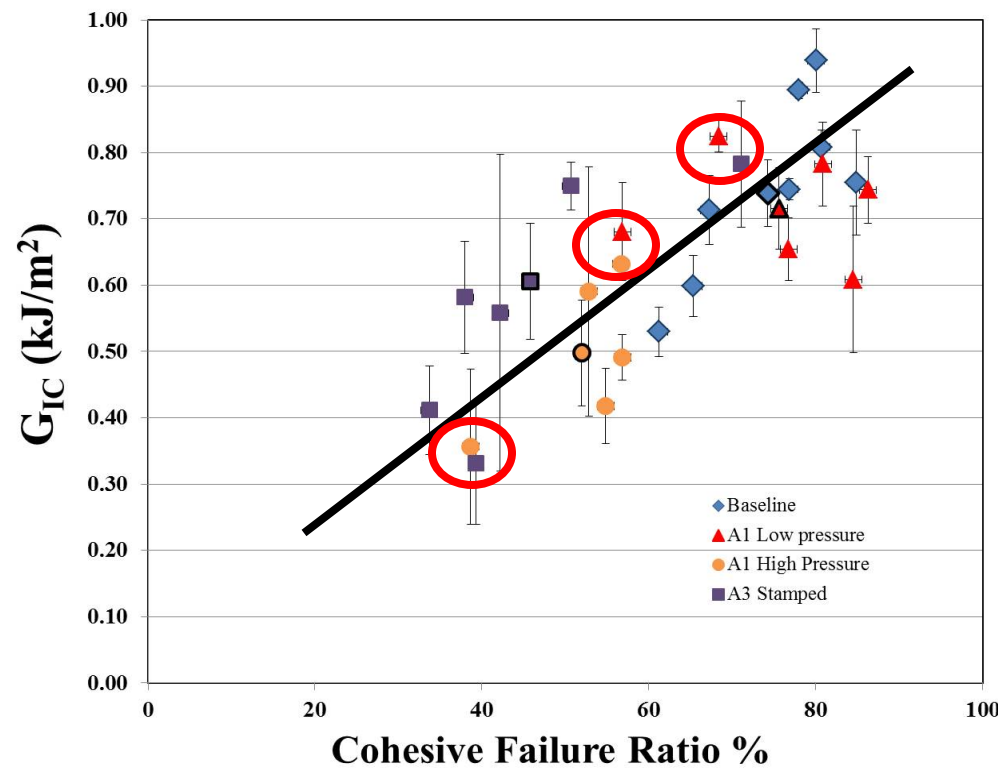
## Contamination Results



# Previous Contamination Efforts

## Discrete Methods – DCB Testing

Create scaled bond strength – vary contamination size and area



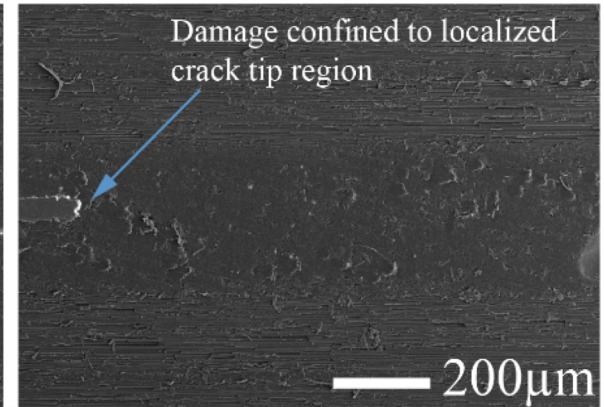
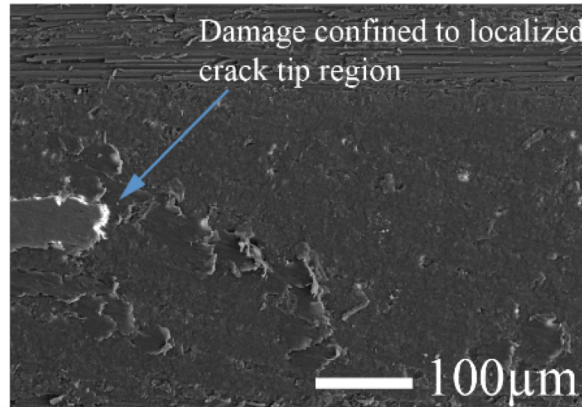
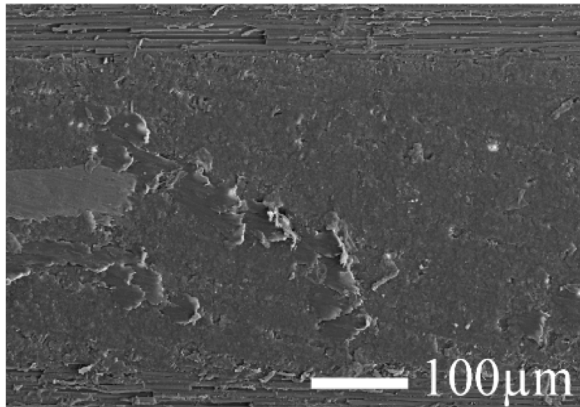
18% Drop in  $G_{IC}$  per 10% decrease in cohesive ratio  
 Contamination was modeled as circular cracks

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

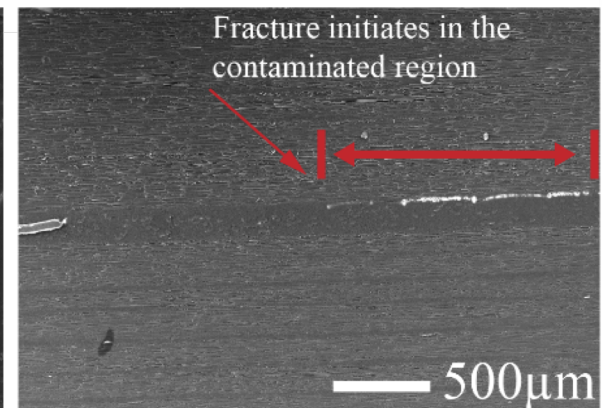
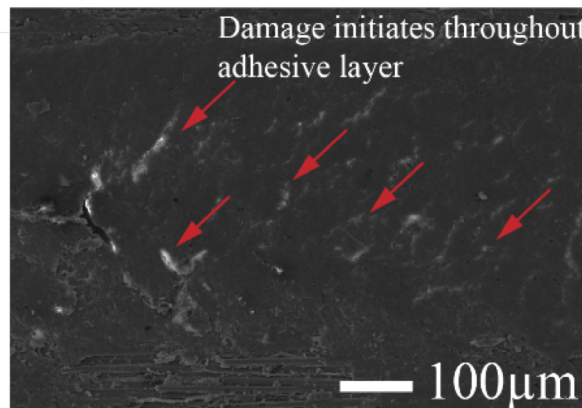
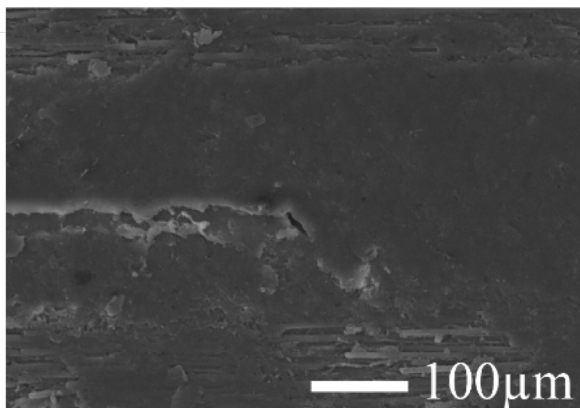
# Previous Contamination Efforts

## Discrete Methods – $\mu$ ENF Testing

Baseline



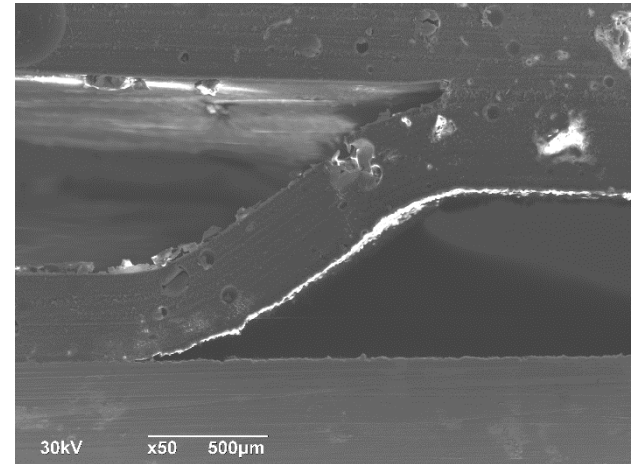
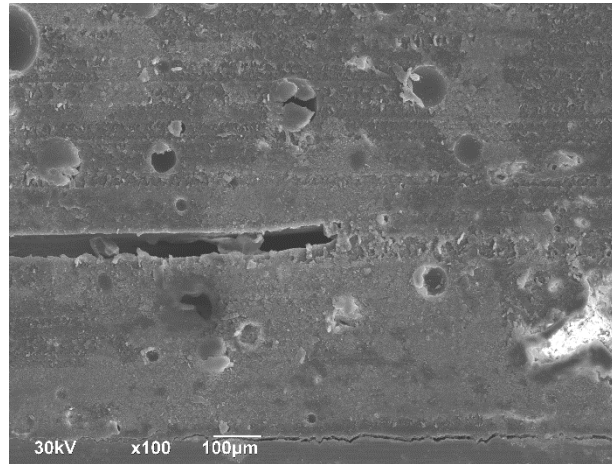
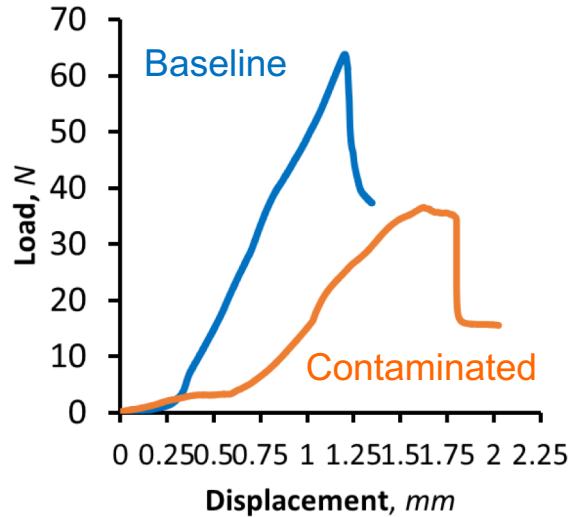
A3 Contaminated



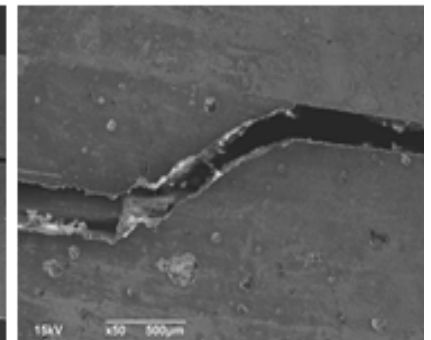
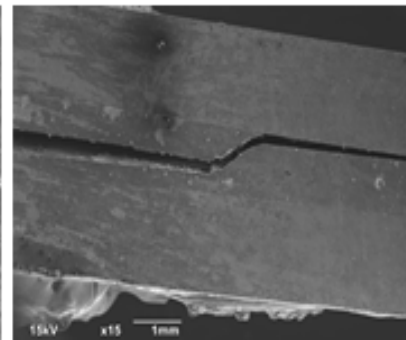
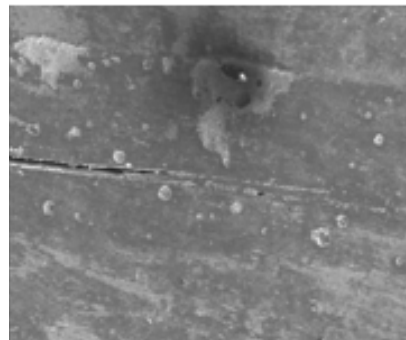
Prior to loading

At peak load (1000N)

# 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.



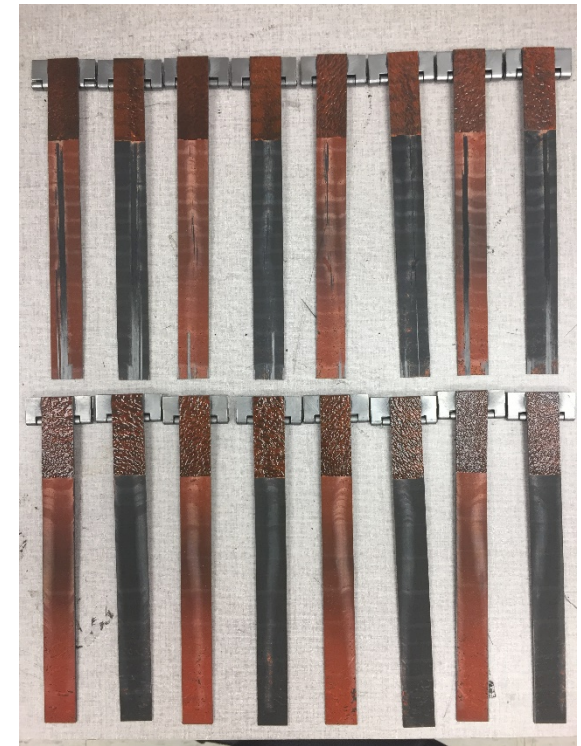
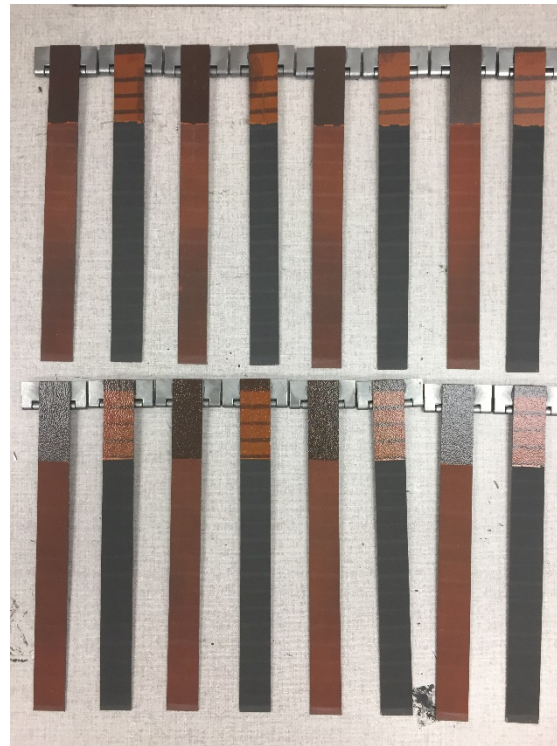
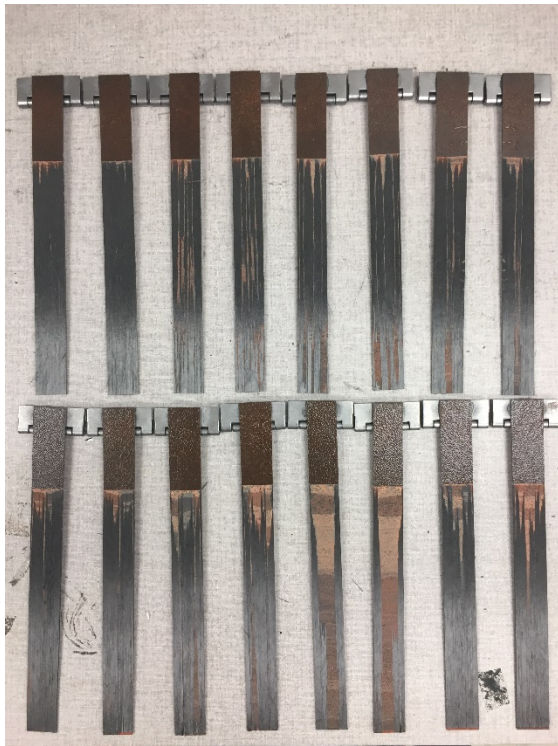
# Failure Modes – Uniformly Sprayed Approach (13%)

**Baseline**

**Contaminated**

**Wipe Sand Wipe**

**Unexposed**



**Exposed**

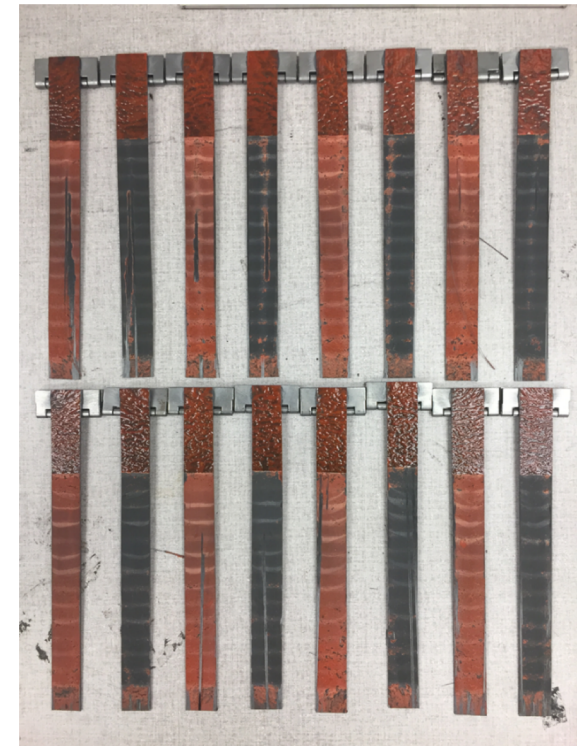
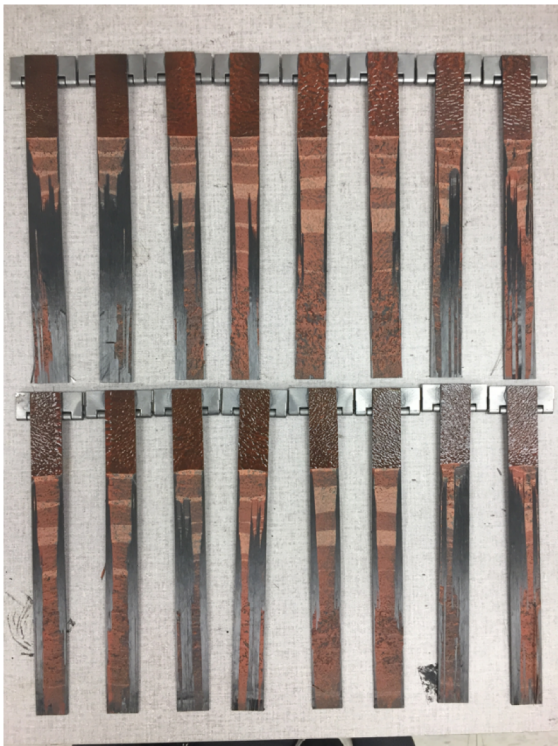
## Failure Modes – Uniformly Sprayed Approach (40%)

**Baseline**

**Contaminated**

**Wipe Sand Wipe**

**Unexposed**



**Exposed**