Effect of Surface Contamination on Composite Bond Integrity and Durability

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- **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 how durability is affected by contamination.
  - Current test methods don’t allow for real time imaging of crack propagation.

- **Objective**
  - Investigate undesirable bonding conditions by creating scalable and repeatable weak bonds.
  - Investigate a means to mitigate the undesirable conditions via surface preparation methods.
  - Investigate the effect of harsh environmental conditions on adhesive bonds.
  - Quantify fracture toughness from imaging and obtain additional information on crack tip through stress intensity factor

Effect of Surface Contamination on Composite Bond Integrity and Durability

- **Principal Investigators**
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- **Students**
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- **FAA Technical Monitor**
  - Ahmet Oztekin
- **Industry Participation**
  - Exponent, 3M, Embraer, BTG Labs

Outline

- Materials
- Overview of Contamination Approaches
- Bond Quality Evaluation
- Discrete Contamination Approach
- Continuous Contamination Approach with Mitigation Methods
- Microscale DCB Testing
- Potential Future Efforts

Materials

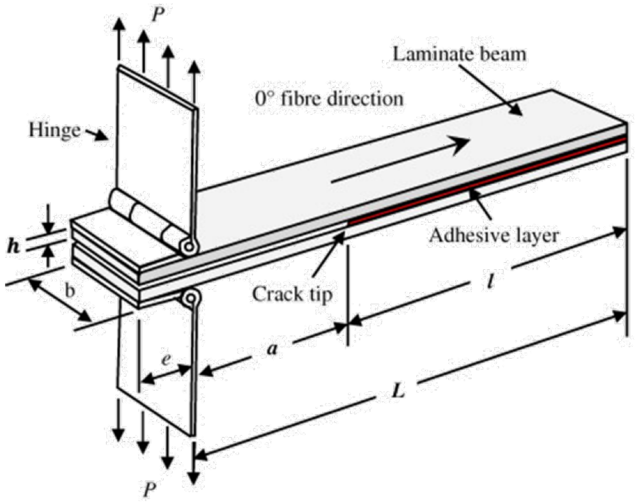
- **Material type and curing procedure for specimens:**

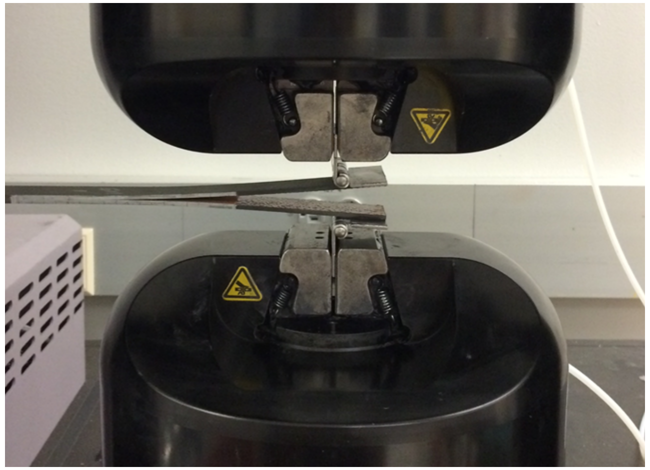
  Unidirectional carbon-epoxy system, film adhesive, secondary curing bonding and contaminants.

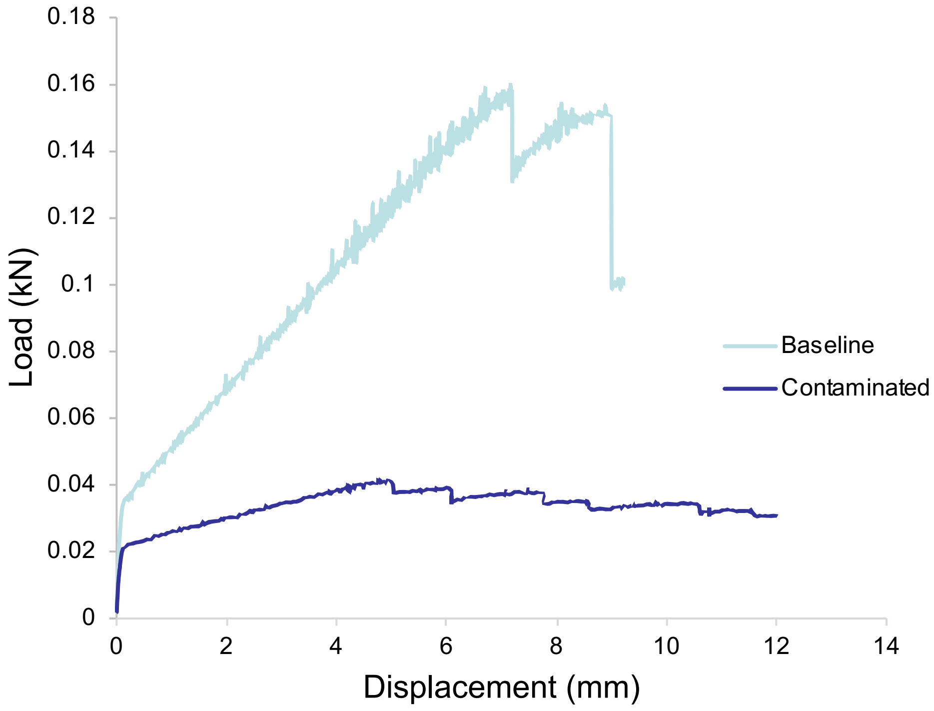
- **Materials utilized:**
  - Toray P 2362W-19U-304 T800 Unidirectional Prepreg System (350F cure)
  - 3M AF 555 Structural adhesive film (7.5x2 mills, 350F cure)
  - Precision Fabric polyester peel ply 60001
  - Frekote 700-NC from Henkel Corporation

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







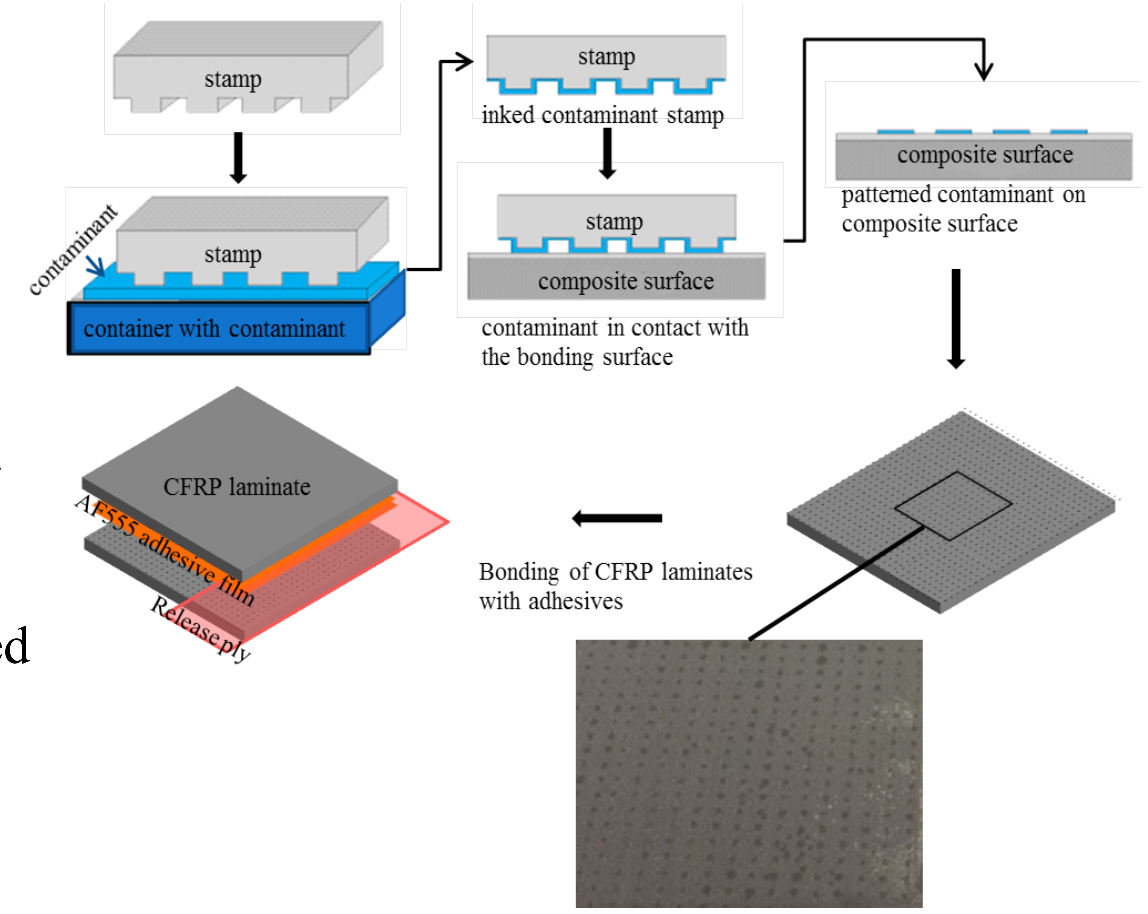
Discrete Contamination Approach

GOAL - Develop a process to create a scalable and repeatable weak bond via bondline contamination.

Contaminant – Frekote release agent.

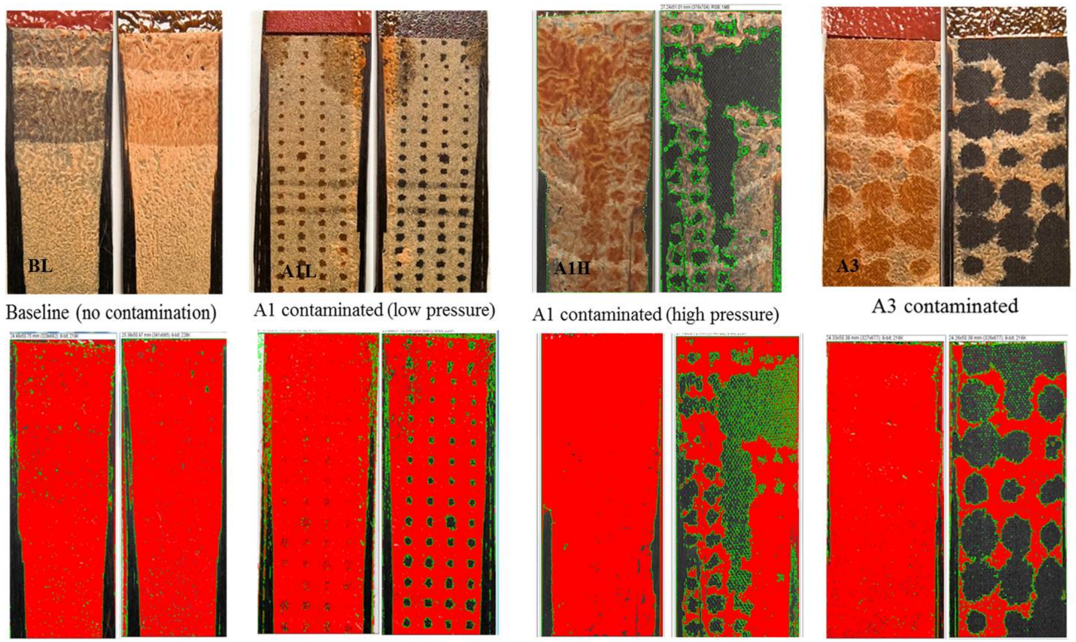
Discrete Method:

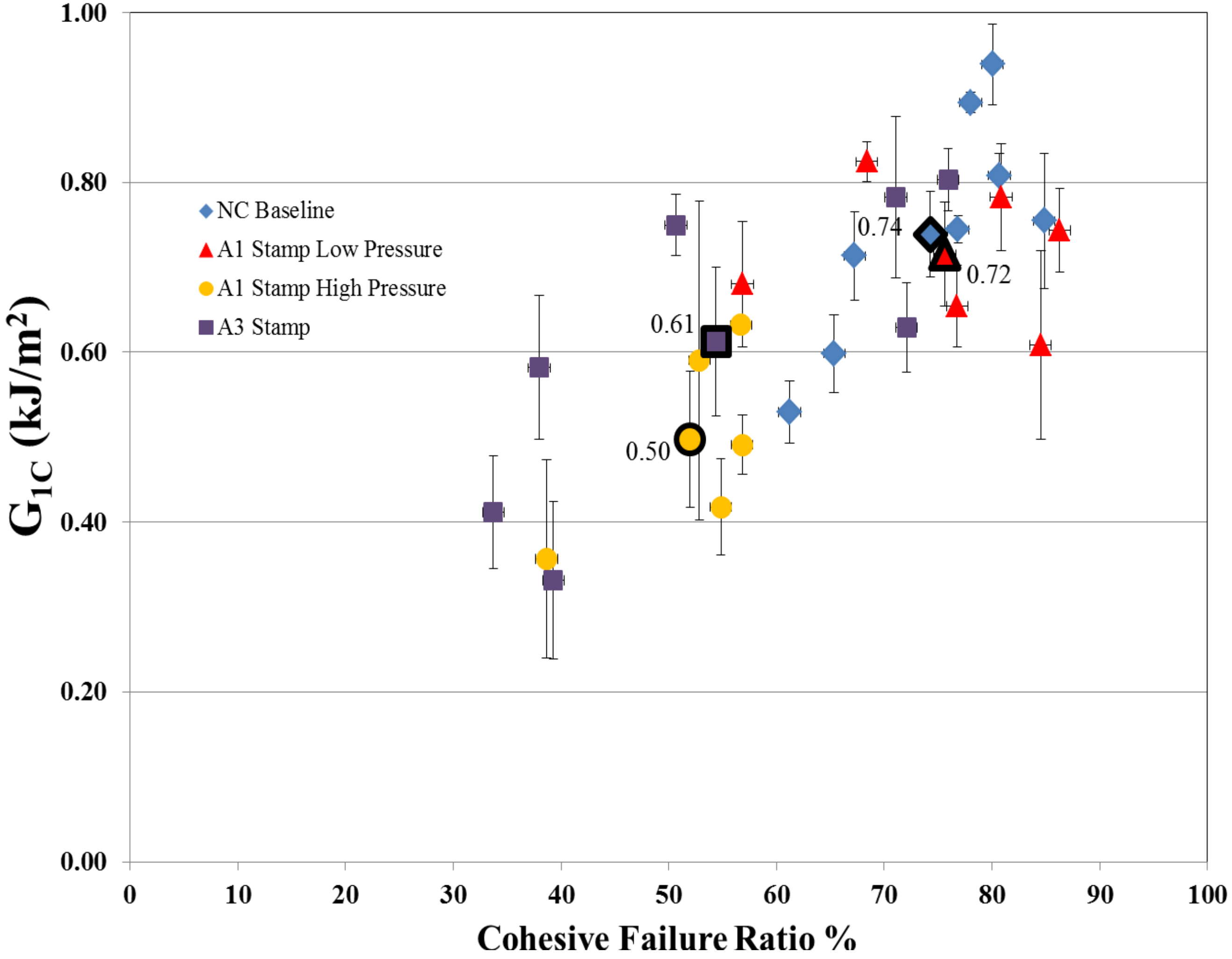
- Stamp with spatially ordered dotted pattern of contamination.
- Patterns with 1 mm (A1) and 3 mm (A3) were studied.
- Equal applied contamination area, different localized contamination.
- Low pressure (LP) and high pressure (HP) were applied and compared on A1.



Discrete Contamination Results

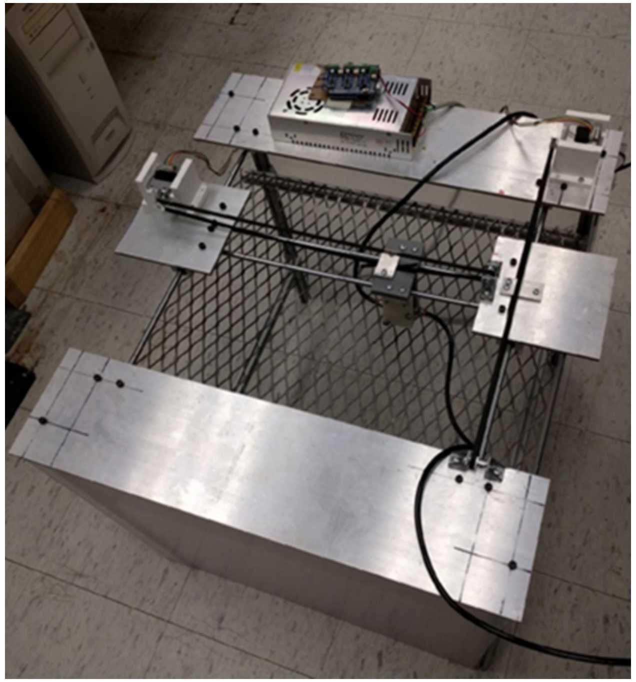
- Contamination – Ordered Array
- Create scaled bond strength – vary contamination size

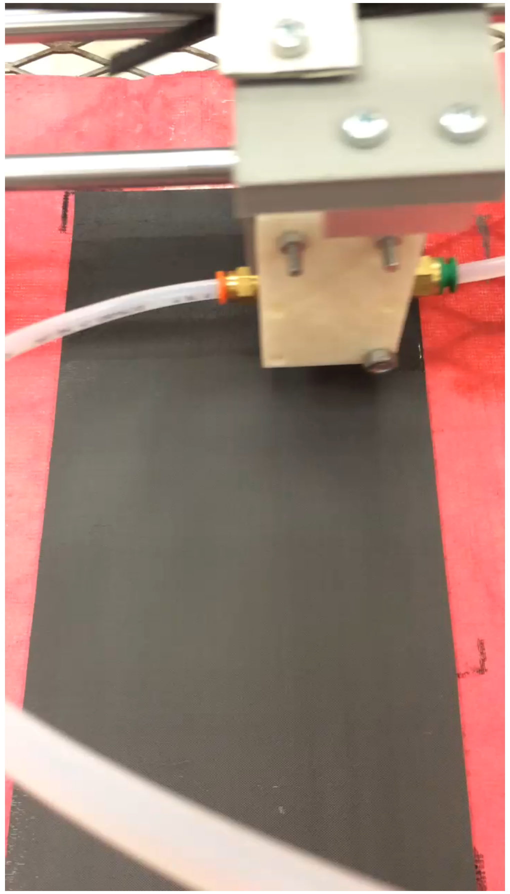




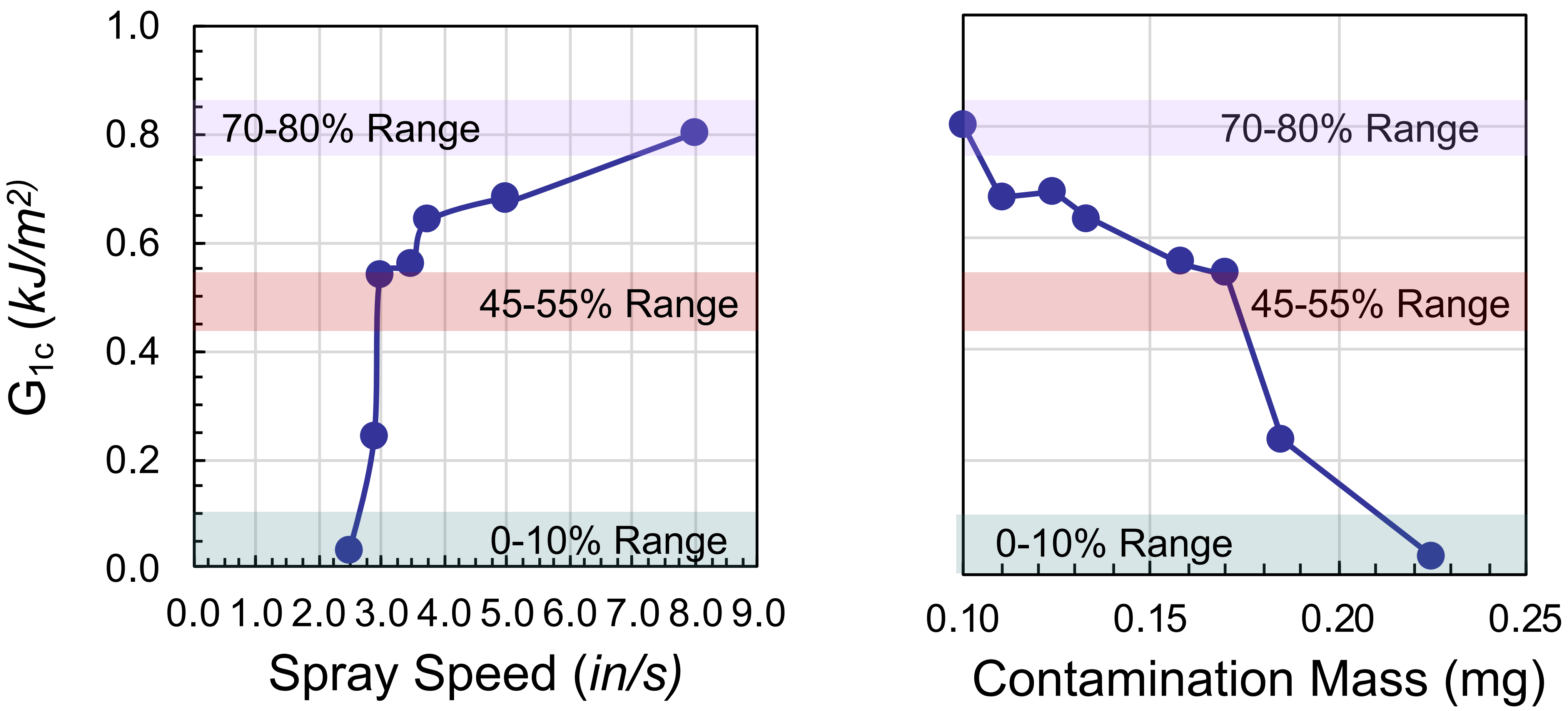
Continuous Contamination Approach

- **Continuous Method**:
  - 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 by a of pre- and post- weight measurement analysis.
    - Adjusting spray speeds and mass measurements of the contaminant on a 1" x 1" aluminum foil, allows for the correlation of the strength of the weak bond





Continuous Contamination Results

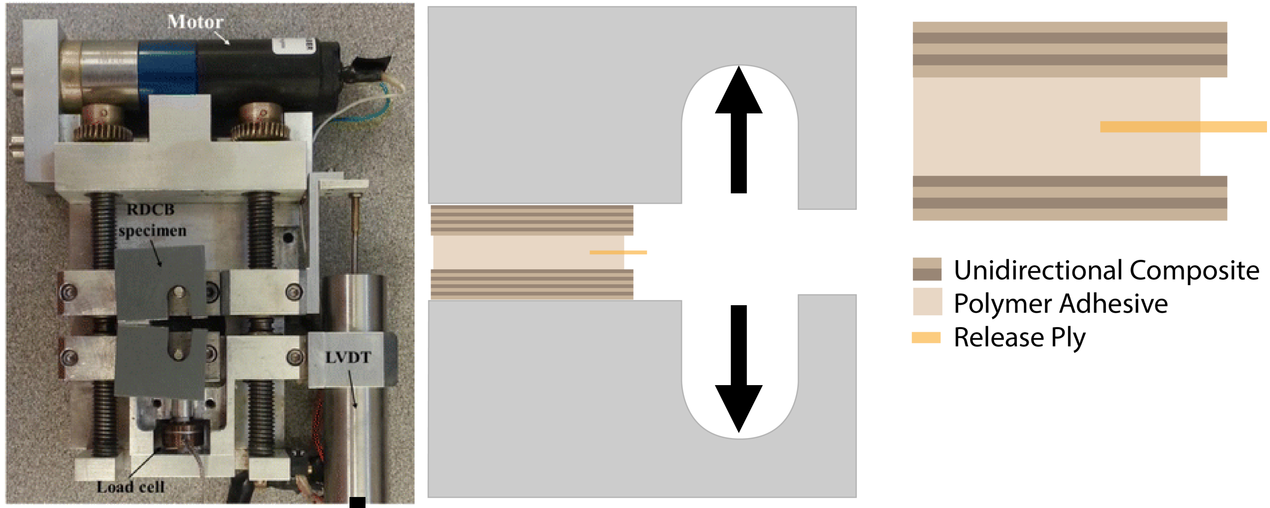


Fracture Mechanism Analysis using In-situ Electron Microscopy

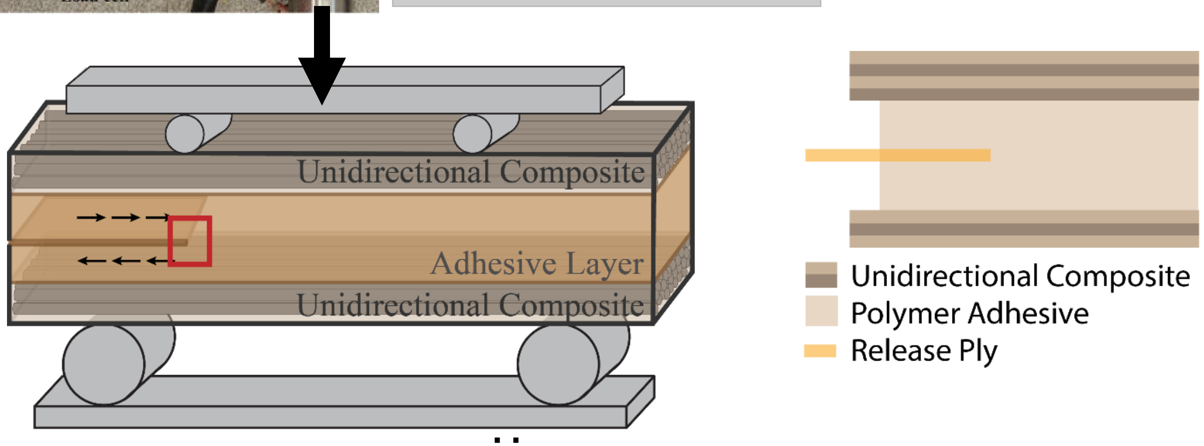
GOAL – To obtain real time imaging of crack propagation to quantify fracture toughness

Test Development

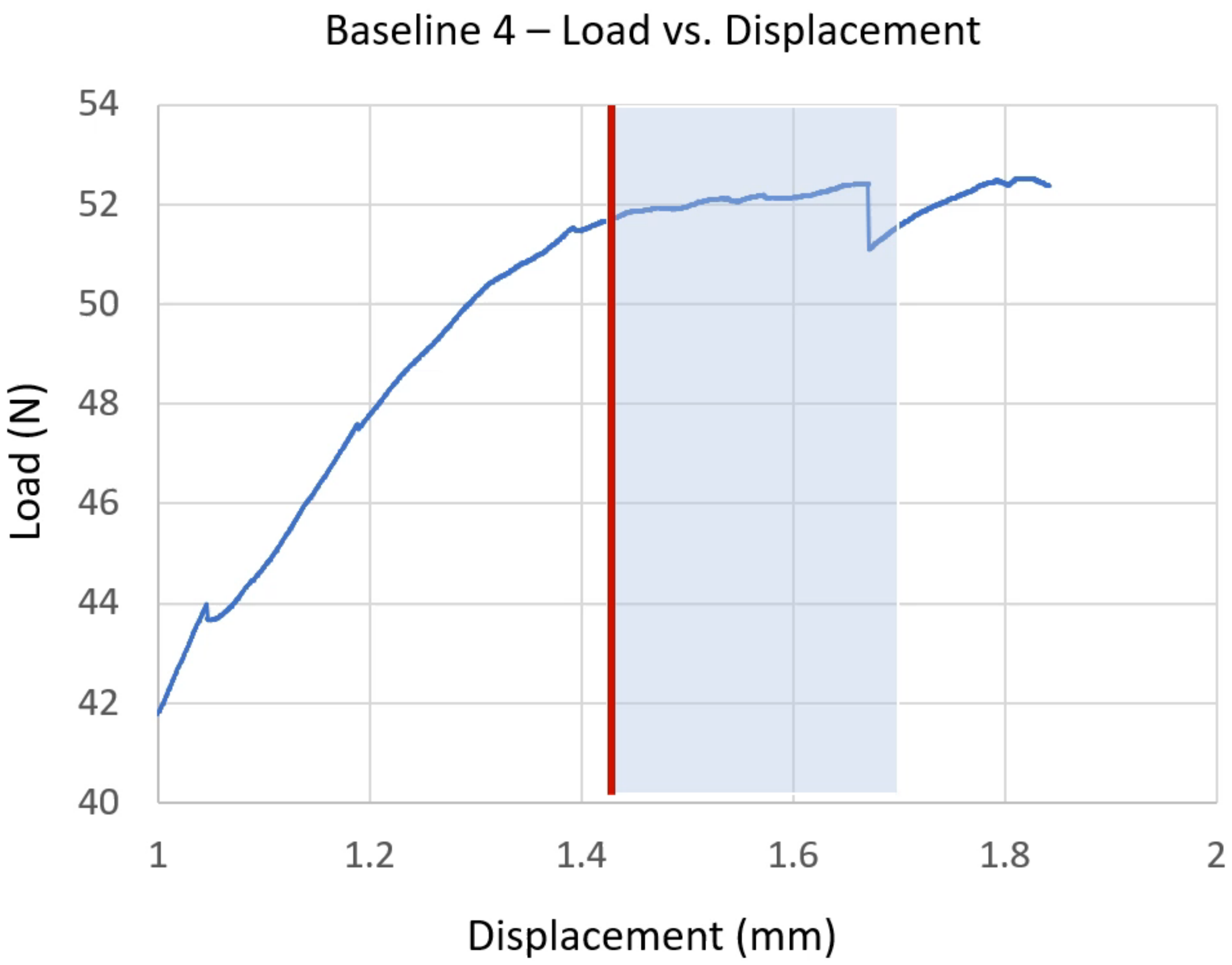
μDCB (Dual Cantilever Beam)
Assess the mechanisms of mode I fracture. Fixture was designed based on literature of metal-adhesive bond testing.

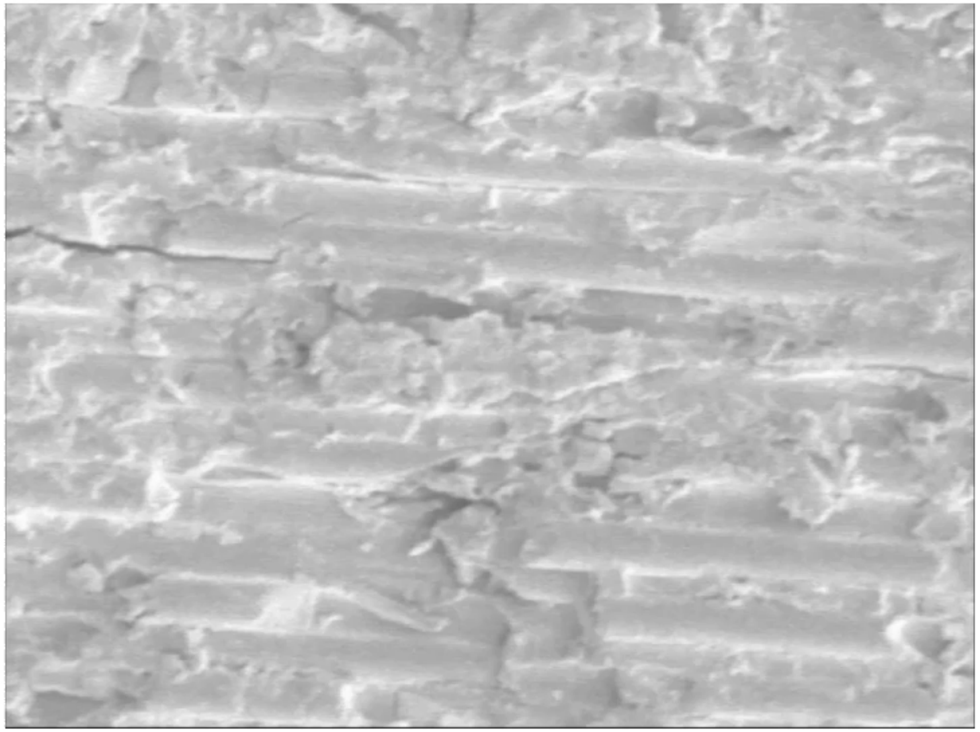


μENF (End Notch Flexure)
Assesses the mechanisms of mode II fracture. Fixture was designed based of traditional ENF testing of composite bonds



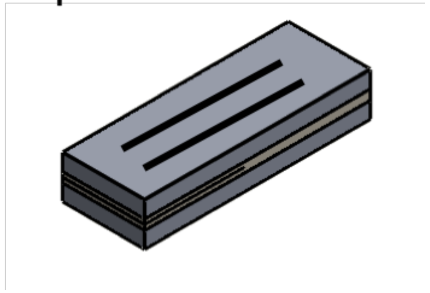
Fracture Mechanism Analysis using In-situ Electron Microscopy – Micro DCB

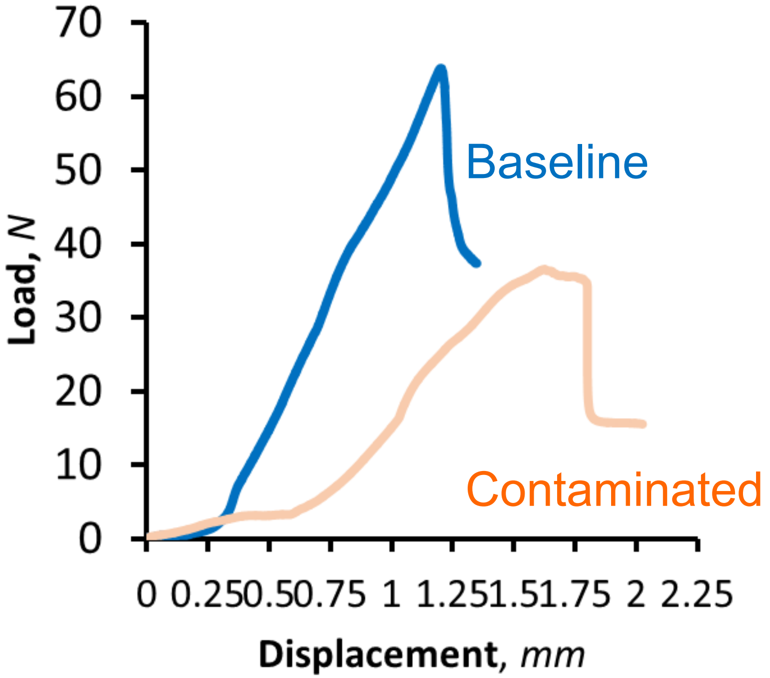




Fracture Mechanism Analysis using In-situ Electron Microscopy – End Notch Flexure

Specimen Details





Baseline

L/W: 40mm x 10mm

thickness: 5.2 mm

Pre-crack: 8 mm

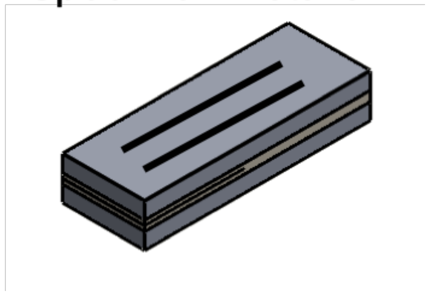
10 layer unidirectional composite panels

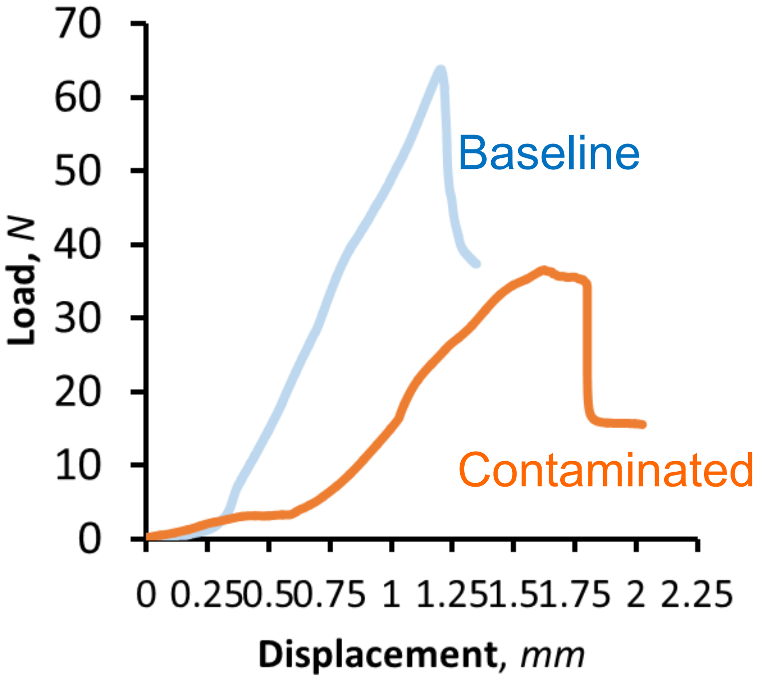
Observations

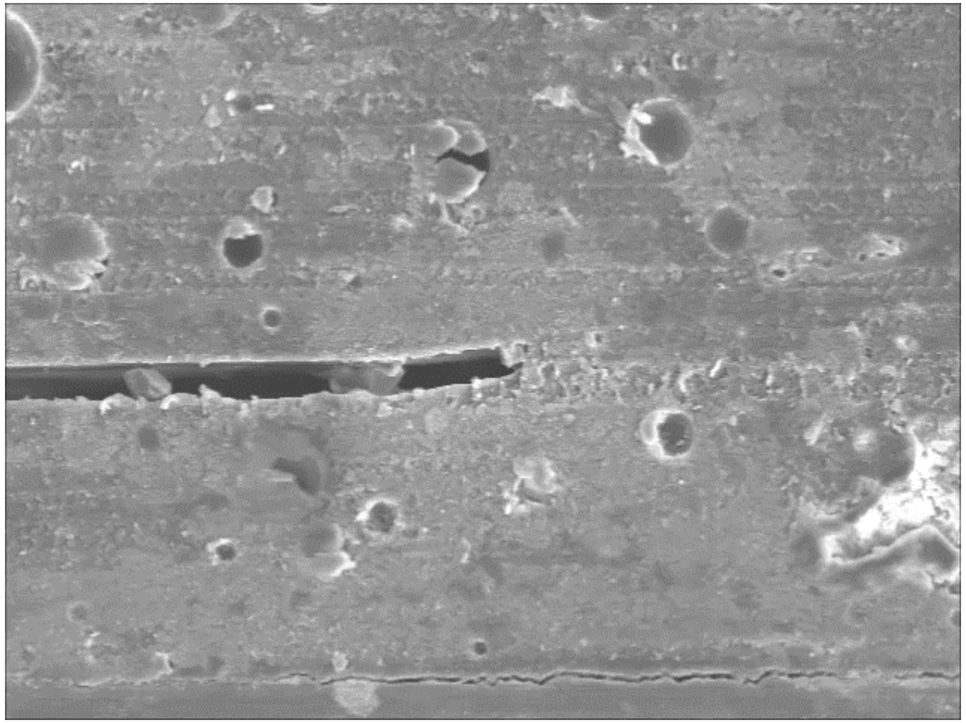
- Initially bond is very stiff
- Controlled crack propagation begins at ~50N Load
- Unstable crack growth begins at the pre-crack then travels to composite-adhesive interface

Fracture Mechanism Analysis using In-situ Electron Microscopy – End Notch Flexure

Specimen Details







Contaminated

L/W: 40mm x 10mm

thickness: 5.2 mm

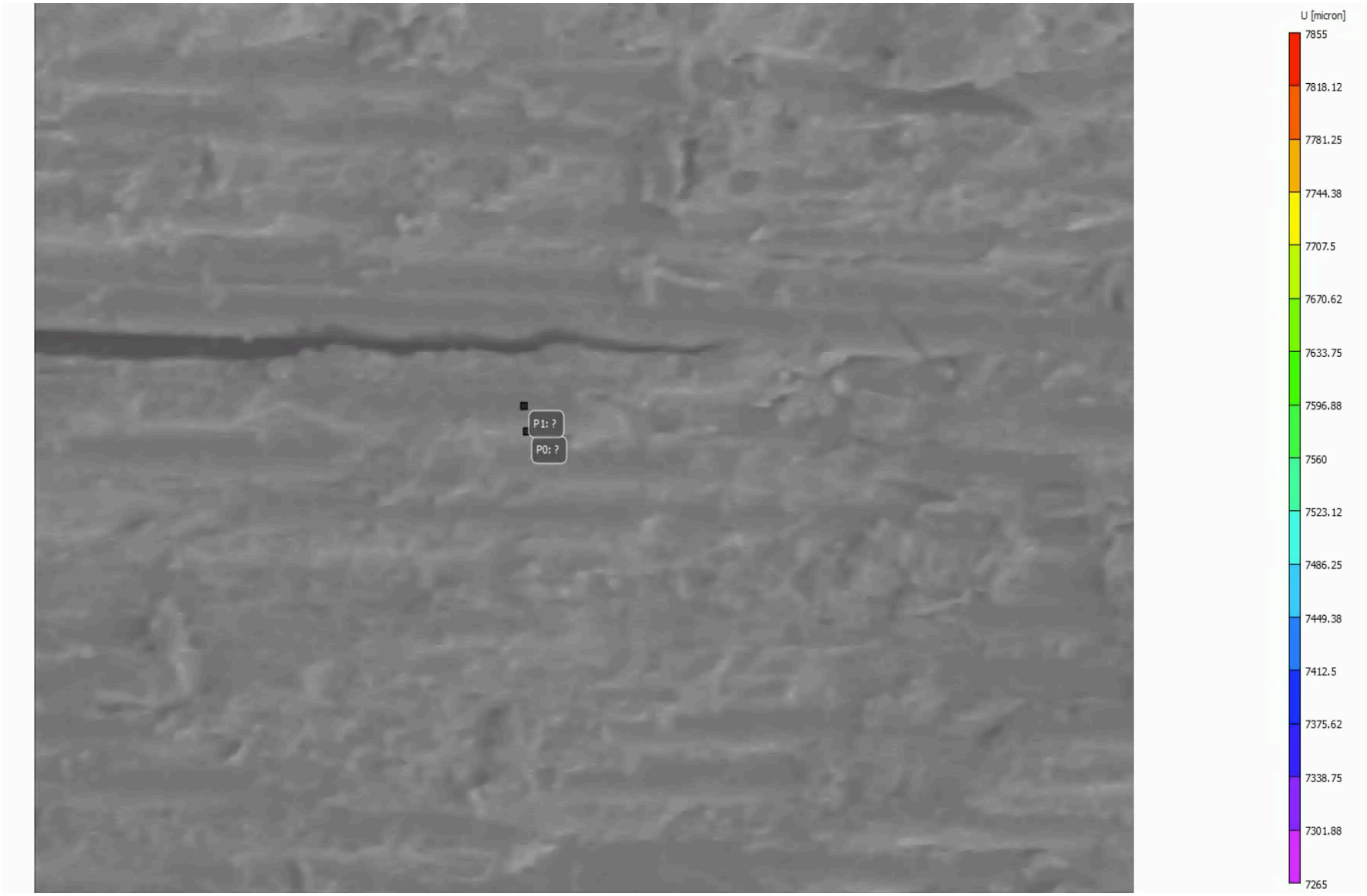
Pre-crack: 8 mm

10 layer unidirectional composite panels

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

Fracture Mechanism Analysis using In-situ Electron Microscopy – Micro DCB

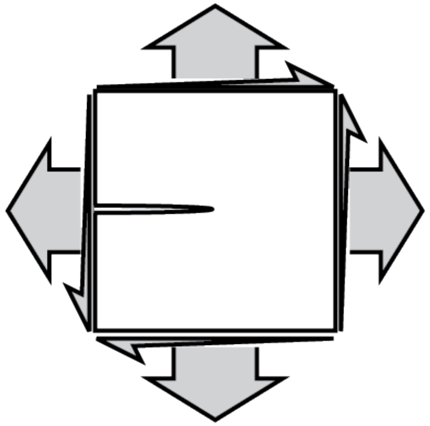


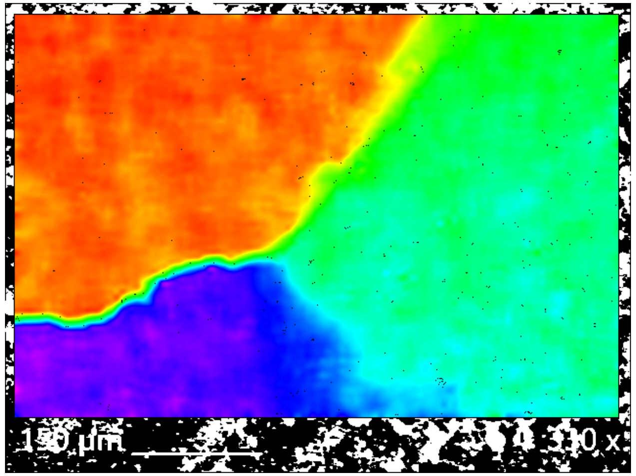
Strain Mapping

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>

Complications with in situ testing
Small sample sizes and edge effects
Sample testing environment





From LEFM

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

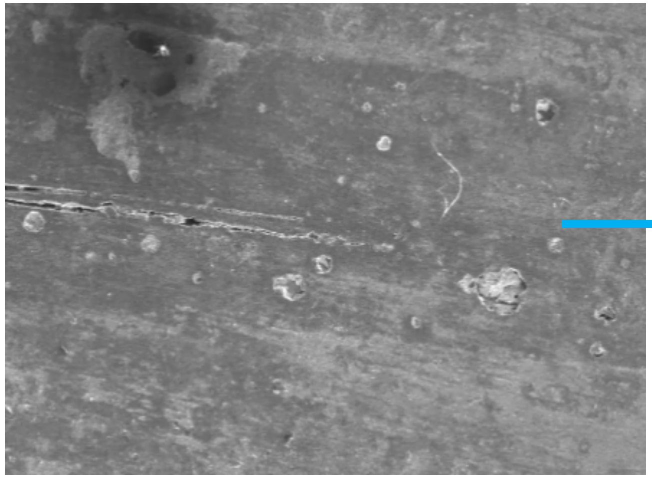
$$\sigma_{yy} = \frac{K_I}{\sqrt{2\pi r}} \cos\frac{1}{2}\theta\left(1 + \sin\frac{1}{2}\theta \sin\frac{3}{2}\theta\right)$$

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

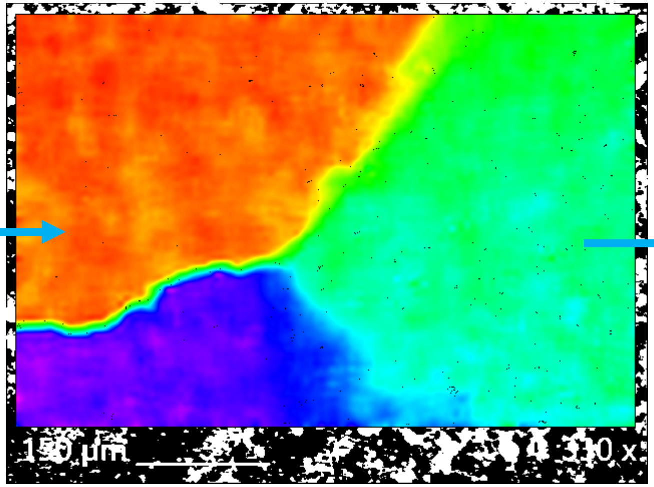
$$u_x = \frac{K_I}{8\mu\pi}\sqrt{2\pi r}\left[(2\kappa - 1)\cos\frac{\theta}{2} - \cos\frac{3\theta}{2}\right]$$

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

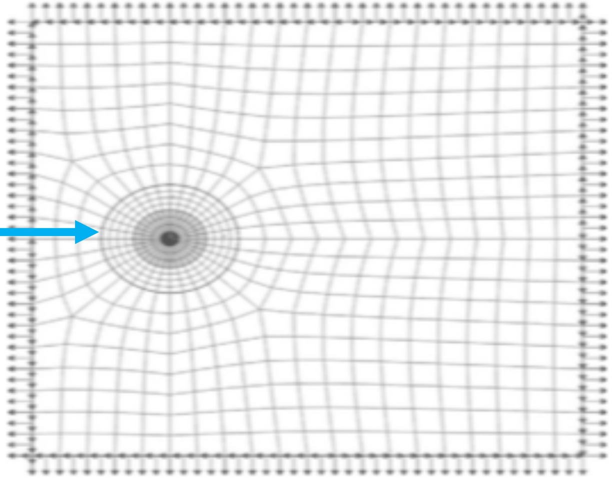
Strain Mapping



In situ Microscopy



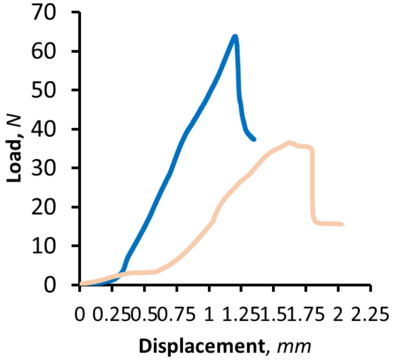
Digital Image Correlation



Digitized Displacements

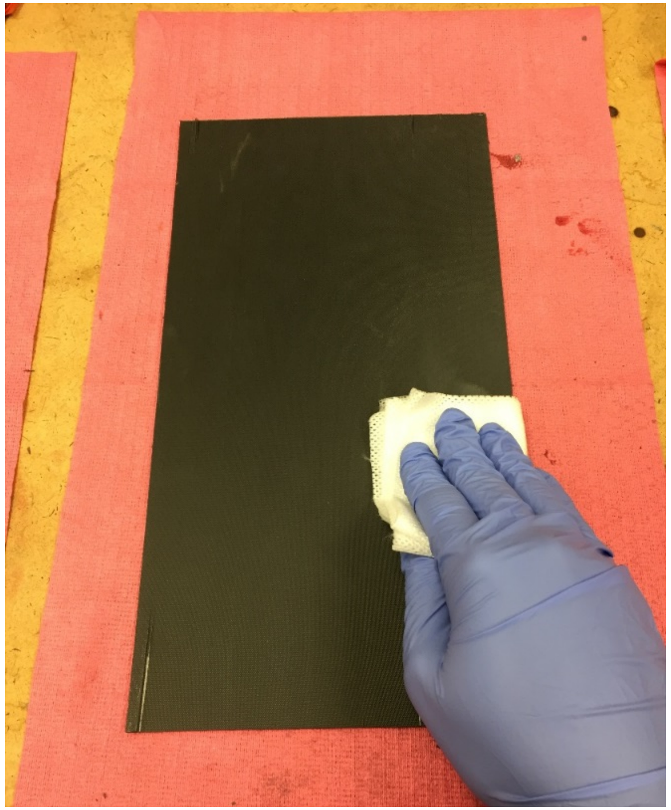
$$u_x = \frac{K_I}{8\mu\pi}\sqrt{2\pi r}\left[(2\kappa - 1)\cos\frac{\theta}{2} - \cos\frac{3\theta}{2}\right]$$

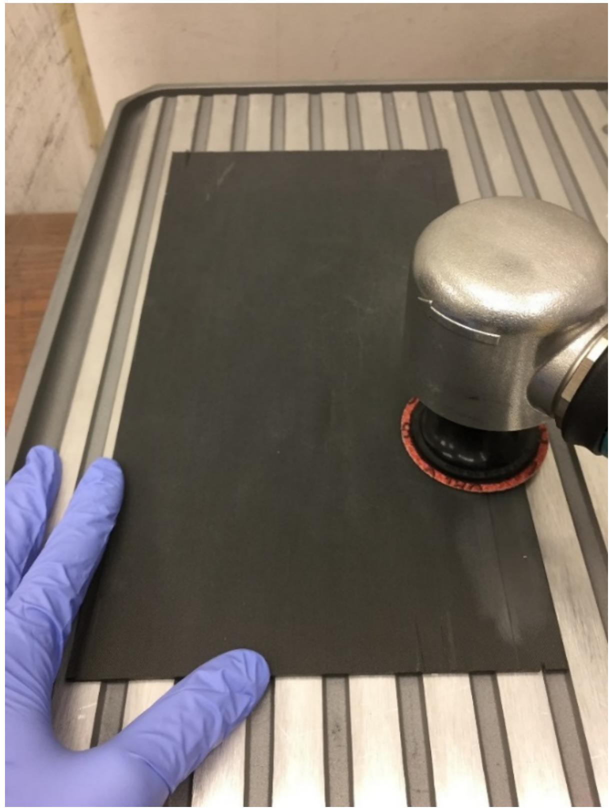
$$u_y = \frac{K_I}{8\mu\pi}\sqrt{2\pi r}\left[(2\kappa + 1)\sin\frac{\theta}{2} - \sin\frac{3\theta}{2}\right]$$



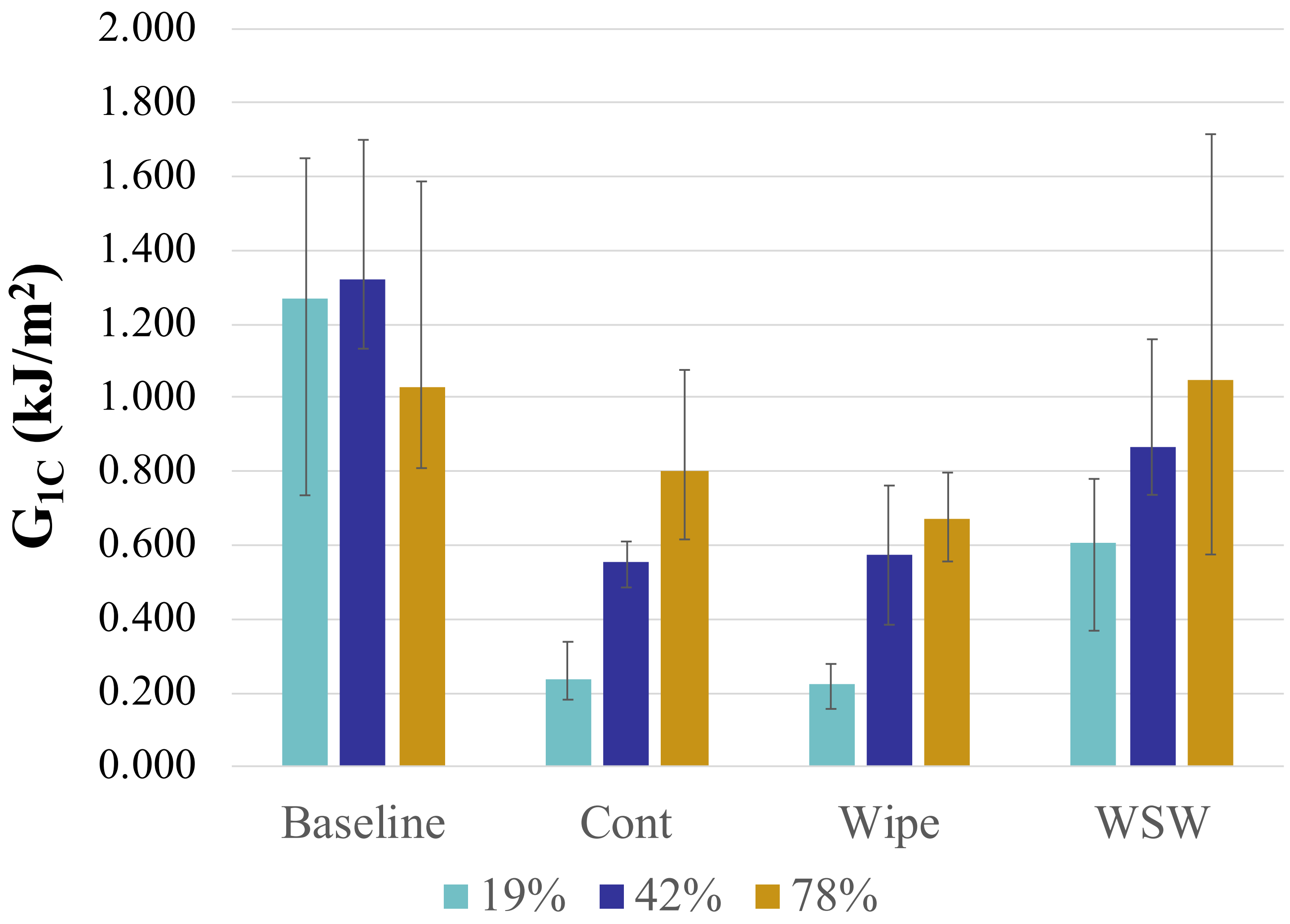
Mitigation Procedures in Continuous Contamination

- **GOAL** – Evaluate processes to mitigate the influence of contamination of the bondline
- Two methods of mitigation:
  - ***Solvent Wipe -*** Attempt to remove contaminate off the surface with soaked cloth
  - ***Sanding of Material -*** Actively remove material using abrasive





Mitigation Results in Continuous Contamination



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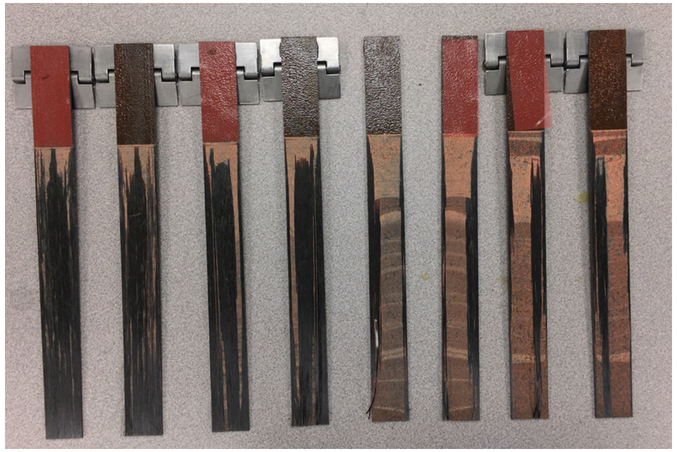
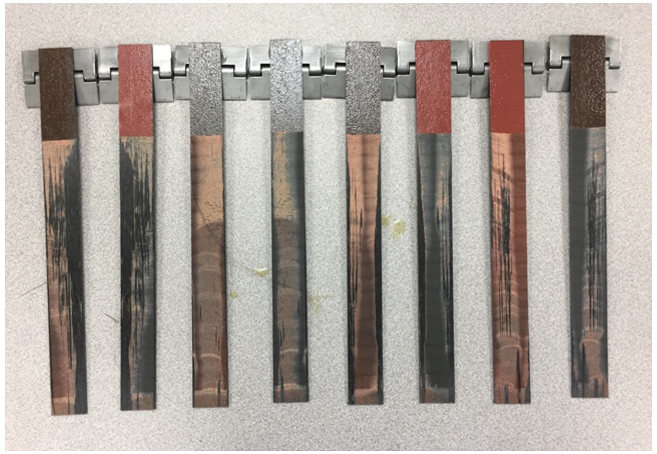
Failure Modes – 19%

Mixed-mode failure
Variable combination of interlaminer and cohesion

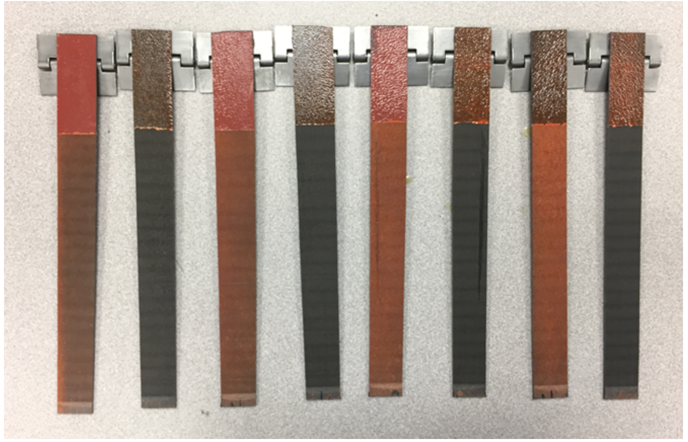


Baseline

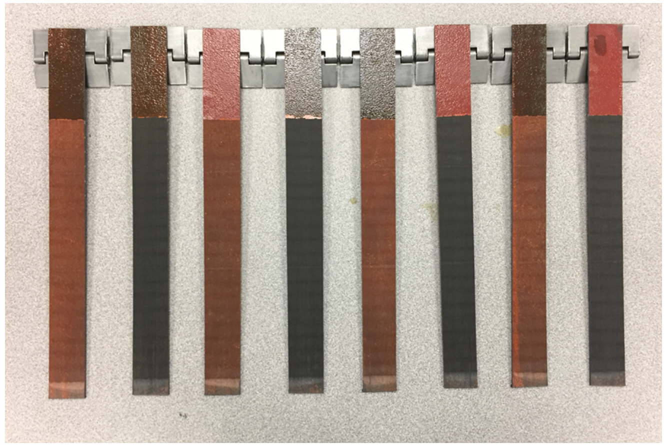


WSW

Adhesion failure
Separates from the surface of adherent



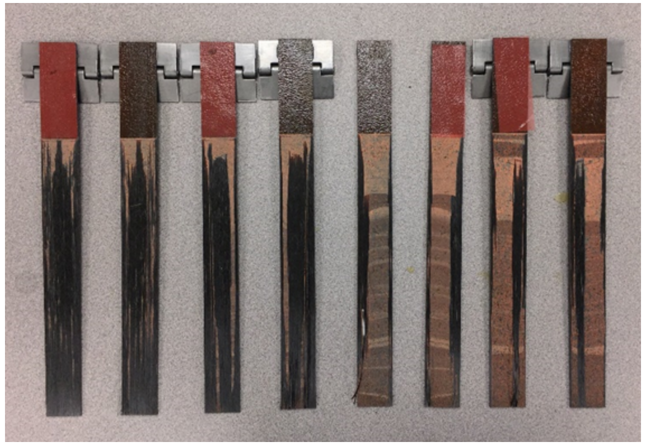
Contaminated



Wipe

Failure Modes – 42%

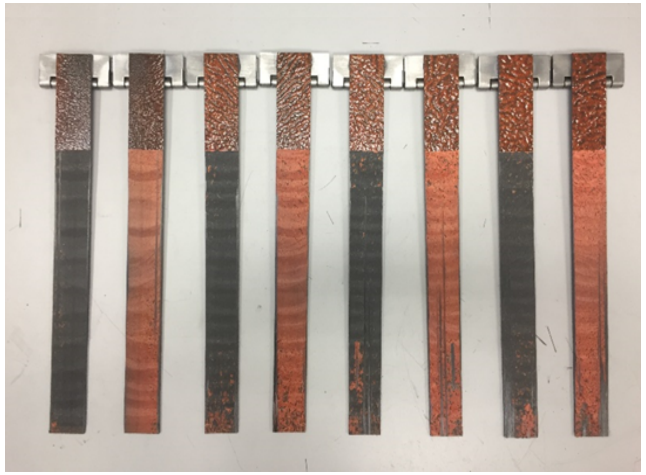
Mixed-mode failure
Variable combination of interlaminer and cohesion



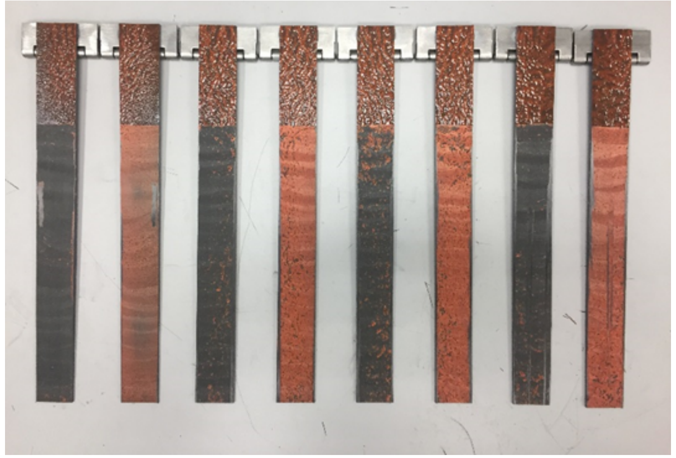
Baseline

WSW

Adhesion failure
Separates from the surface of adherent



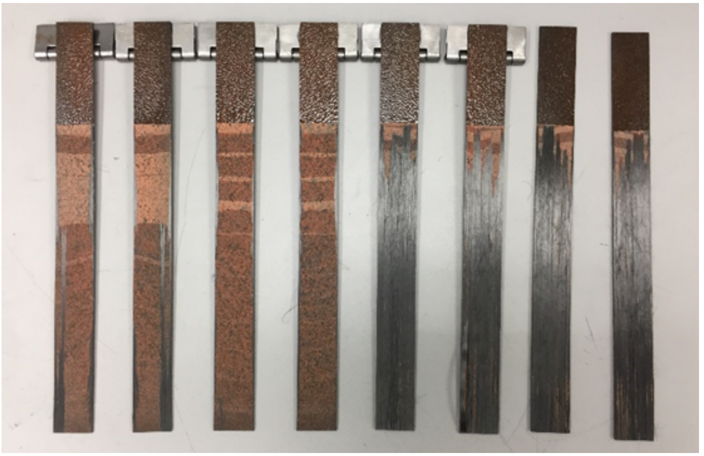
Contaminated



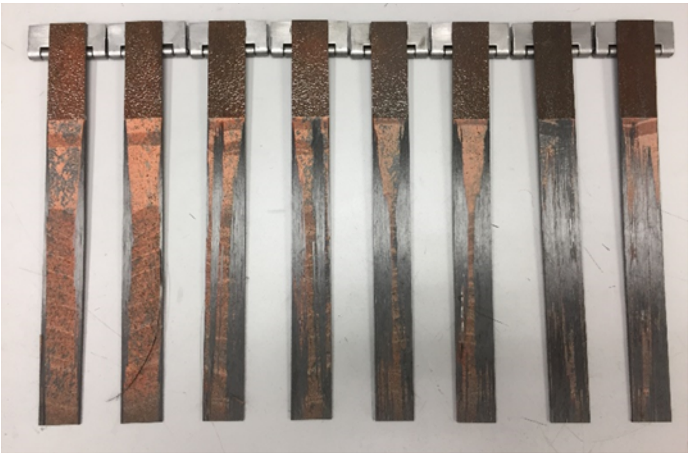
Wipe

Failure Modes – 78%

Mixed-mode failure
Variable combination of interlaminer and cohesion

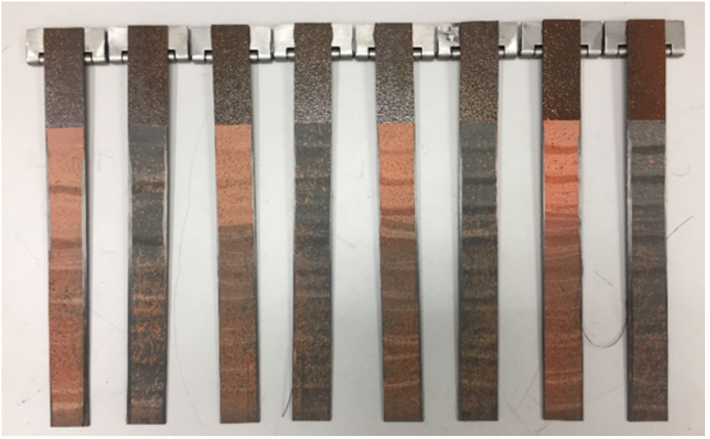


Baseline

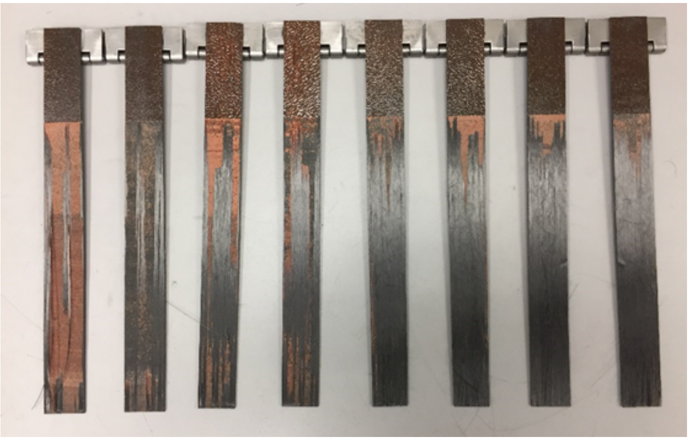


WSW

Adhesion failure
Separates from the surface of adherent



Contaminated



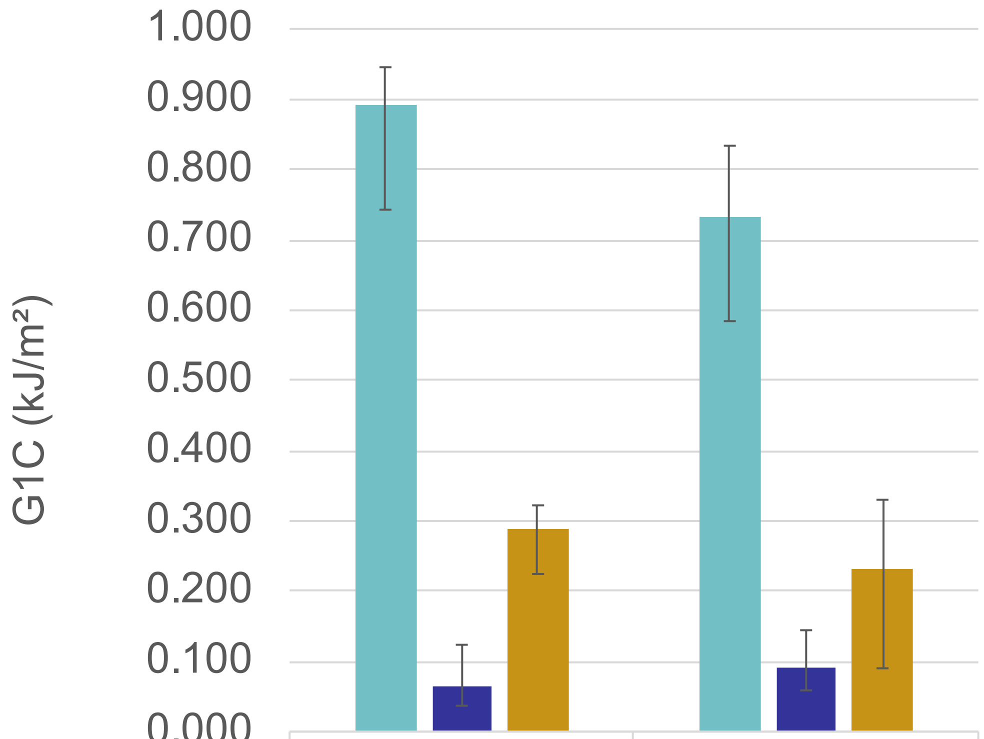
Wipe

Durability Characterization: Environmental Aging for Continuous Contamination

- 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.

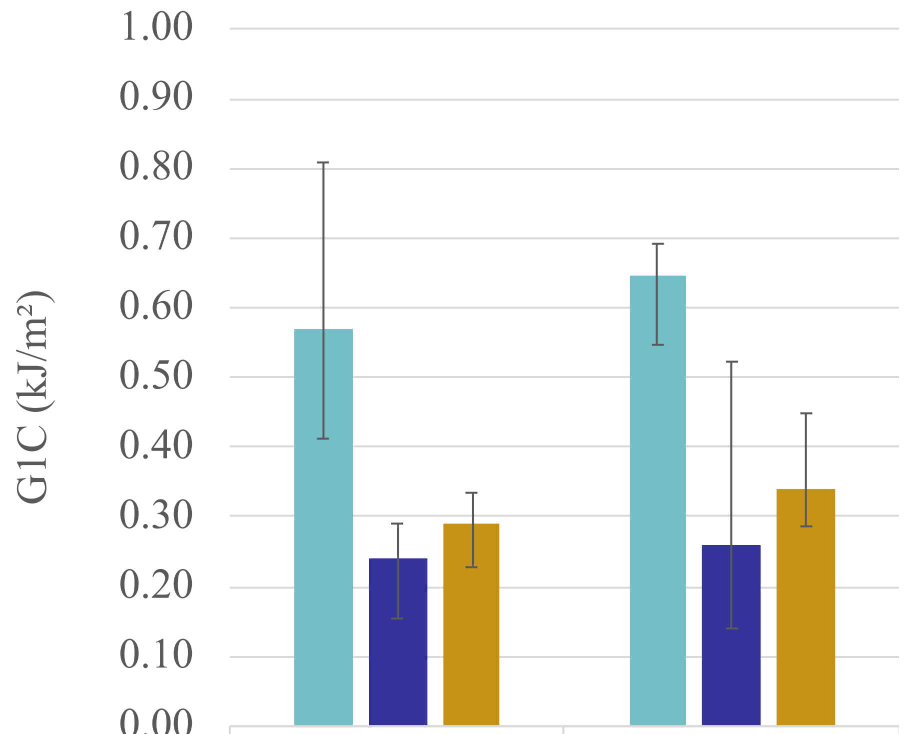
Environmental Aging Results for Continuous Contamination

7% Cumulative Baseline Summary



	Unexposed	Exposed
Baseline	0.892	0.734
Contaminated	0.065	0.091
WSW	0.288	0.232

42% Cumulative Baseline Summary



	Unexposed	Exposed
Baseline	0.57	0.65
Contaminated	0.24	0.26
WSW	0.29	0.34

Summary

- Contamination procedures were developed using Frekote to refine 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 discrete pattern composed of circles with different diameters (1 mm and 3 mm)
- A customized contamination rig was used to obtain three different levels of continuous contamination (~20, 40 and 80% bond strength)
- Means to evaluate mechanisms and initiation of failure via in-situ electron microscopy. Potential methods for quantifying fracture properties.
- 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 method.
- Environmental aging was evaluated for durability characterization.

Path Forward

<u>Proposed New Task</u>: Evaluation of Peel Tests Verses Shear Tests for Adhesively Bonded Systems

- The use of Lap Shear Tests for evaluating bond strength and the bonding process is much simpler and easier to implement than DCB tests.
- Although DCB tests are one of the most common tests for evaluating the bonding process, little research has been conducted that demonstrates its advantages over shear tests.
- Hypothesis: Lap Shear Test methods are less sensitive to non-optimal bonding conditions due to a non-linear loading case that can mask imperfections.

New Effort

- Utilize undesirable bonding conditions to validate the sensitivity of Peel Tests verses Shear Tests.
- Initially evaluate DCB and Lap Shear tests.
- Use continuous and discrete contamination approaches at various contamination levels to understand and quantify the levels of sensitivity for each type of test.