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Effect of Surface Contamination on Composite Bond Integrity and Durability

Ben Boesl - Assistant Professor
Florida International University

JAMS 2016 Technical Review
March 23 , 2016

Composite Bond Integrity/Long-Term Durability of Composite Bonds

- 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 characterizing the initial performance at various contamination levels
  - Characterize the durability performance of the system using the same contamination levels
  - Support CMH-17 with the inclusion of content for bonded systems

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Effect of Surface Contamination on Composite Bond Integrity and Durability

- **Principal Investigators**
  - Dwayne McDaniel, Ben Boesl
- **Students**
  - Vishal Musaramthota, Shervin Tashakori
- **FAA Technical Monitor**
  - Curt Davies
- **Industry Participation**
  - Exponent, 3M, Embraer

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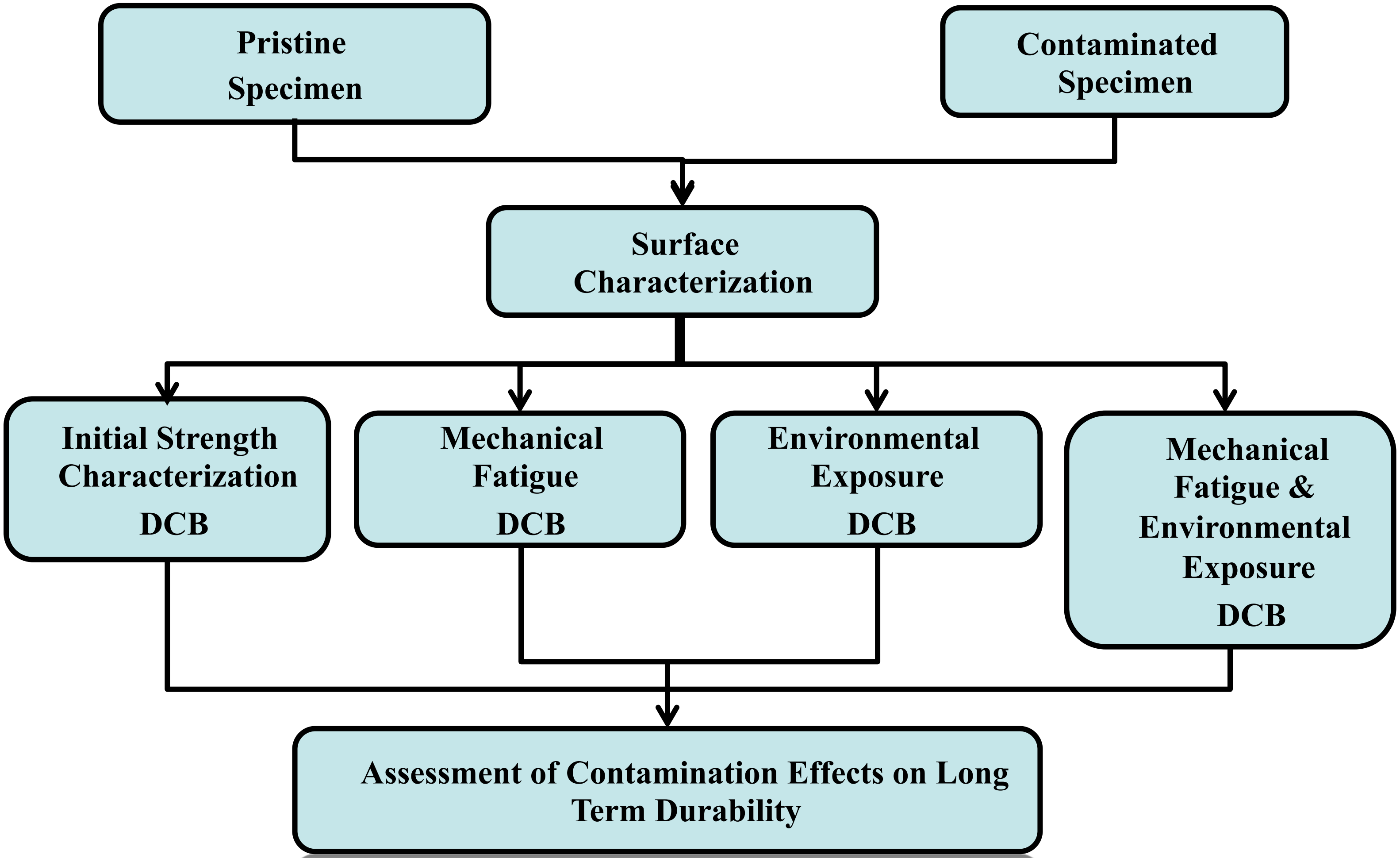
Durability Assessment Procedure



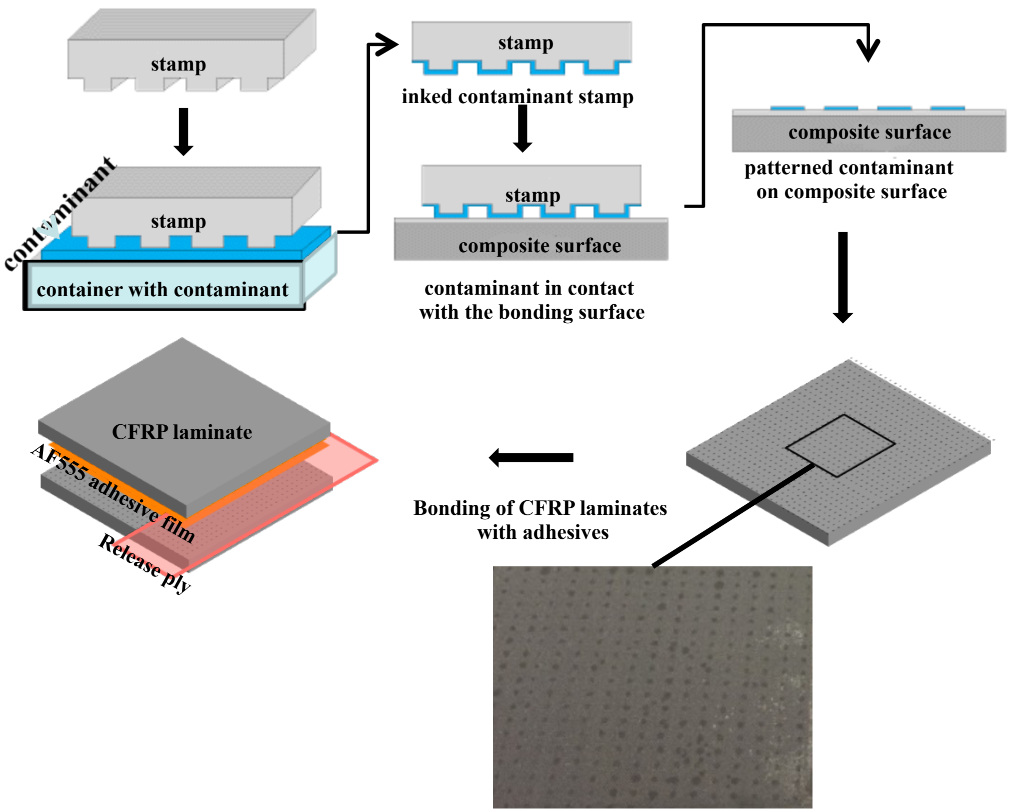
Bonding System Materials

- Material type and curing procedure for specimens: unidirectional carbon-epoxy system, film adhesive, secondary curing bonding and contaminants.

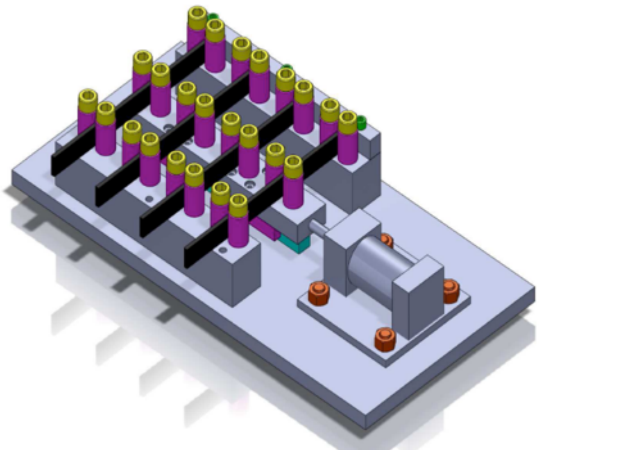
- Materials utilized:
  - Toray P 2362W-19U-304 T800 Unidirectional Prepreg System (350F cure)
  - 3M AF 555 Structural adhesive film (7.5x2 mills, 350F cure)
  - Precision Fabric polyester peel ply 60001
  - Freekote 700-NC from Henkel Corporation

- Specimen Conditioning:
  - Environmental Chamber : 50°C, 95% RH, for 8 weeks and 1.5 years
  - Fatigue Loading: 3 point bending arrangement, 1 inch double amplitude, 2.6 million cycles

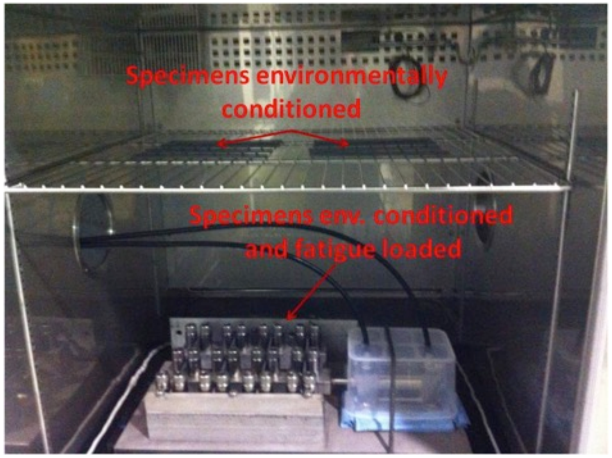
Discrete Contamination Procedure and Durability Test Setup



Variation in stamp size and application pressure



3-point bend fixture



Environmental chamber

Assessment of Bond Quality

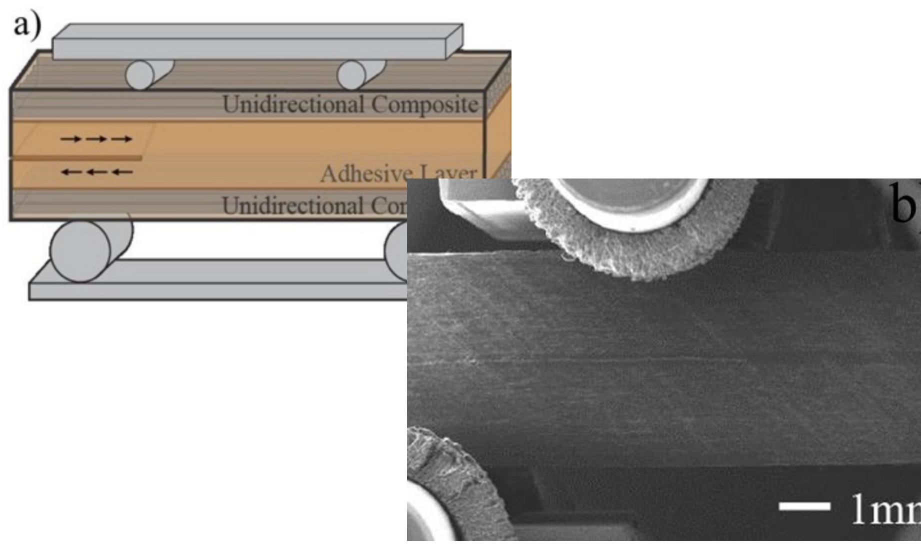
Double Cantilever Beam (DCB) tests are conducted to determine the adhesive critical energy release rate (G_{IC}).

Reveals data for the energy release rate, crack propagation mechanism and provide the dominant mode of failure

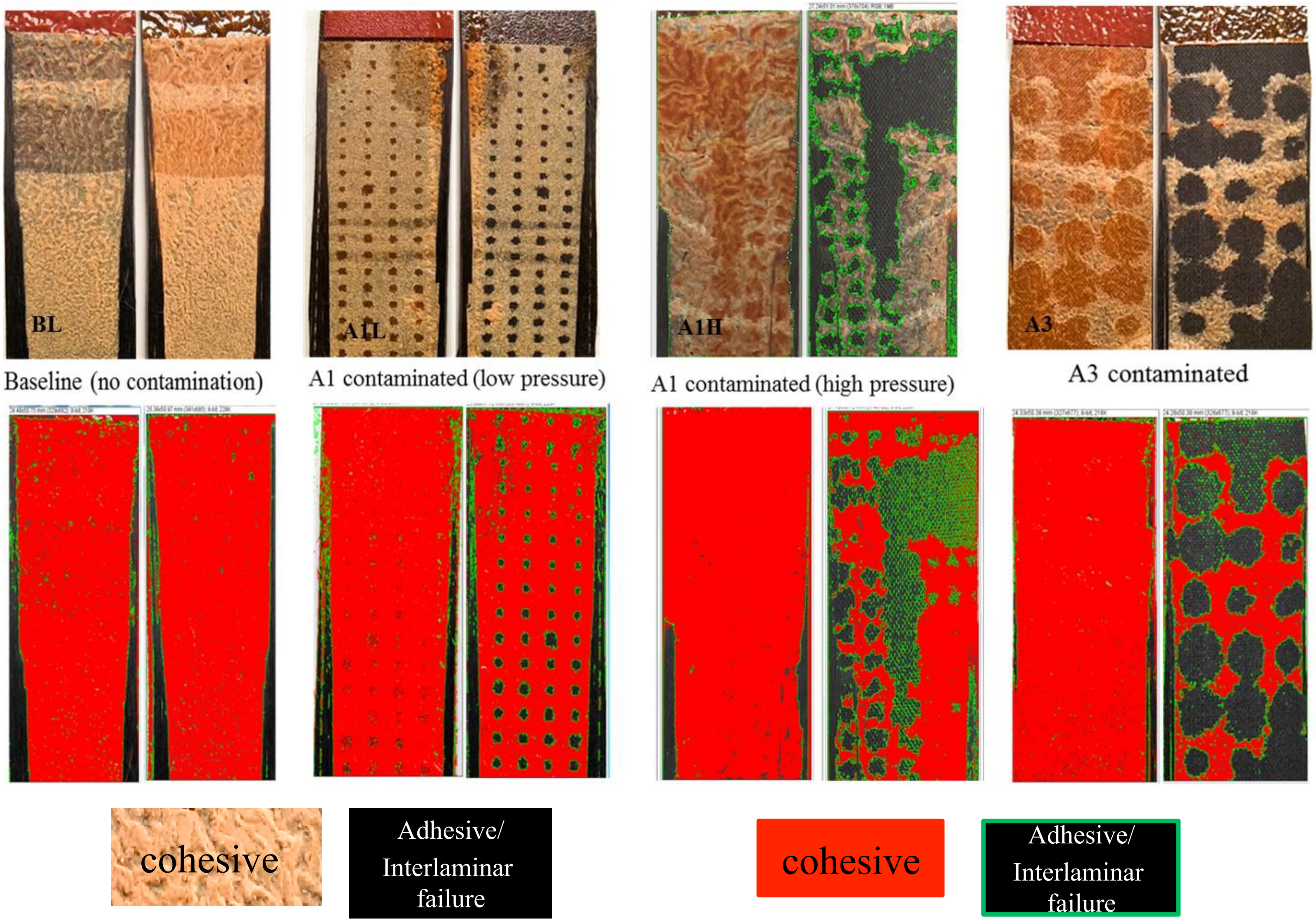
End Notch Flexure (ENF) tests are conducted within an electron microscope (*in situ*) to determine the initiation and propagation of damage.

Reveals mechanisms of damage propagation via crack growth progression and crack opening profiles.

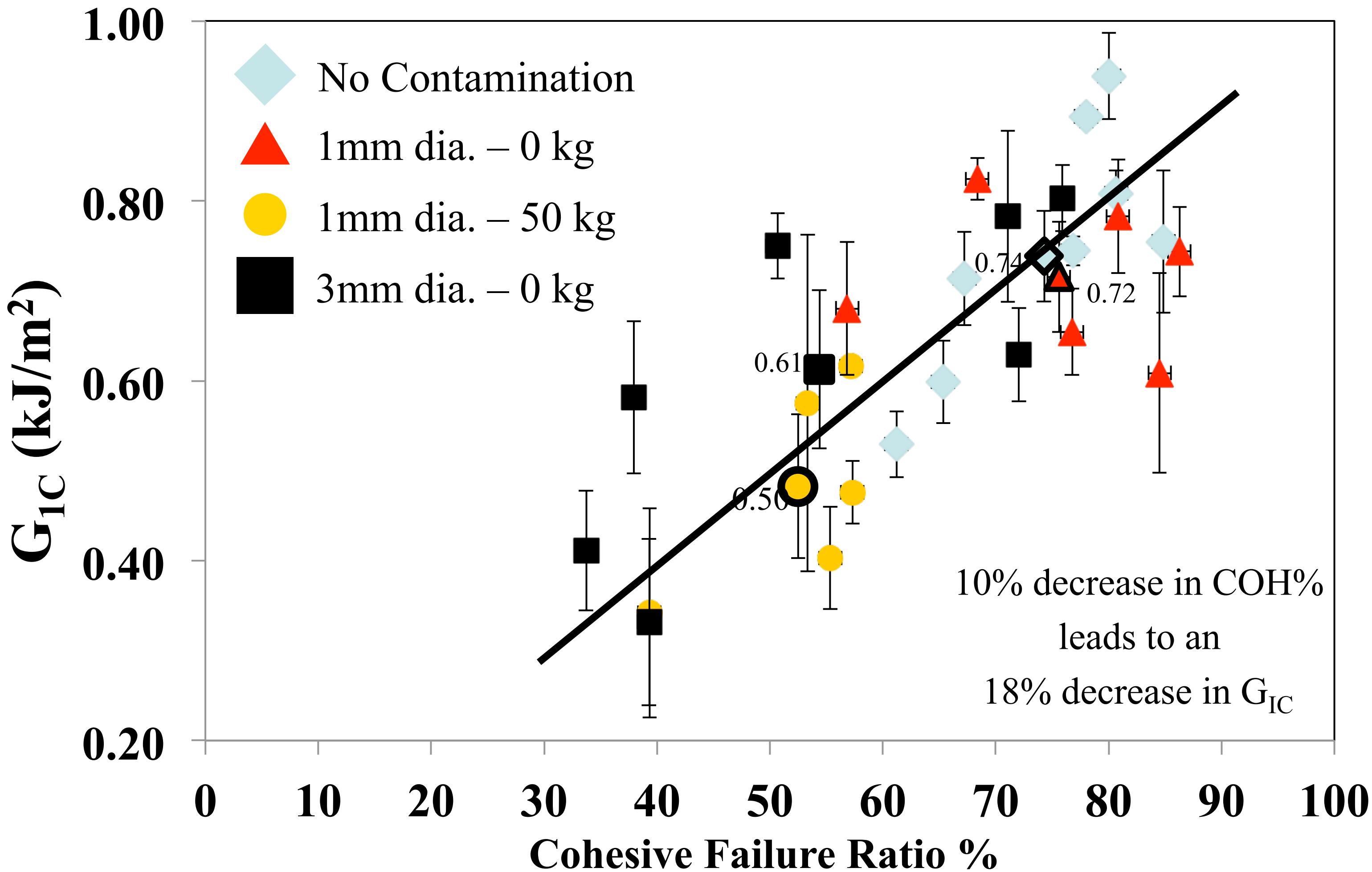
Configuration: Loading rate - 5.0 mm/min in the direction perpendicular to the specimen from one of the edges



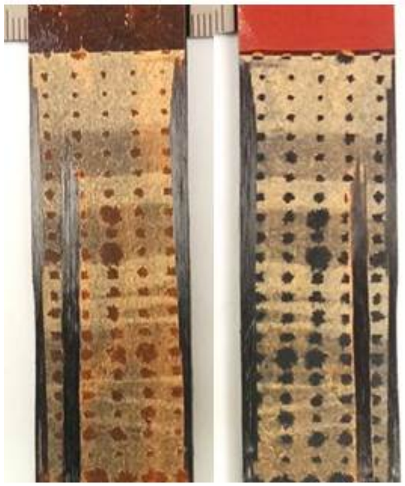
Quantification of Modes of Failure



DCB Testing Results



Comparison of Contamination Level vs. Mechanical Response



A1L-06

G_{1C} - 0.78 kJ/m^2
COH % - 68.38

A3-05

G_{1C} - 0.78 kJ/m^2
COH % - 71.07

Varying Stamp Size
Similar Cohesive Area
Similar Bond Quality

A3-05

G_{1C} - 0.78 kJ/m^2
COH % - 71.07

A3-07

G_{1C} - 0.33 kJ/m^2
COH % - 39.30

Similar Stamp Size
Varying Cohesive Area
Significant Change in Bond Quality

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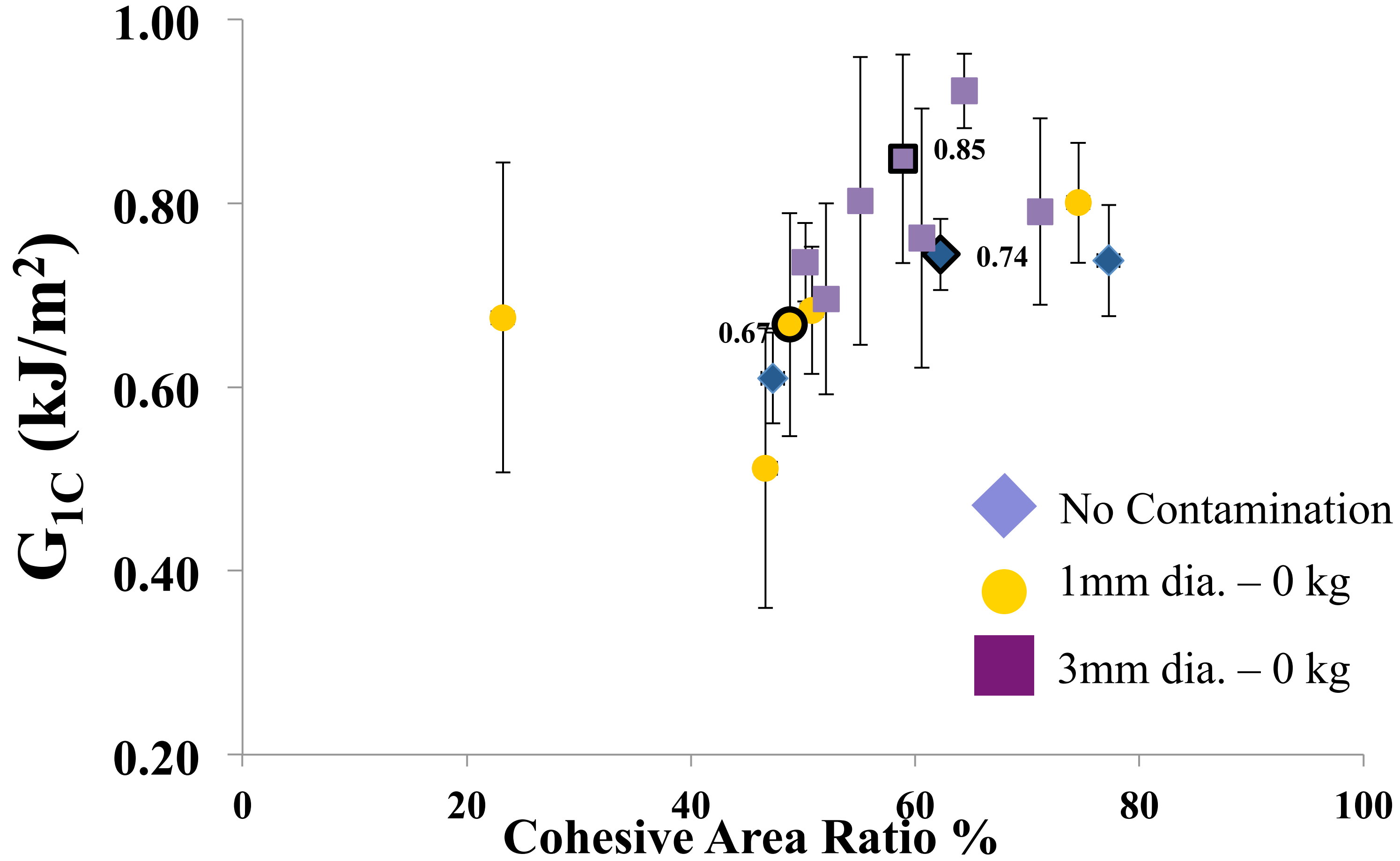
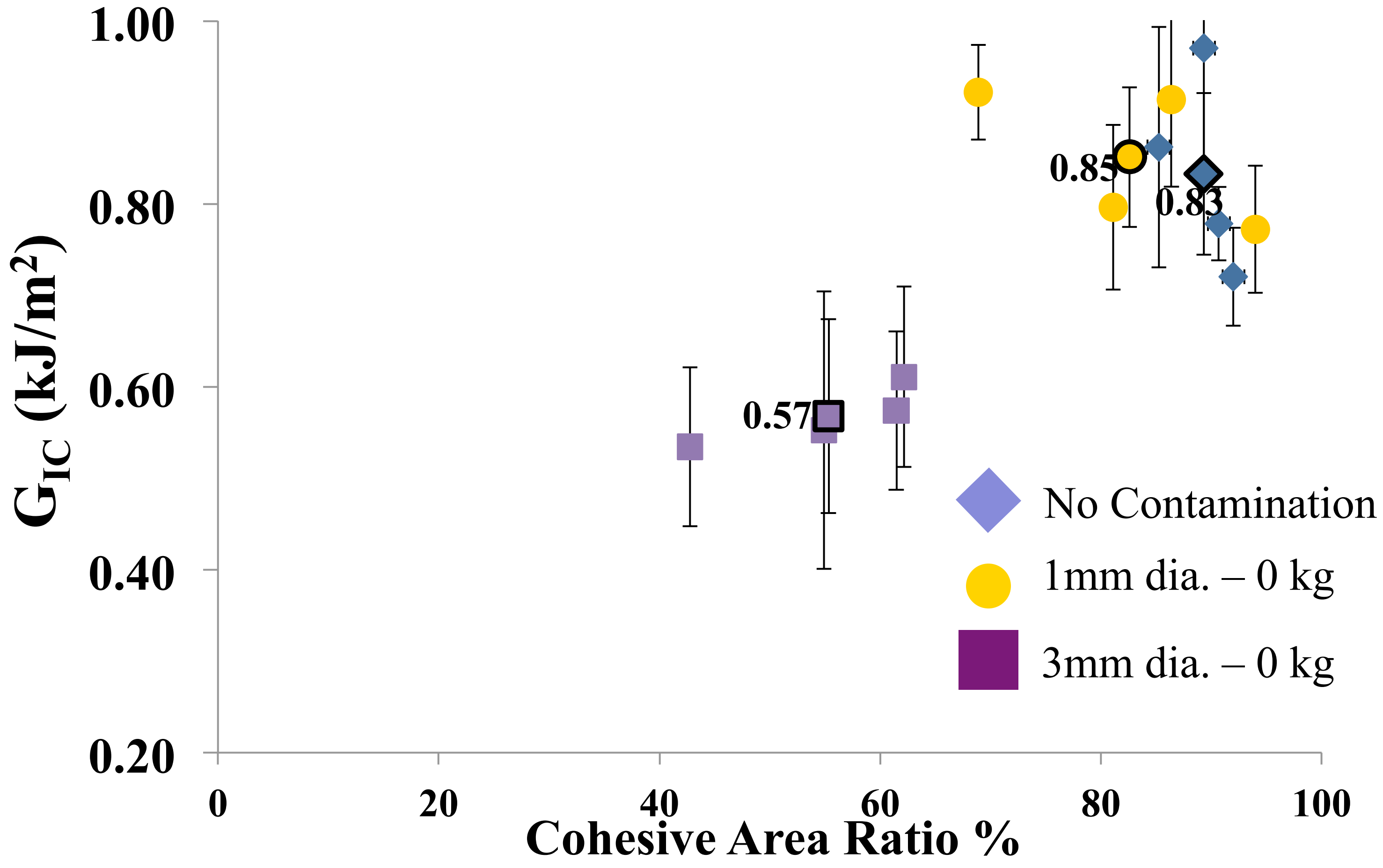
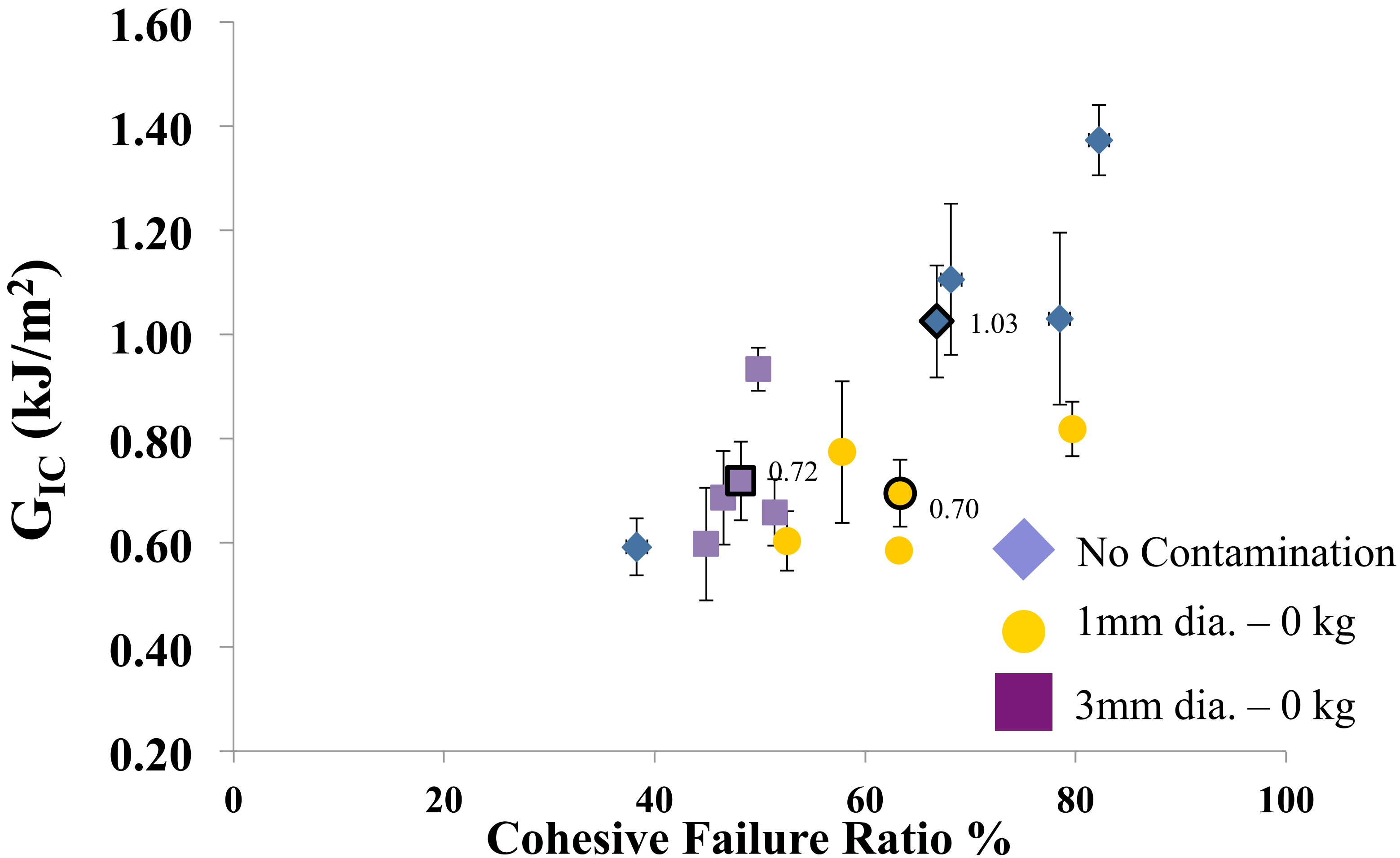
Environmental Conditioning



Fatigue in Ambient Air



Combined Fatigue & Env. Exposure



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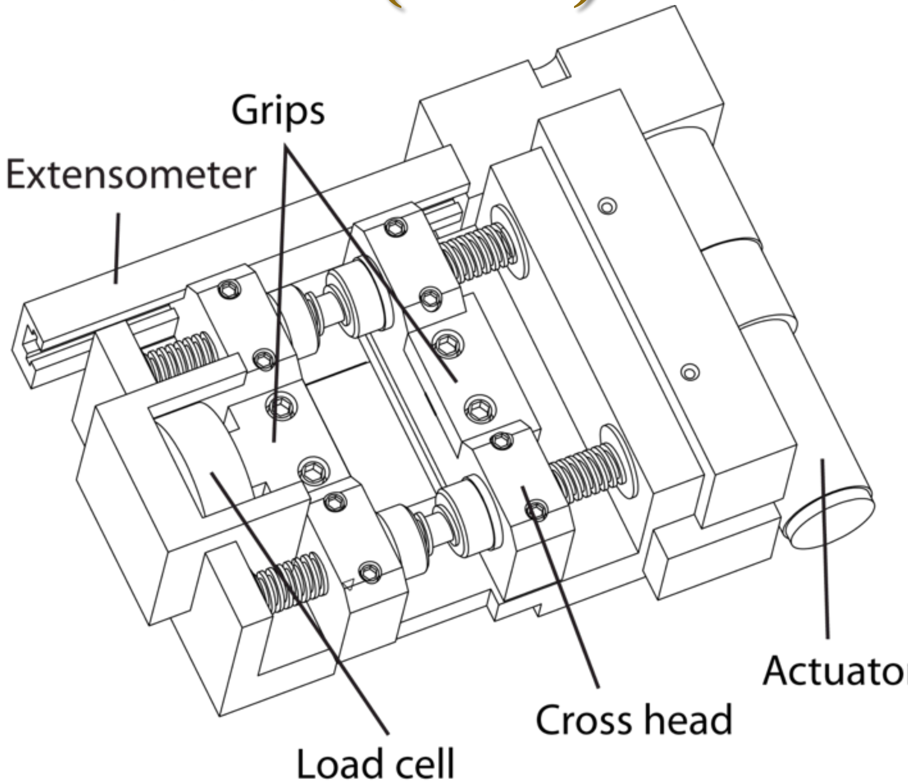
In situ Micro-scale Evaluation
End Notch Flexure (ENF)

Description
In situ load frame for simultaneous loading and imaging of samples within the FIB chamber.

Capabilities
High resolution strain measurement
Programmable loading programs
Very low strain rate are achievable

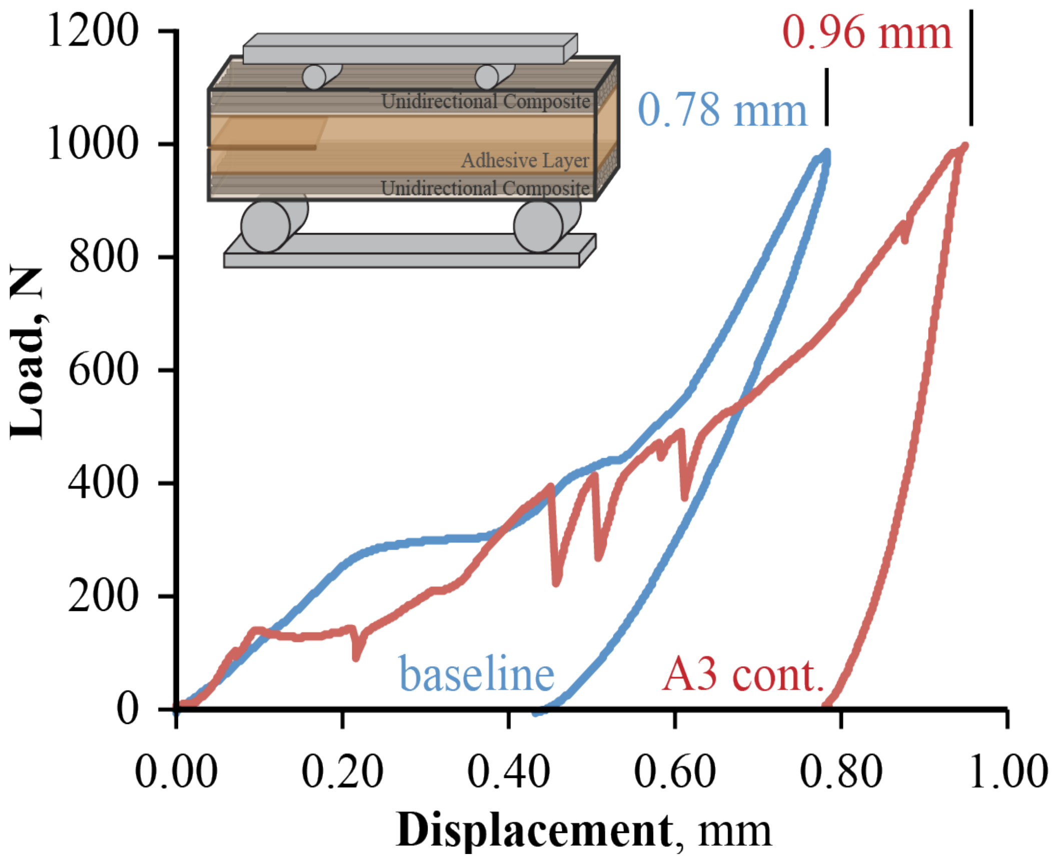
Testing modes
- Tension
- Compression
- Fatigue
- 3 point bending
- 4 point bending
- Fracture
- Compact tension

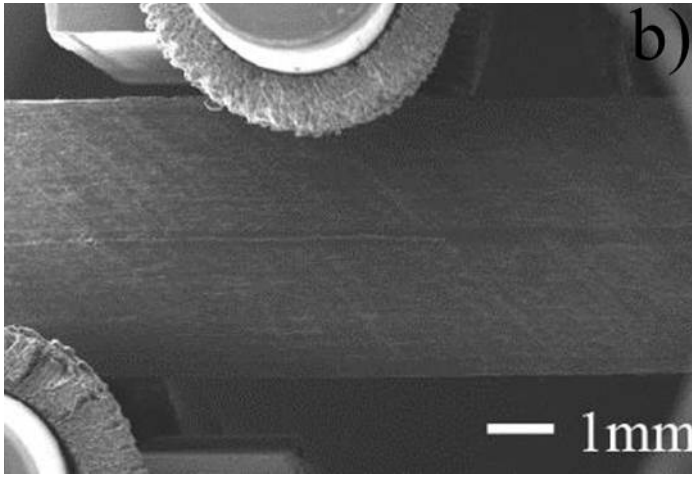


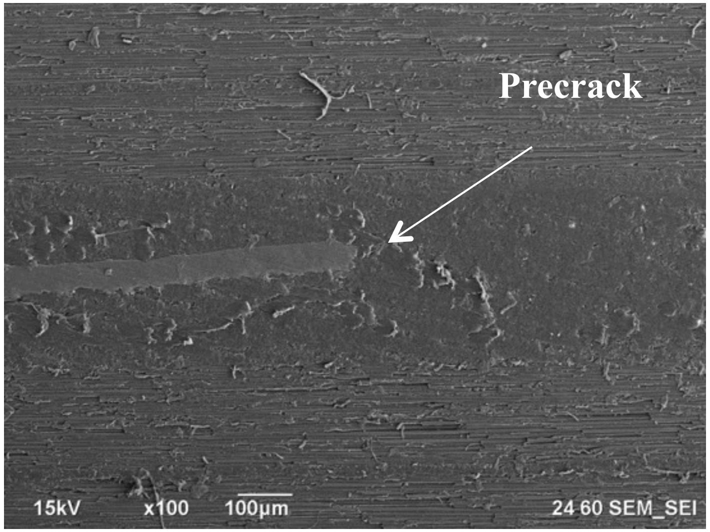
Specifications

Load Capacity	4500N	Max. Strain Travel	30 mm
Load Cell Accuracy	0.2%	Linear Scale Accuracy	20 nm resolution

In situ Micro-scale Evaluation End Notch Flexure (ENF)

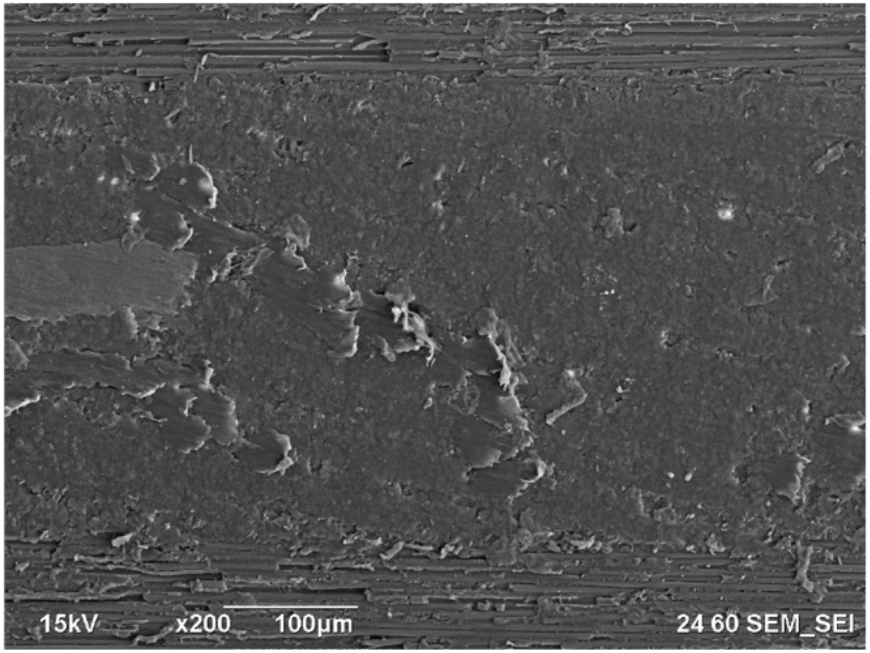




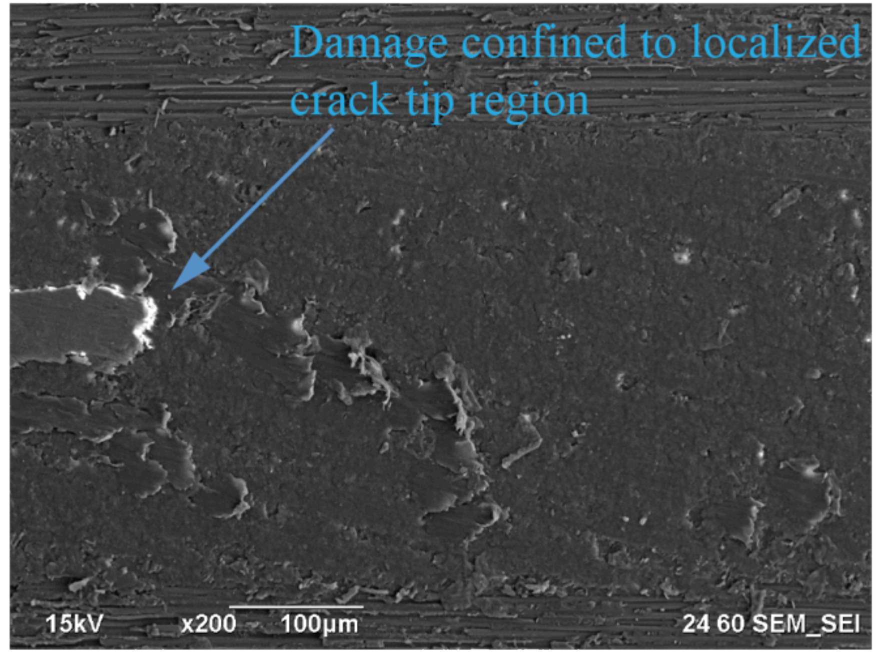


In situ Micro-scale Evaluation
End Notch Flexure (ENF)

Baseline

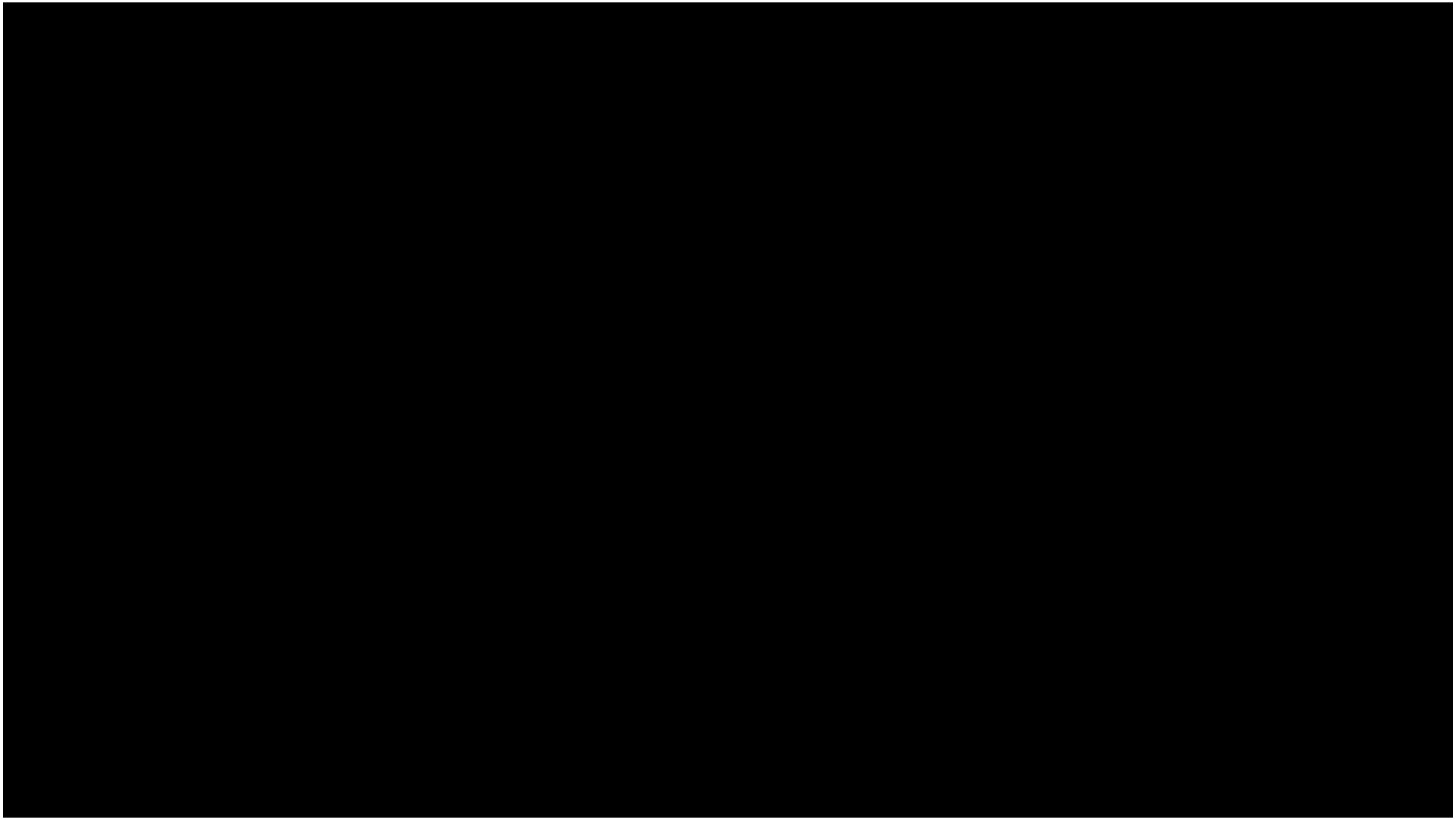


Prior to Loading

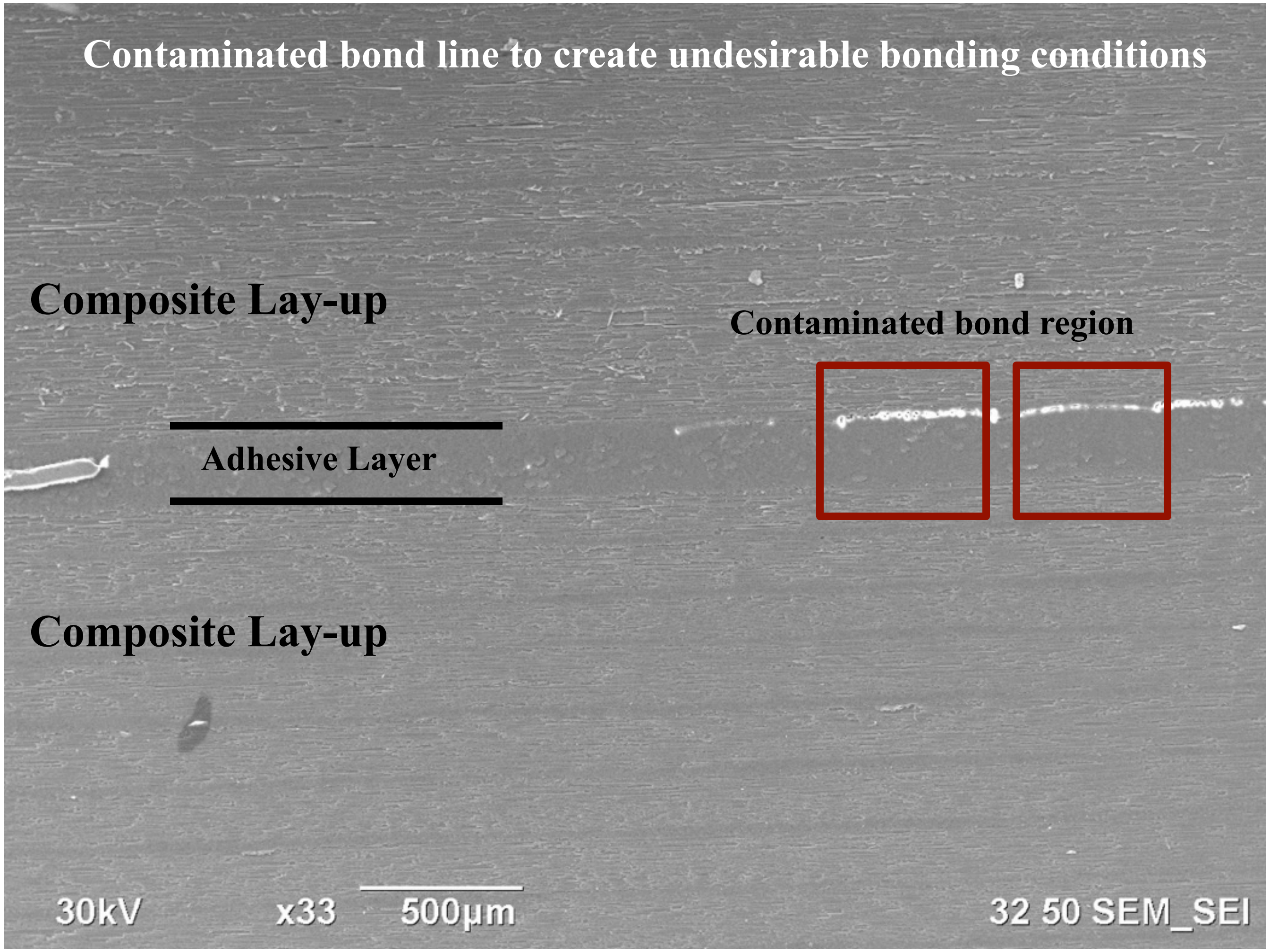


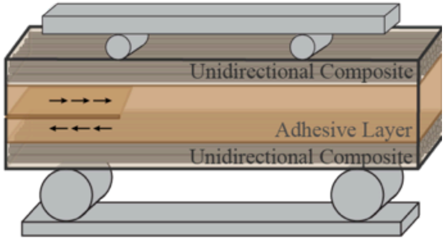
At Peak Load (1000N)

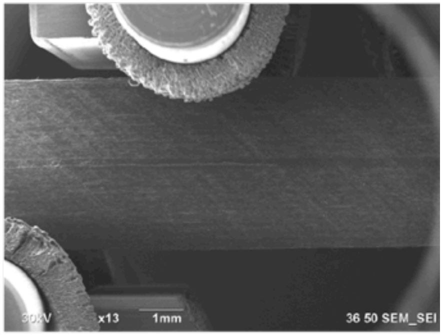
In situ Micro-scale Evaluation End Notch Flexure (ENF)



In-situ Micro-scale Evaluation
End Notch Flexure (ENF)







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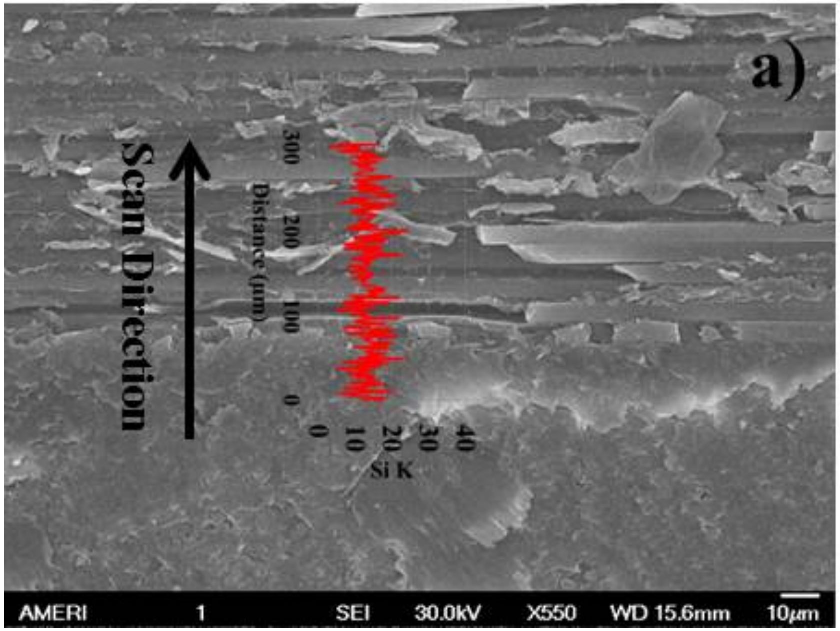
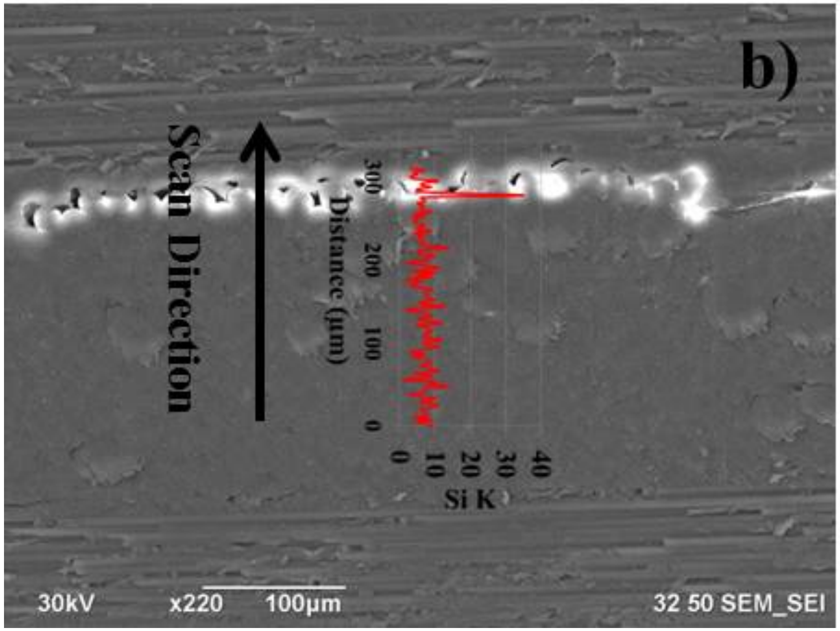
In situ Micro-scale Evaluation
End Notch Flexure (ENF)

EDS Analysis was used to measure Si content along the bondline, at the point of initial failure a significant increase in Si content was detected, indicating the presence of Freekote contaminant



Baseline



Contaminated

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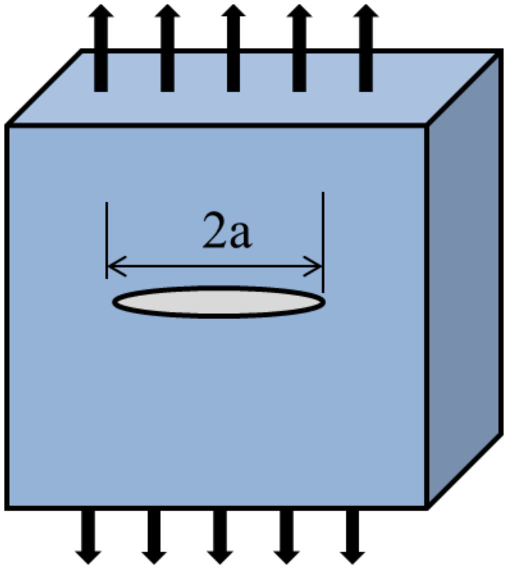
Existing Framework – LEFM Analysis



Penny Shaped Crack embedded in a solid

↓

Solid subjected to remotely applied stress

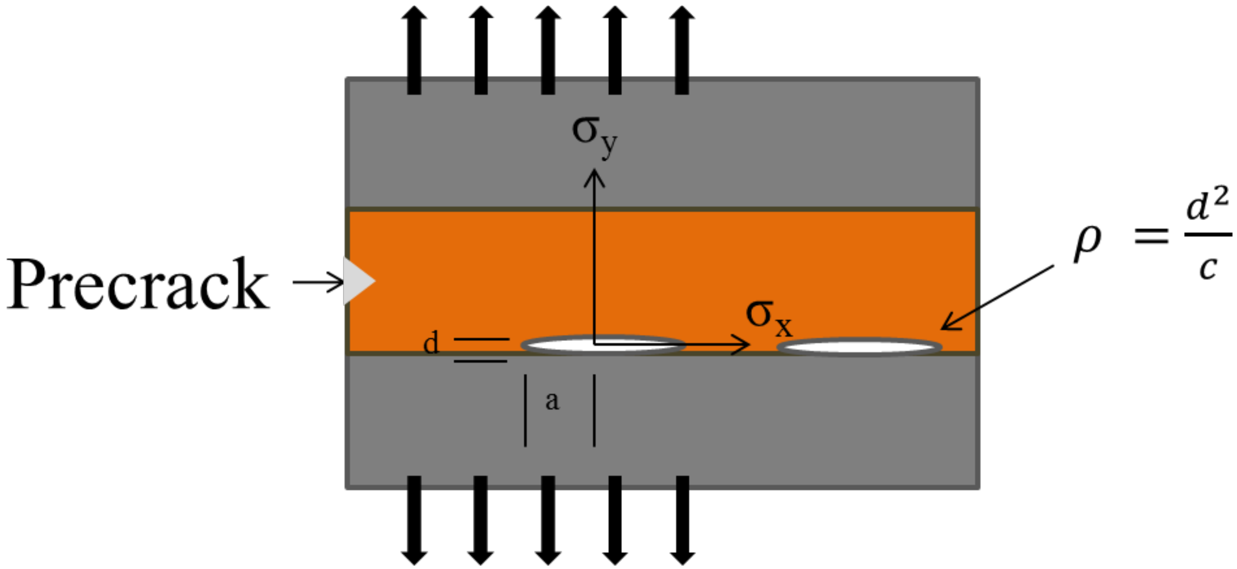
2a is the diameter of the penny shaped crack

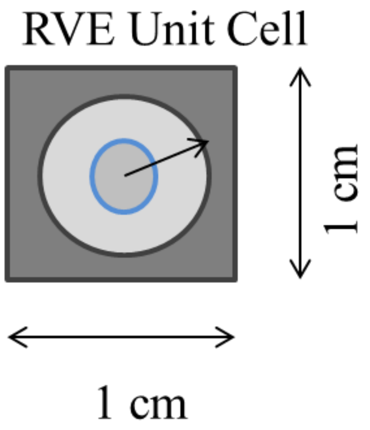
According to Penny shaped crack theories, Stress Intensity factors can be evaluated

Stress Intensity Factor at the crack plane, $K_C = \sigma_y \sqrt{\pi a}$

σ_y - Remotely applied stress

Developmental Framework



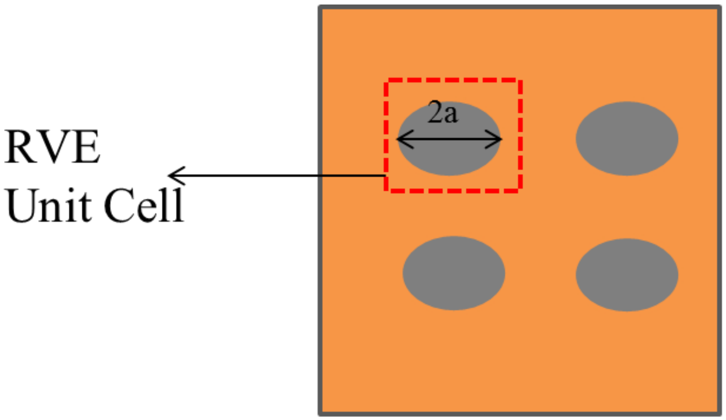


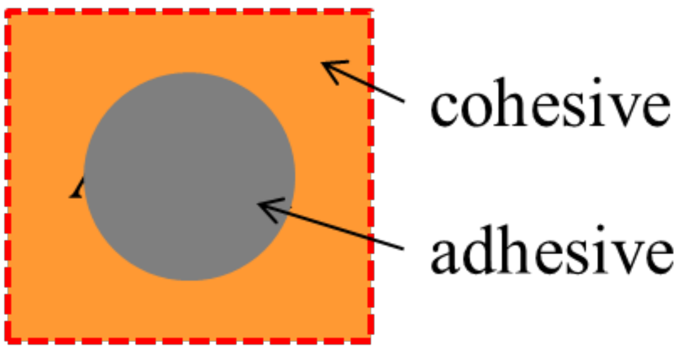
Penny shaped cracks = Contaminated sites.

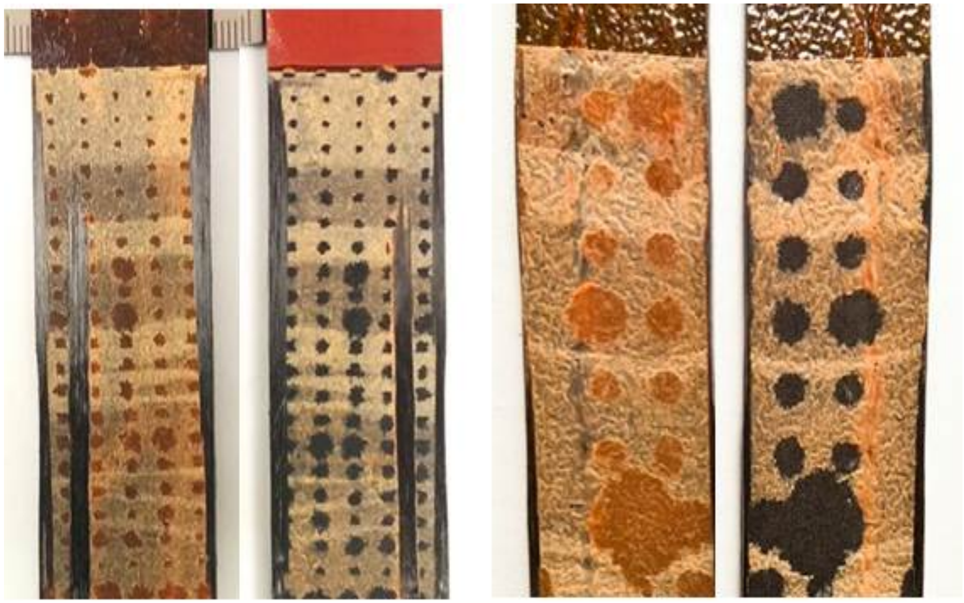
Modifications to the theory:

a) RVE Unit Cell considerations
b) Crack size as varied in a RVE Unit Cell

Developmental Framework







A1L-06

G_{1C} - 0.78 kJ/m^2
COH % - 68.38

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G_{1C} - 0.78 kJ/m^2
COH % - 71.07

Developmental Framework

Stress Intensity factor K_c for RVE Unit Cell

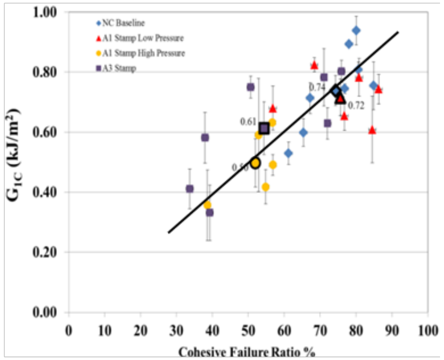
Relationship between Stress Intensity Factor, K_C and **Energy Release Rate** G_C

$$K_C = \sqrt{E\, G_C}$$

$$G_C = \frac{{K_C}^2}{E}$$

$$G_C = \frac{(A_{cohesive} * \sigma_s \sqrt{\pi a})^2}{E}$$

Input Parameters



Typical Structural Adhesives possess

Young's modulus between 3 GPa

Tensile Strength between 4-6 Mpa

Varying Crack Flaw Size from 2 nm - 5µm

Output Parameter

The relationship between critical energy release rate and cohesive area

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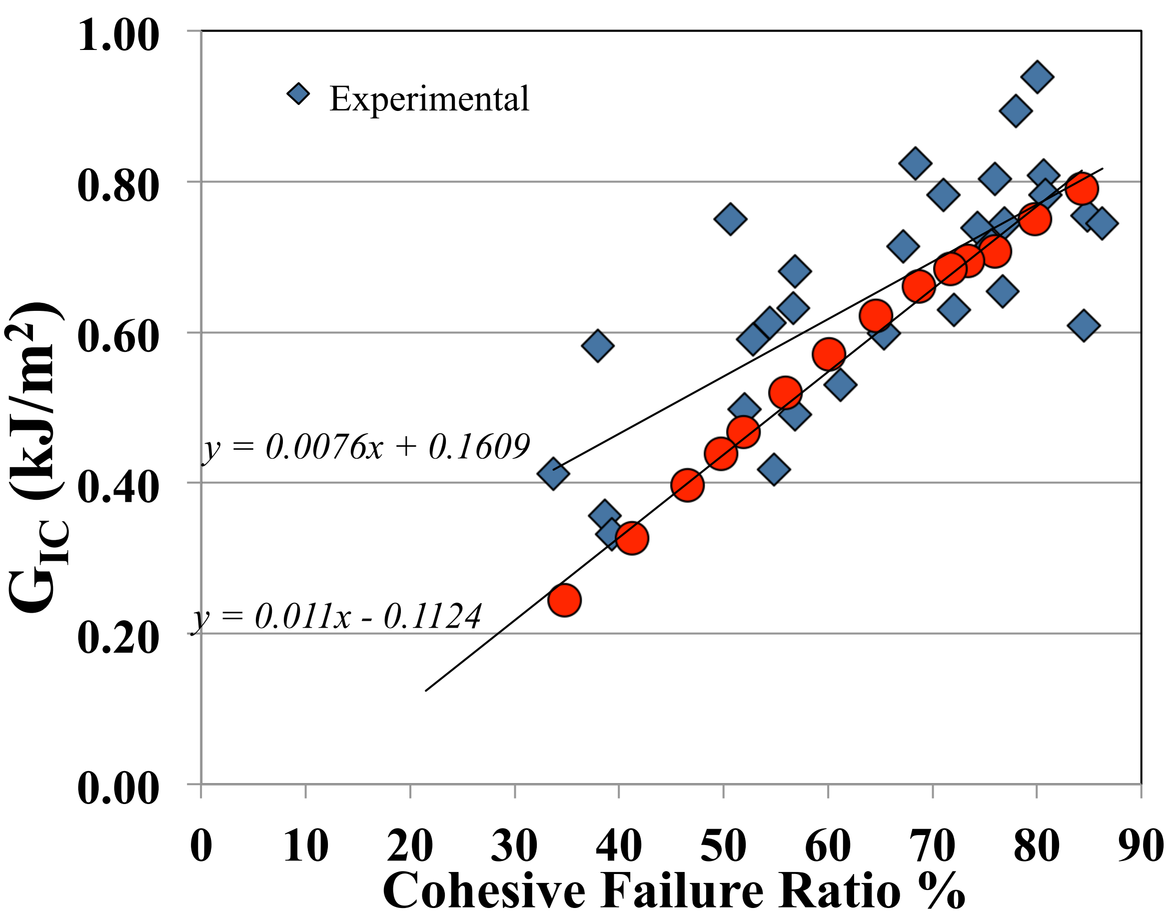
Experimental vs Predicted

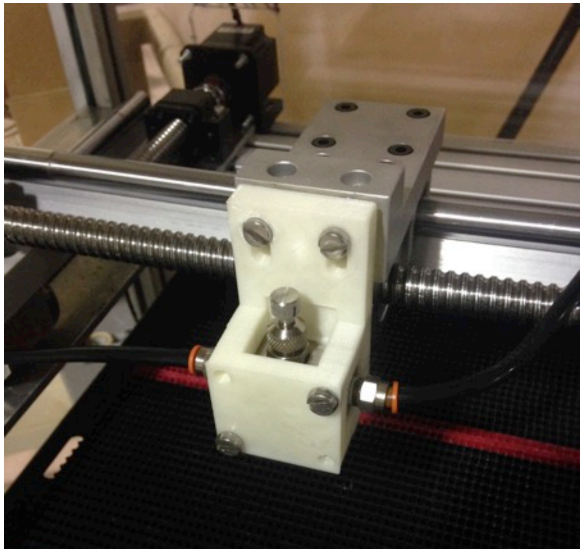


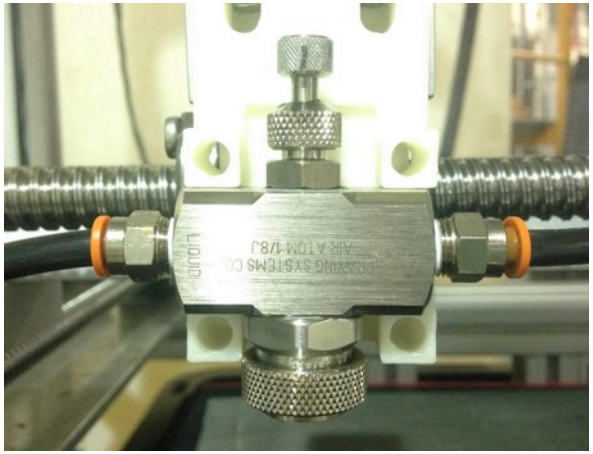
Tensile Strength – 4.6 MPa & Young Modulus, E= 3 GPa

Uniform Contamination Approach

Spraying Approach

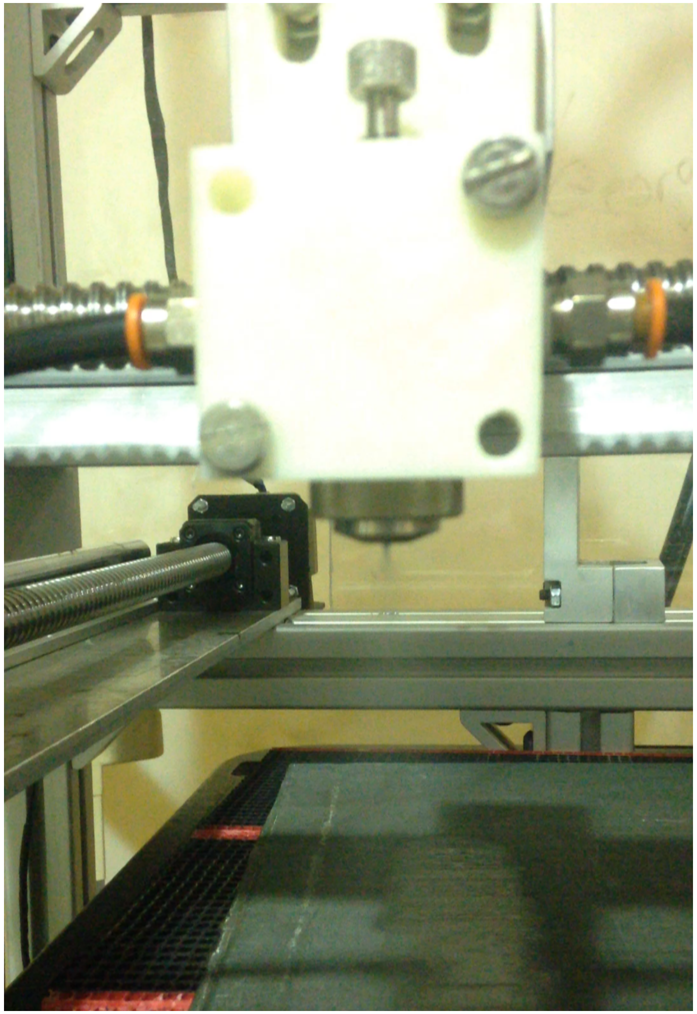
- Uniform spraying of contaminant and surfactant using Frekote and Hexane at various concentration levels
- Emulates factory conditions of contamination on laminates prior to bonding
- Potential for creating various levels of a weak bond – by adjusting the concentration of Frekote and speed of application
- Allows a comprehensive choice of contaminants i.e. all low viscous fluids can be easily adopted to meet the needs of a wide variety of applications.





Alternate Contamination Approach

Task 1	Nozzle selection
Task 2	Selection of Spraying Time
Task 3	Varying levels of contamination [i.e., Frekote mixed with 25% Hexane, 50% Hexane and 75% Hexane]
Task 4	Surface characterization using gravimetric analysis and FTIR



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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 Support

Recent Activities

- M&P group developed draft outline for Vol. 3 Sec 5.9.1 Assembly Processes for Bonded Joints
- Outline content is extensive and provides a framework for creating a volume dedicated to bonding in the future.
- Working group recently held at FIU to begin the process of populating the outline with information. (FIU, 3M, Embraer, Lockheed, FAA).

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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Expanded Vision: New Volume focused on Bonding

CMH-17 Volume 7 Bonding of Thermoset Composite Structures

1. General Information
2. Material Data
3. Guidelines for Property Testing
4. Design and Analysis of Bonded Joints
5. Assembly Processes
6. Quality Control
7. Supportability
8. References

Current Vol 3 Section 5.9 becomes Vol 7 Section 5 with simultaneous optimization to Volume 3 Rev H

- Volume 3 Section 5.9.1 points to Volume 7
- New content for bolted and hybrid joints?
- Many revisions under development
- Relevant adhesive and join content moved to Volume 7 with pointers from Volume 3

By CMH-17 Meeting in August:

- General Outline for discussion and comment
- Existing content map with proposed changes to Vol 3
- Gap analysis

5 Assembly Processes

- **5.1 Introduction**
- **5.2 General Considerations**
  - **5.2.1 Types of Bonds**
  - **5.2.2 Definitions**
- **5.3 Secondary Bonding**
  - **5.3.1 General Considerations**
  - **5.3.2 Quality Considerations for Bonding**
  - **5.3.3 Surface Preparation**
  - **5.3.4 Protecting the Prepared Surface**
  - **5.3.5 Adhesive Application**
  - **5.3.6 Bond Assembly**
  - **5.3.7 Adhesive Cure**
  - **5.3.8 Bond Inspection**
- **5.4 Co-curing**
  - **5.4.1 Advantages**
  - **5.4.2 Special Considerations**
- **5.5 Co-bonding**
  - **5.5.1 Advantages**
  - **5.5.2 Special Considerations**
- **5.6 Multi-Stage Bonding**
- **5.7 References**

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Conclusions/Summary

Future Work:

- In situ analysis of fatigued and environmentally exposed samples to examine fracture properties and damage initiation.
- Investigate additional contamination procedures to change surface chemistry and determine fracture properties of additional cases.
- Change contaminate application locations and dimensionality to investigate additional morphologies.

Benefit to Aviation:

- Better understanding of durability assessment for adhesively bonded composite joints.
- Assisting in the development of bonding quality assurance procedures.

Questions ?

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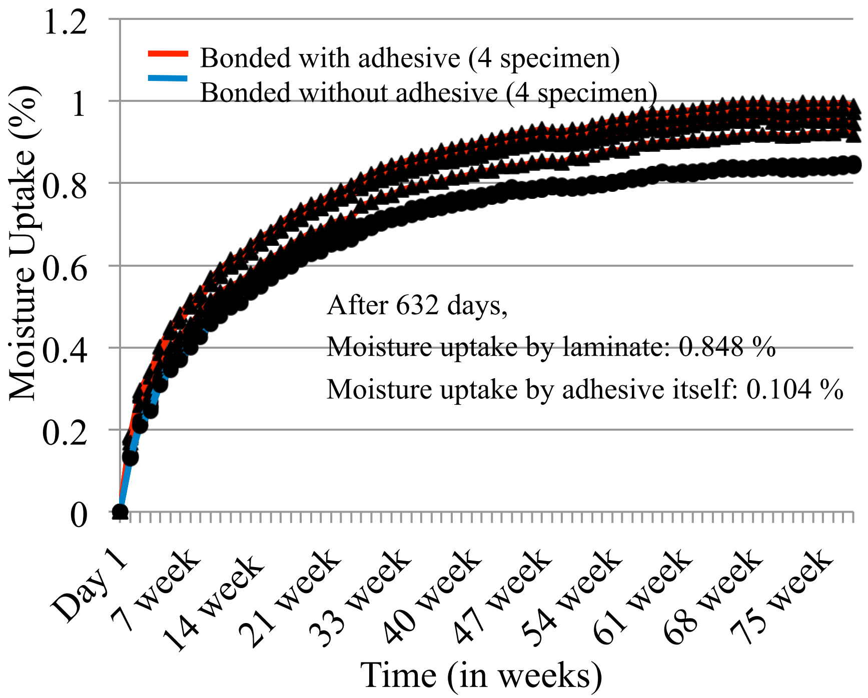
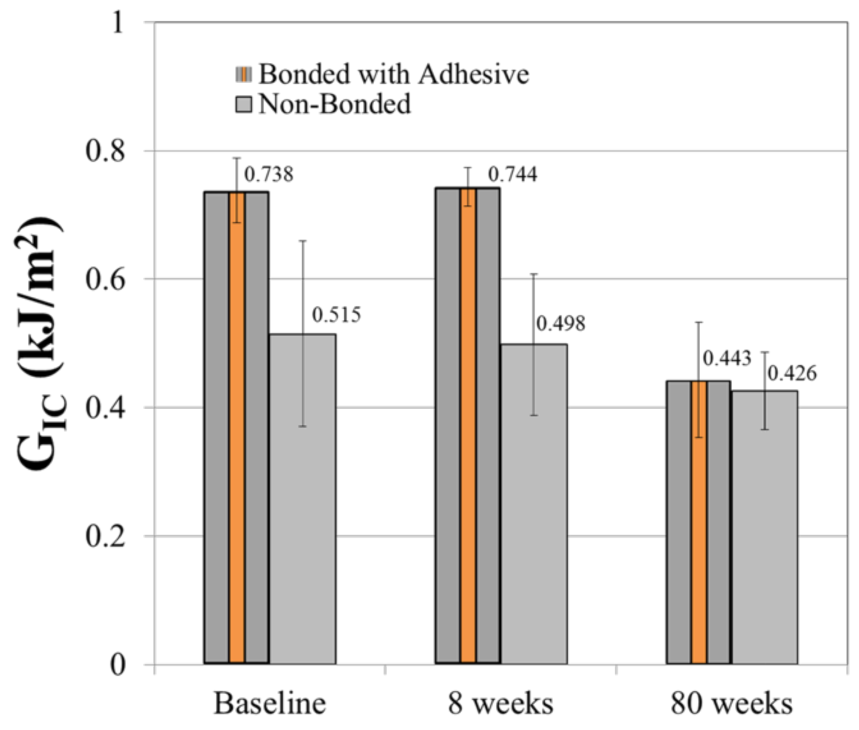
Weight Measurements and Fracture Toughness Testing - DCB

Moisture Uptake Analysis

– determine saturation limit at 50°C, 95% RH





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