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Effect of Surface Contamination on Composite Bond Integrity and Durability

Dwayne McDaniel

Florida International University

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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 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 & Researchers**
  - Dwayne McDaniel, Xiangyang Zhou
- **Students**
  - Vishal Musaramthota
- **FAA Technical Monitor**
  - Curt Davies
- **Industry Participation**
  - Exponent, NRC, 3M

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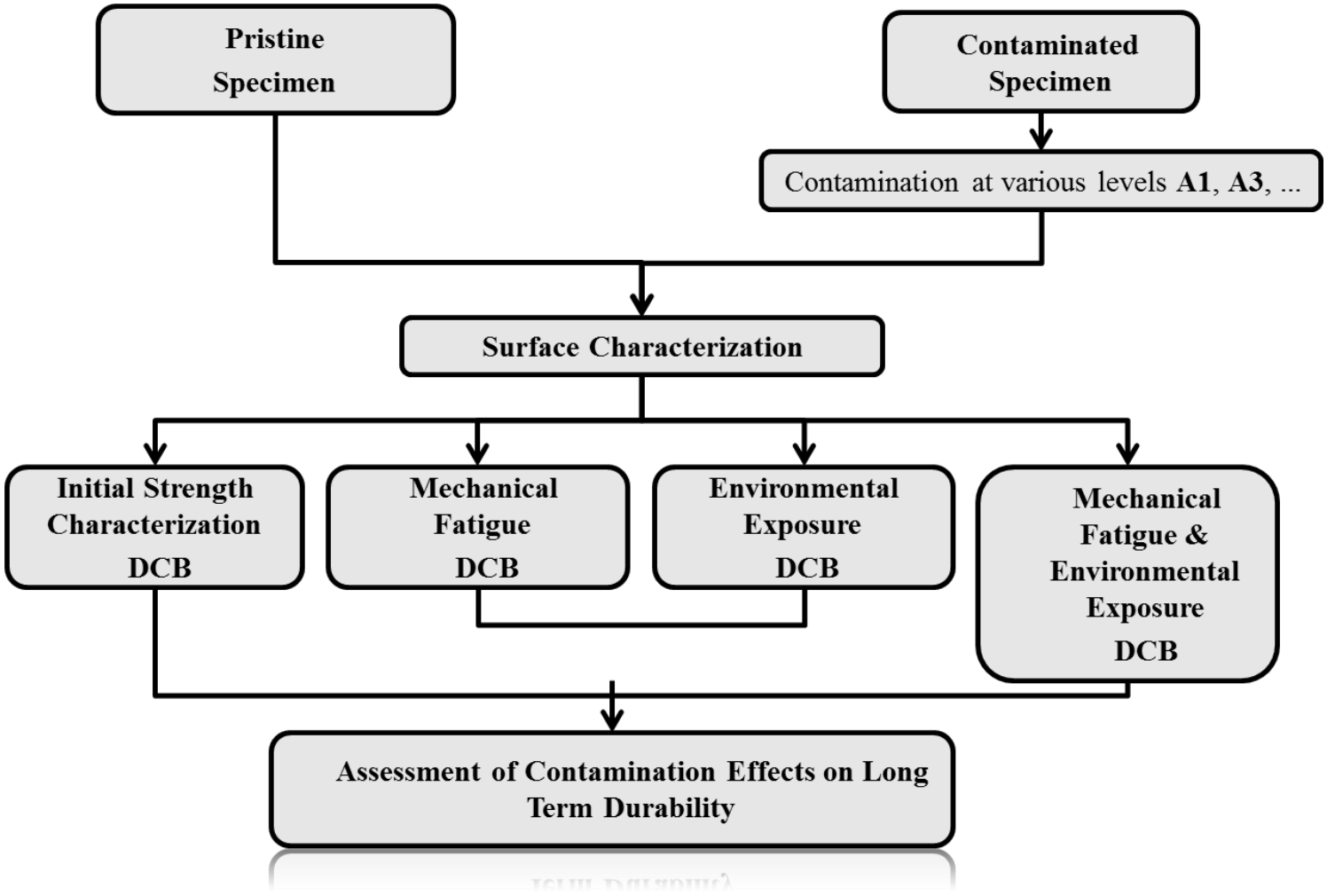
Durability Assessment Procedure



Bonding Material System

- 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 mils, 350F cure)
  - Precision Fabric polyester peel ply 60001
  - Silicone Spray from CBS Aerosol & Paint, Inc
  - 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

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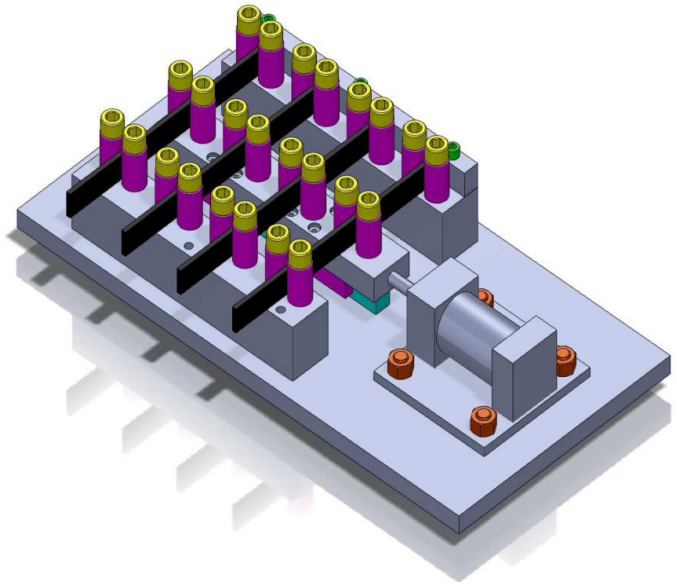
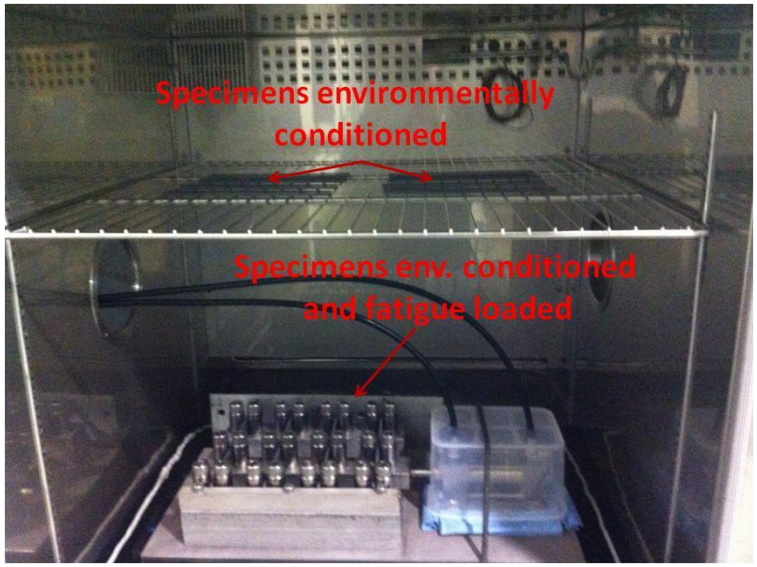
Fatigue Fixture and Accelerated Aging Procedure

The fatigue fixture can be placed in the environmental chamber to study the combined loading and environmental effects.

- Manufactured using stainless steel materials
- Center section slides on a ball bearing carriage
- Designed to load up to four 11.5 in specimens with a deflection up to 2 inches DA
- Current stainless steel pneumatic /hydraulic actuator is rated to 400 psi with a 1 inch bore diameter
- Pneumatic controller can operate up to 2 Hz at 150 psi



Rendering of fatigue fixture



Environmental chamber with fatigue fixture

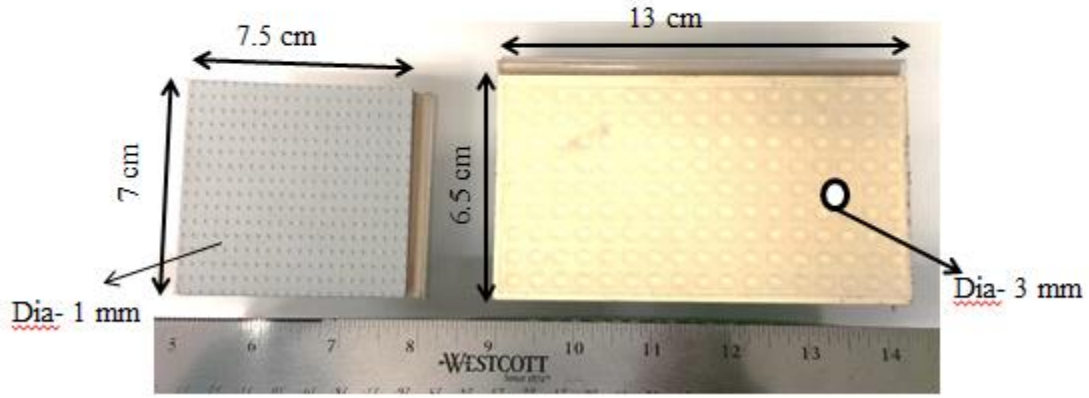
Contamination Procedure

Undesirable bonding conditions will be used to evaluate how the specimen conditioning can effect durability. The contamination procedure aims to create weak bonds by placing a spatially ordered array of contaminated areas on the surface prior to bonding.

Stamped Approach

A1 Stamp

A3 Stamp

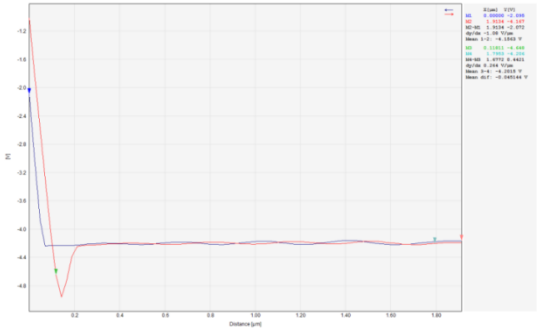


Varying levels of contamination !!!!

Surface Characterization Methods

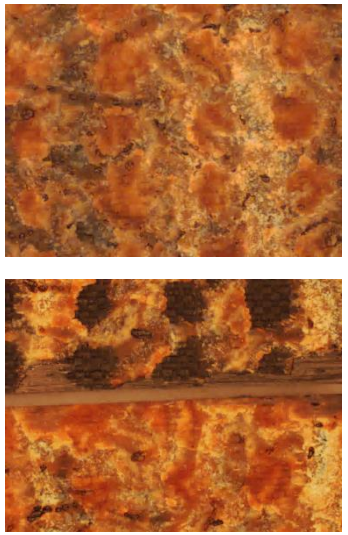
Contaminated Specimens

- FTIR data can be used to identify surface molecular bonds – identify contamination.
- Water contact angle - Determines the wetting characteristic behavior of various liquids on the composite surface.
- AFM –can record the attraction/repulsion forces between the AFM probe and the surface. This data is used to generate topography and force volume measurements to quantify changes in adhesion forces.
- Optical Microscopy- Inspecting fracture surfaces, Line profile analysis.



Force curve on the peel ply imprint peek obtained

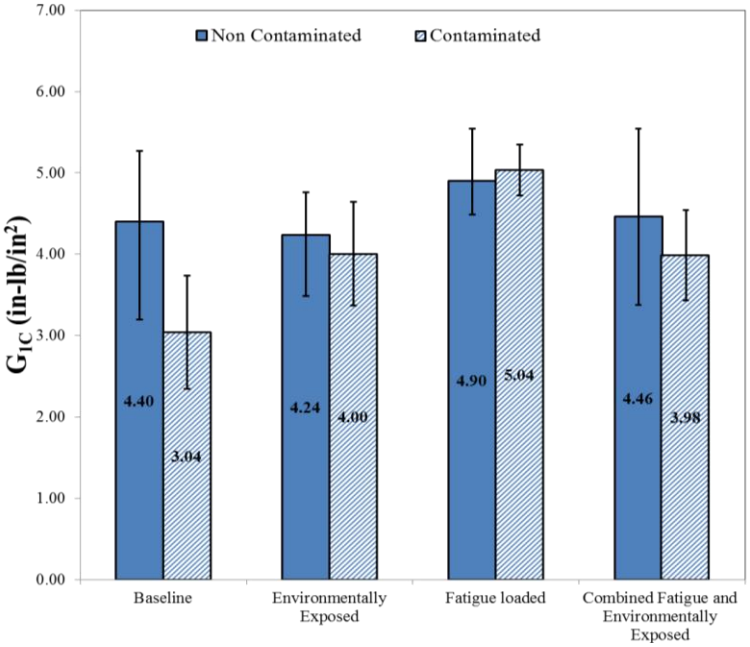
Contact Angle image of CFRP substrate



Optical Microscopy image of failed surfaces

Previous Results – A1 Measurements

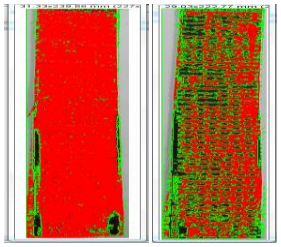
Fracture Toughness



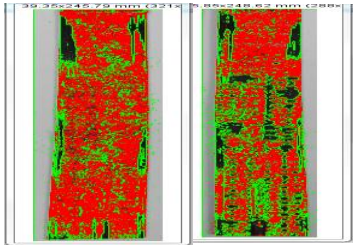
Bondline Thickness

Specimen	Non Contaminated	A1 Contaminated
Baseline	9.29 mils	12.99 mils
Environmentally Exposed	14.72 mils	13.86 mils
Fatigue	15.47 mils	13.50 mils
Combined Env. Exposed+ Fatigued	11.96 mils	12.71 mils

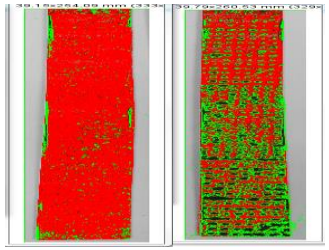
Mode of Failure Analysis



Baseline



Environmentally exposed



Fatigue in air

Gravimetric Analysis

	Before	After	Gain %
A1 stamp	122.41	122.89	0.39

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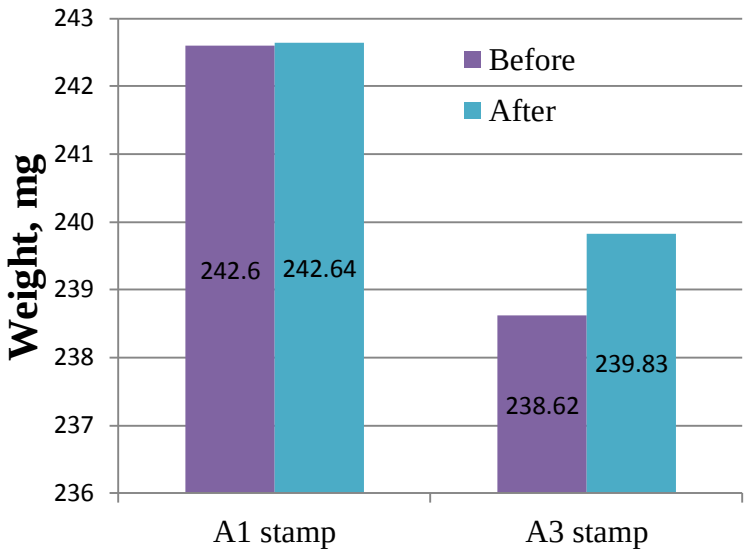
Current Results
Gravimetric Analysis and Bondline Thickness

Gravimetric Analysis

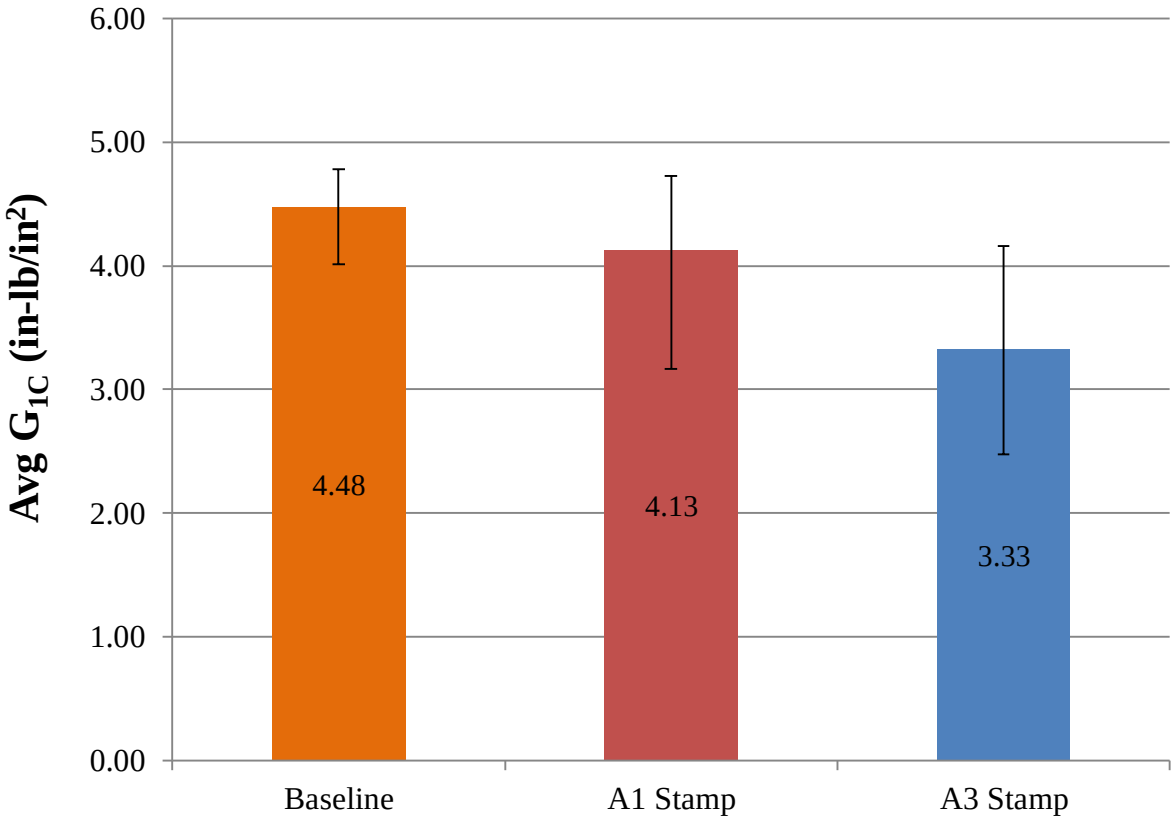


	Before	After	Gain %
A1 stamp	242.6	242.64	0.017
A3 stamp	238.62	239.83	0.507

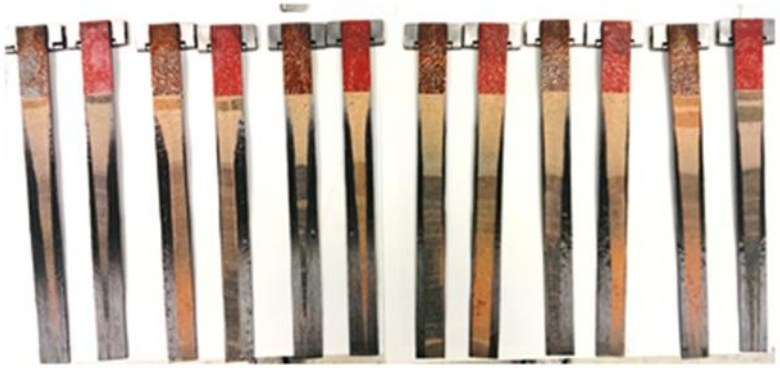
Bondline Thickness Averages

Baseline	A1 Contaminated	A3 Contaminated
11.29 mils	10.09 mils	11.29 mils

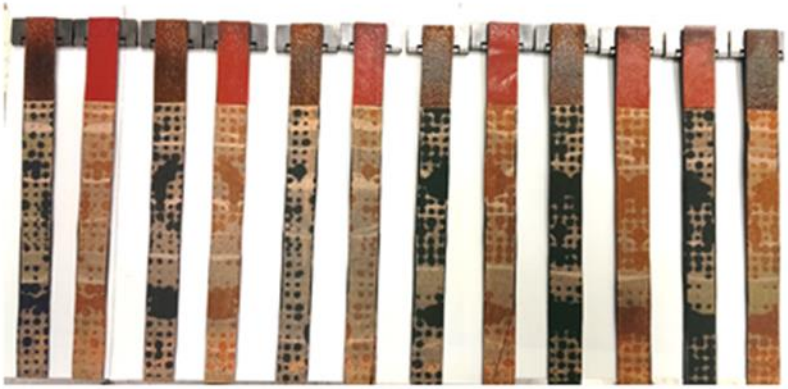
Fracture Toughness Testing- DCB



Fracture Toughness Testing- DCB



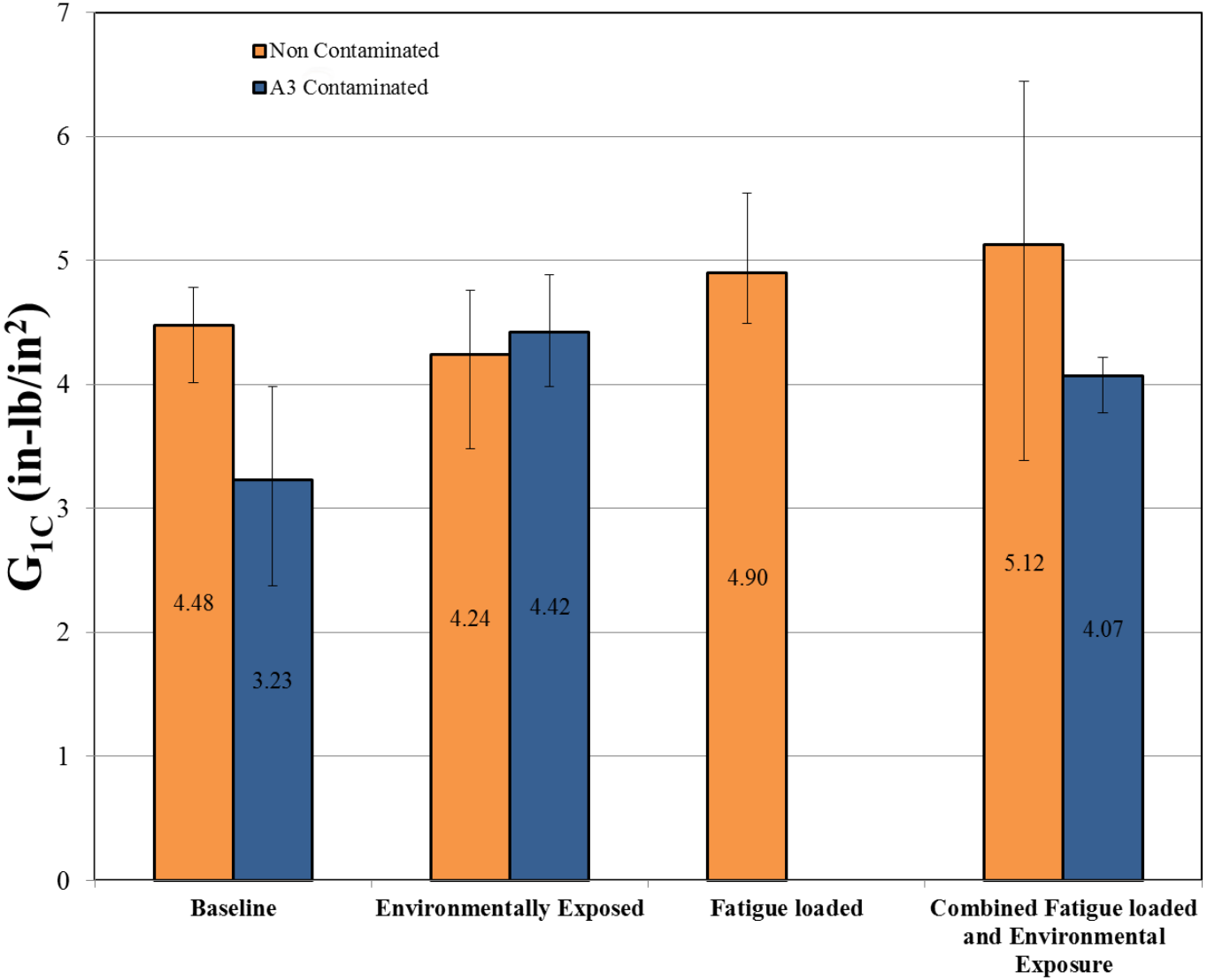
Baseline (Non-Contaminated)



A3 Contaminated

A1 Contaminated

Fracture Toughness- *Current Results*



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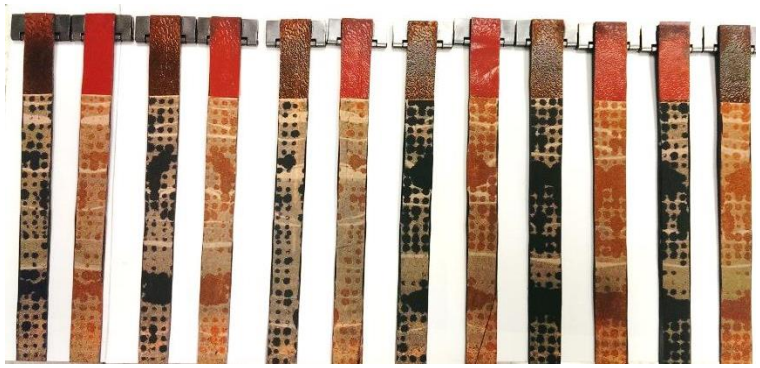
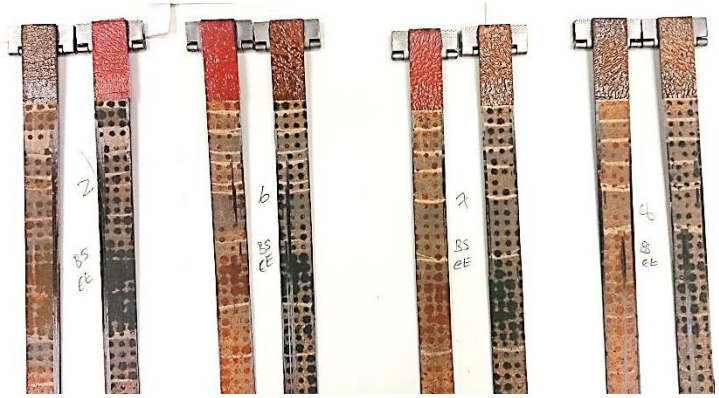
Mode of Failures- A3 Results

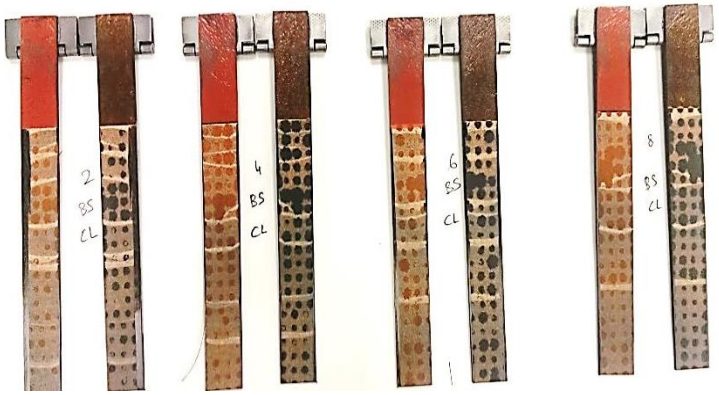


A3- Baseline



A3- Environmentally Exposed

On going

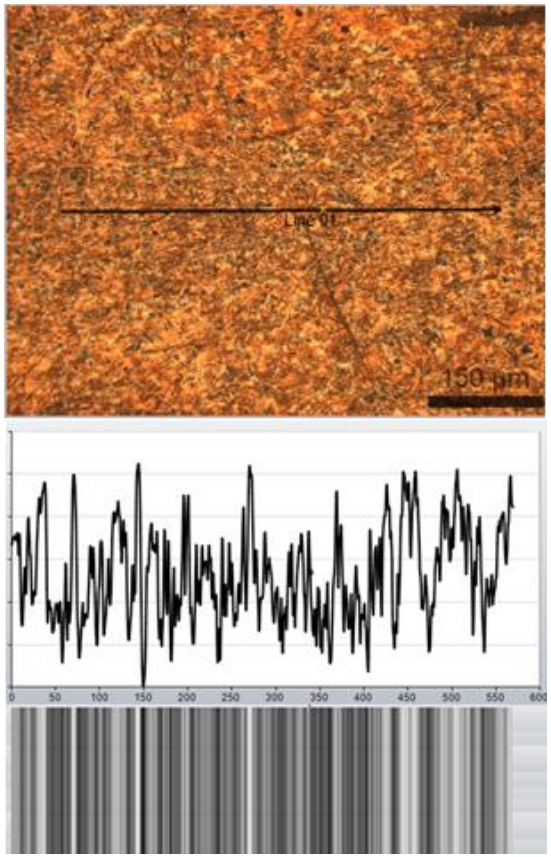


A3- Combined loading
(Fatigued + Environmentally Exposed)

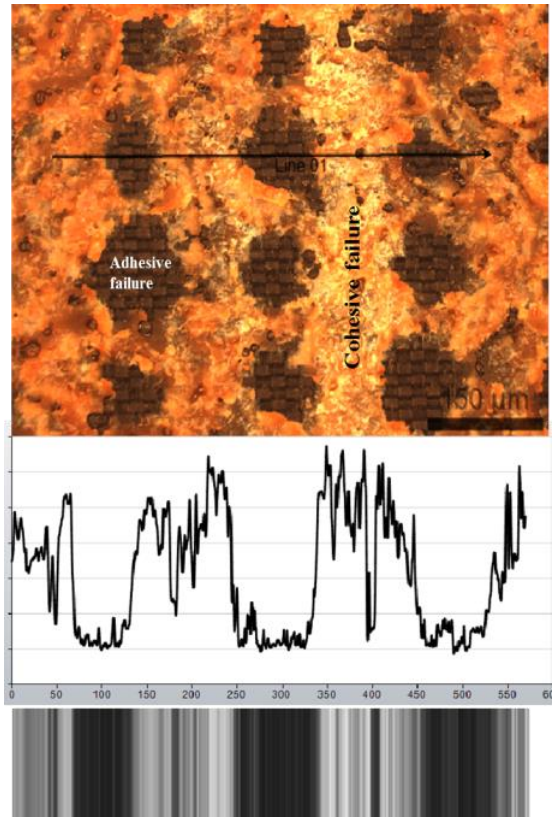
A3- Fatigue

Fractography- Line Profile Analysis

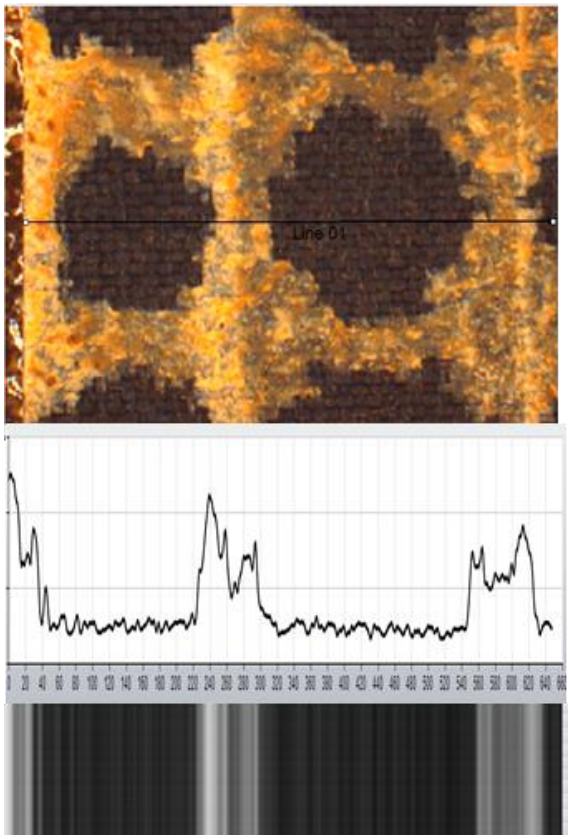
Non-Contaminated



A1-Contaminated



A3-Contaminated

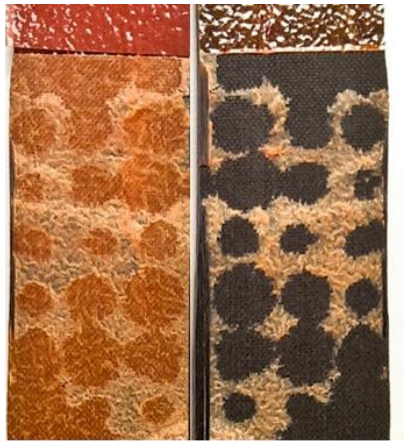


Quantification of Modes of Failure

The modes of failure are quantified for correlation with bond strength

Baseline (no contamination)

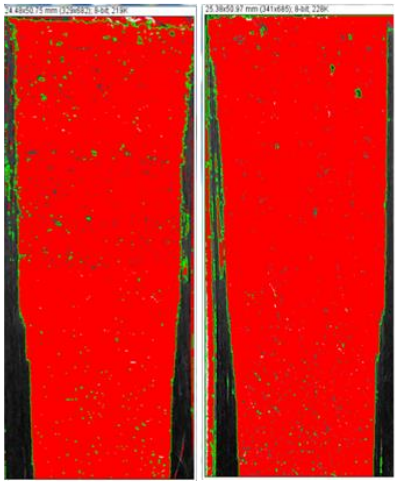
A1 contaminated

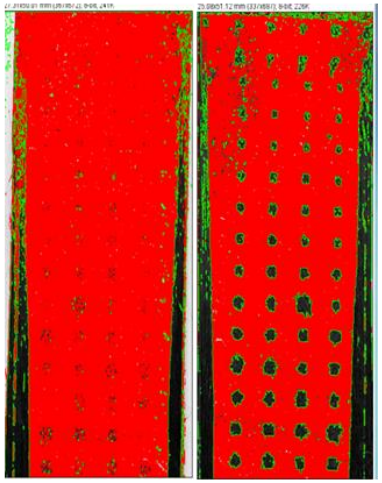


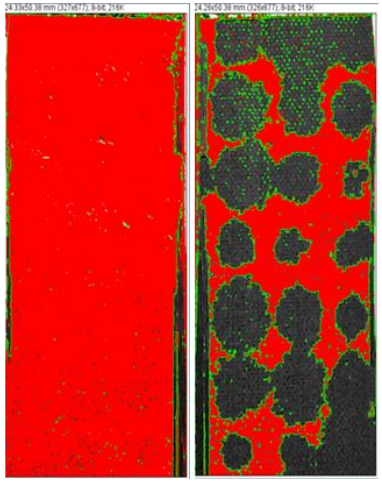
A3 contaminated

cohesive

Adhesive/ Interlaminar failure







cohesive

Adhesive/ Interlaminar failure

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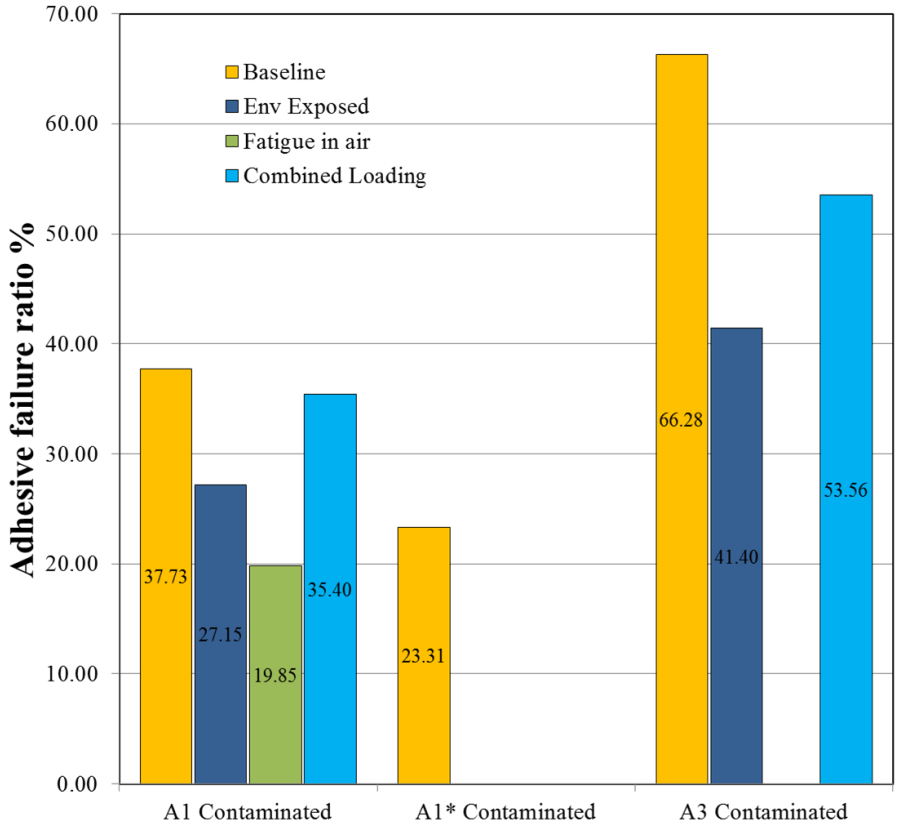
Quantification of Modes of Failure

$$\text{Cohesion ratio} = \frac{\text{cohesively bonded area of specimen}}{\text{total area of specimen}}$$

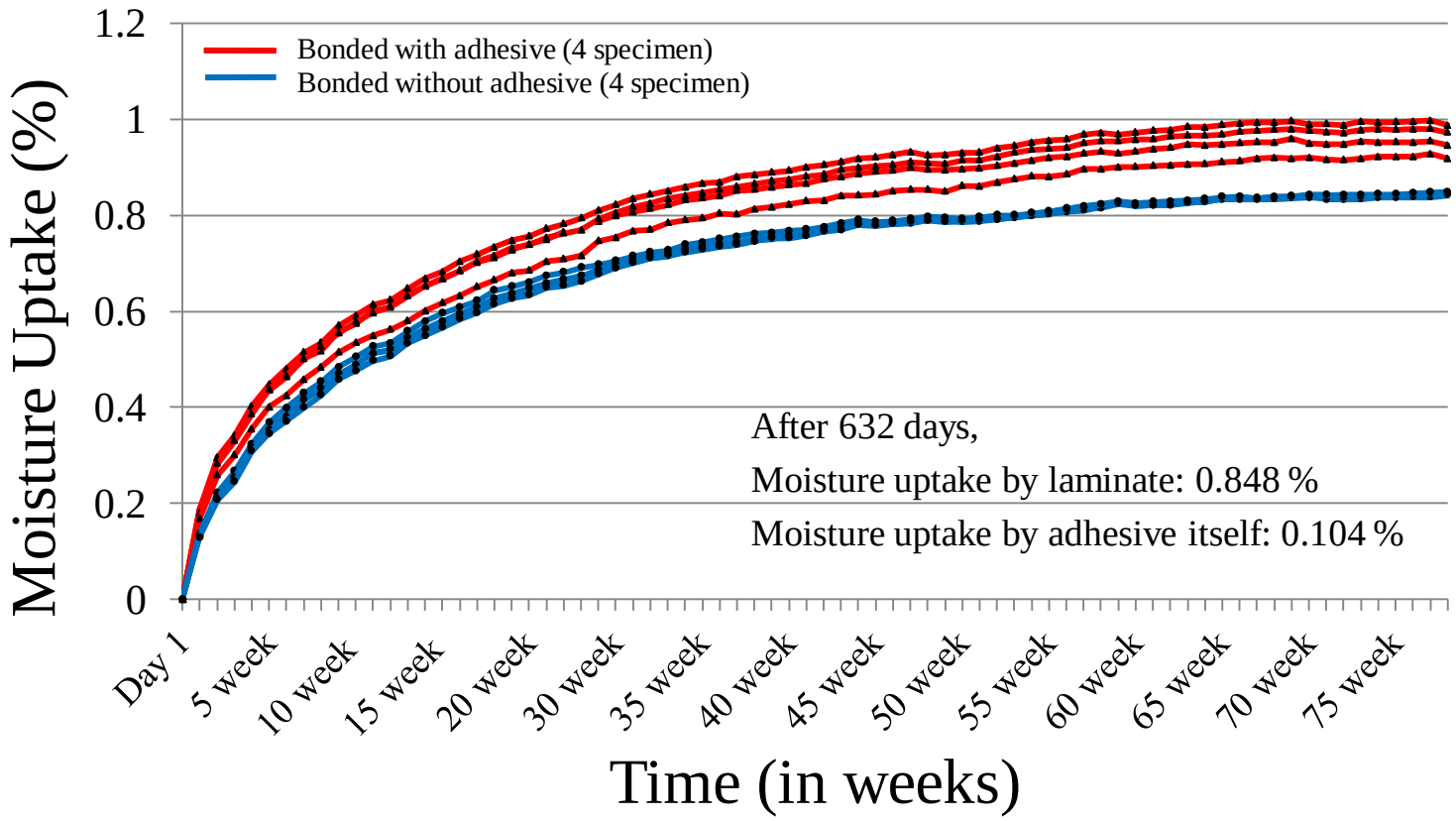
$$\text{Interlaminar/adhesion ratio} = \frac{\text{non-cohesively bonded area of specimen}}{\text{total area of specimen}}$$



Moisture Ingression

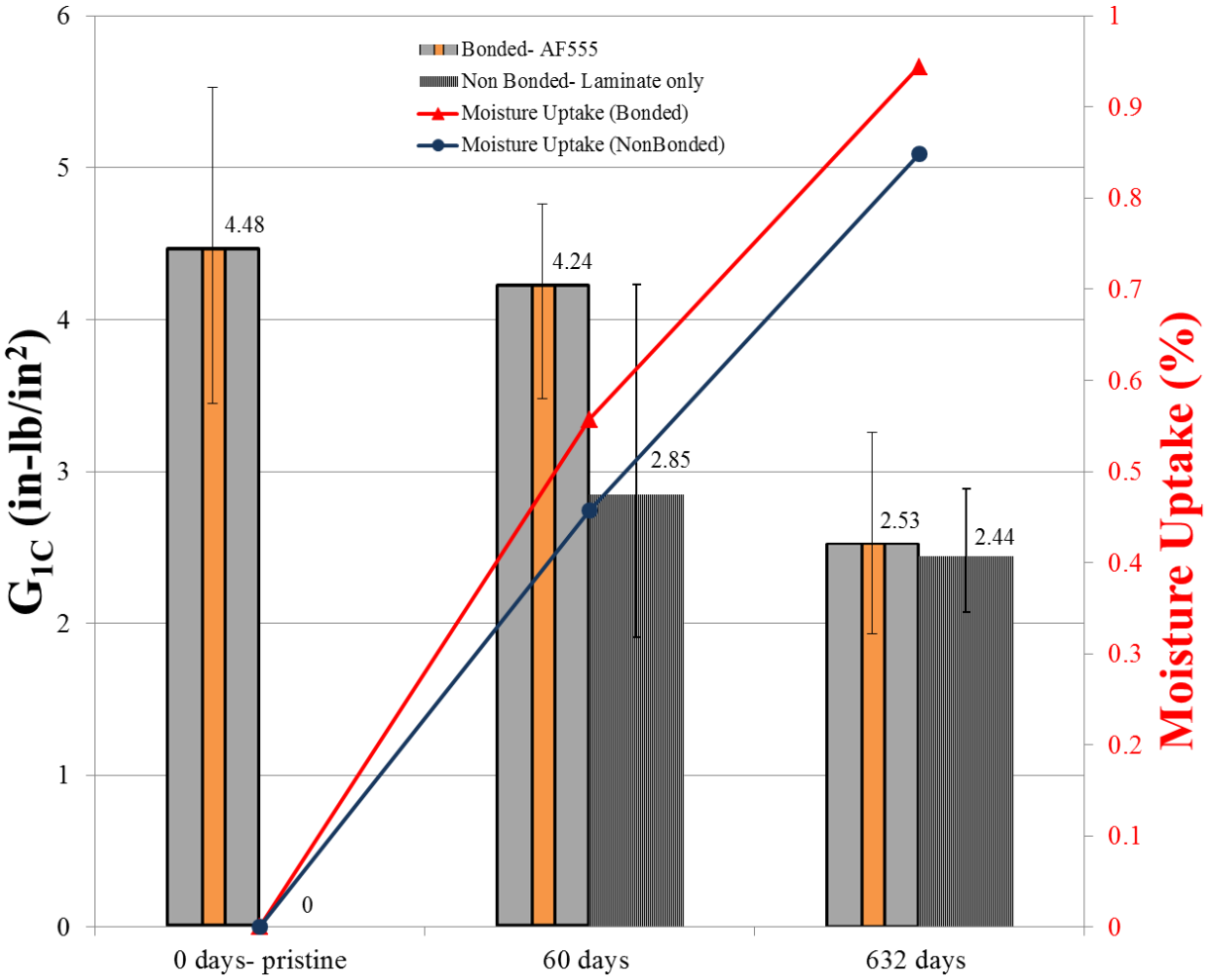
Moisture Uptake Analysis

- Conducted for 78 weeks
- determine saturation limit at 50°C, 95% RH



Fracture Toughness Testing - DCB

Moisture Uptake vs G_{1C}



Summary/Conclusions

- A contamination procedure has been developed to evaluate the effect of contamination on adhesively bonded joints.
- Durability assessment is conducted by conditioning of specimens using a 3-point bending fixture for mechanical fatiguing in air and in environmental chamber.
- Two different stamps were utilized to create a range of contamination levels on the bonded surface (range of weak bonds).
- Adhesion/Cohesion failure mode patterns were observed with the Freekote contamination. The larger area stamp yielding larger adhesion failure areas.
- Line Profile analysis and area analysis of the failure surface are used to quantify the areas of contamination.
- G_{IC} values of baseline testing correlate well with fracture surface area analysis.
- G_{IC} values from conditioned specimens demonstrate trends that suggest the material properties of the laminate and adhesive have been altered. Fracture surface area analysis will require thorough analysis.
- The asymptotic increase in the moisture uptake curve was monitored for 78 weeks, demonstrating the environmental effects on adhesive.

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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.

Objectives

- Evaluate existing content currently contained in the handbook regarding adhesive bonding.
- Obtain feedback from industry (civil transport) regarding their ideas for content that would be valuable and identify gaps that currently exist.
- Compile a list of content from manufactures and researchers and assist in the process of prioritizing new initiatives for the CMH-17 working groups.

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CMH-17 Support

CMH-17 Volumes

Vol. 1: Polymer Matrix Composites – Guidelines for Characterization of Structural Materials
Vol. 2: Polymer Matrix Composites – Material Properties
Vol. 3: Polymer Matrix Composites – Materials Usage, Design and Analysis
Vol. 4: Metal Matrix Composites
Vol. 5: Ceramic Matrix Composites
Vol. 6: Structural Sandwich Composites

Review focuses on existing adhesive bonding content in Vol. 1 and 3

General Scope

Vol. 1: Provide material characterization, test methodology, and data development guidelines to a wide variety of needs for PMC materials.

Vol. 3: Provides guidance in the areas of design, manufacture, and support of PMC structures.

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Areas of Bonding Interest - Military

- Surface prep – use of peel ply, depends on system, need of testing
- Follow up - Grit blasting, sanding, plasma
- Metric for evaluating surface prep quality
- Improved sections on surface prep for adhesive bonding – current sections have a limited write up
- NDI methods – no true solution to detect a weak bond
- Testing to quantify moisture durability

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Areas of Suggested Bonding Content – Transport Manufacturer

- Adhesive properties in the handbook for design guidance
- Test at elevated/wet condition guidance
- Surface preparation inspection/control (Metals and Composites)
- Test matrix for adhesive and prepreg resin compatibility for co-bonding and co-curing
- Test matrix for adhesive and primer compatibility (for metals)
- Tests for aging and durability of bonding
- Adhesive design allowable matrix (certification)
- Adhesive qualification test matrix
- Materials and Process Specification development for adhesives with metals and composites perspective
- Moisture saturation recommended standard and travelers

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Areas of Suggested Bonding Content – Supplier

- Guidance on surface prep – differences between material sets, use of peel plies and the potential effects on bonding
- Information on various methods of prep – grit blasting, sanding and plasma
- Test matrix for adhesive and primer compatibility (for metals)
- Characterization of post surface prep – surface chemistry
- Weak bondline characterization
- More standardization of adhesive properties for design
- Differences with metal and composite laminates in terms of properties for design
- Guidance on prebond moisture – standardize approach
- NDI methods – issues with techniques – approaches still required for determining if bond can maintain load requirements for service life

CMH-17 Support

Summary

- Guidance on surface prep
- Quality control – guidance on bondline thickness, etc.
- Post surface prep evaluation
- Durability evaluation – include guidance on testing
- Bond defect detection and NDI methods
- Adhesive properties for design
- Metal bonds

Path Forward

- Continue to work with interested parties to obtain a more complete list of needed areas.
- Assist in organizing and prioritizing the areas for inclusion into the handbook.

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Questions ?

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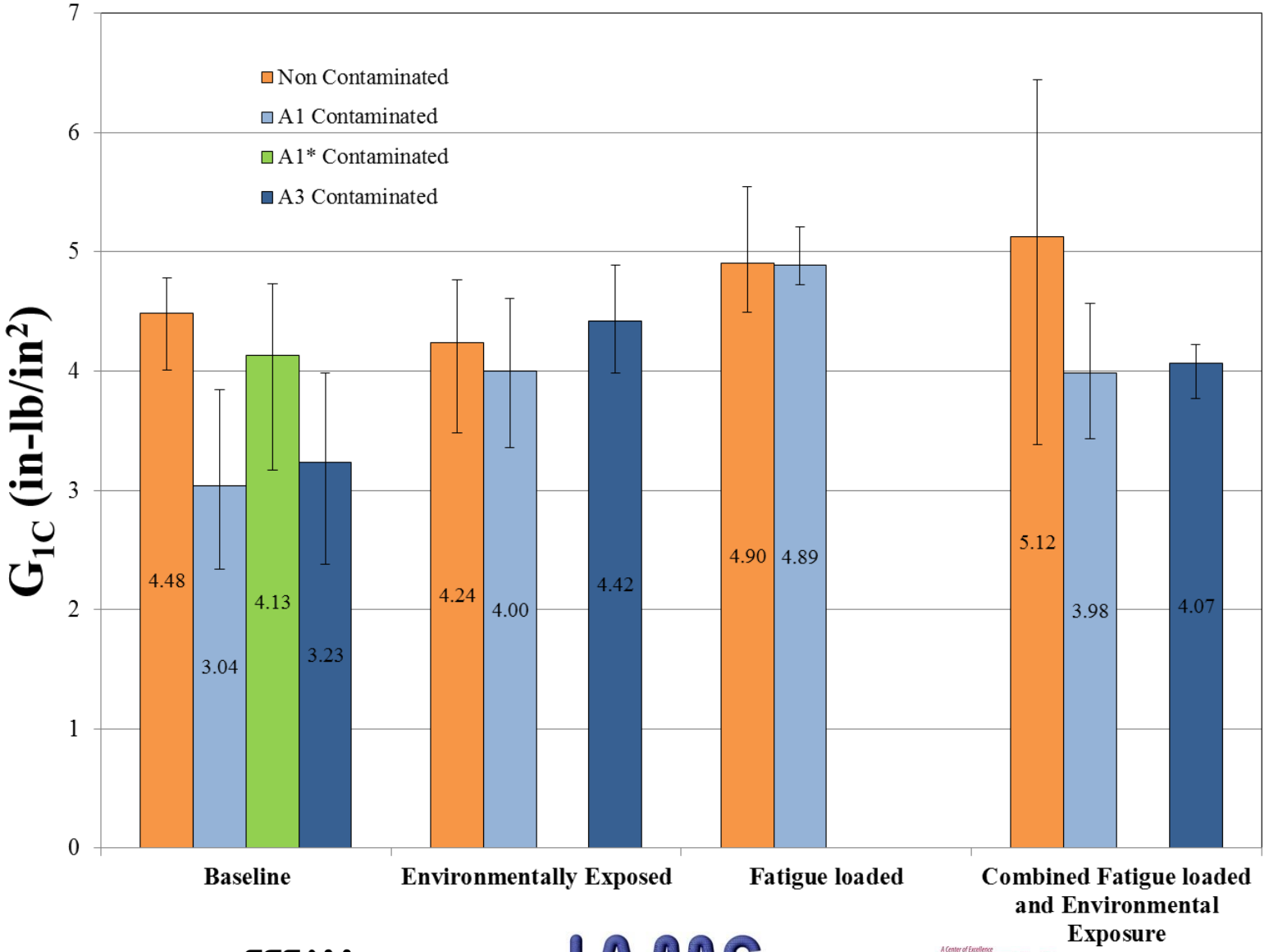
Fracture Toughness Chart- All specimens



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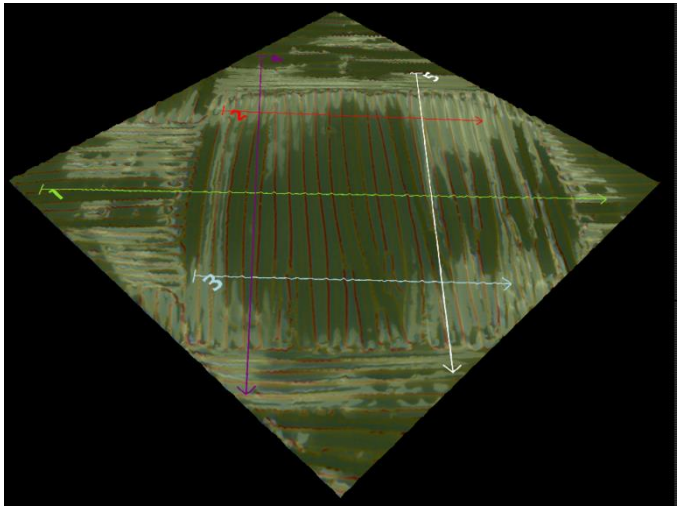
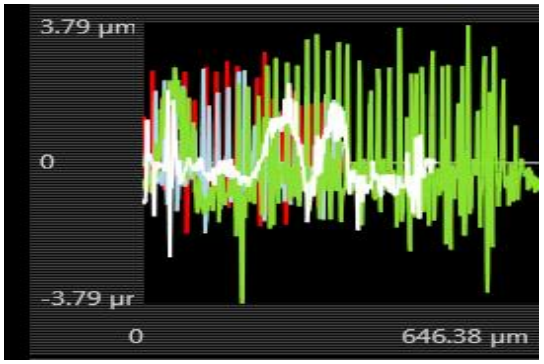
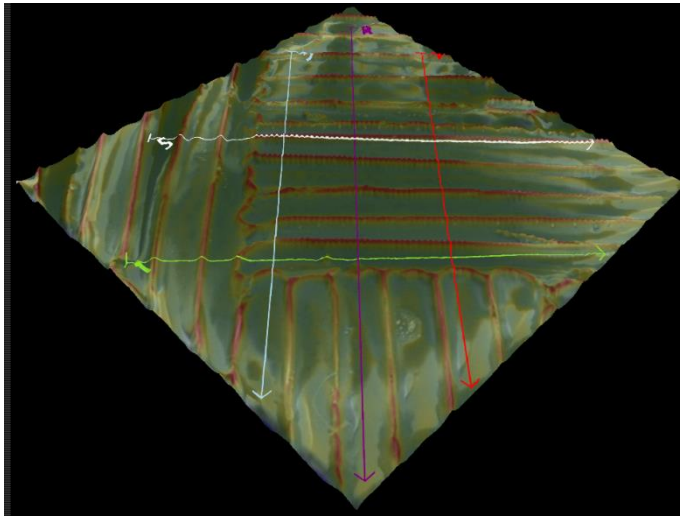
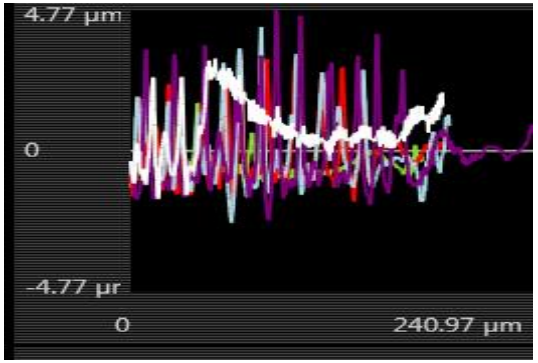
Surface Roughness





	Rz (µm)
1)	5.83 µm ± 0.9
2)	4.38 µm ± 1.1
3)	4.66 µm ± 0.9
4)	2.88 µm ± 1.2
5)	2.58 µm ± 1.4





	Rz (µm)
1)	3.17 µm ± 0.8
2)	4.46 µm ± 1.0
3)	4.62 µm ± 0.3
4)	5.08 µm ± 0.1
5)	3.02 µm ± 0.9

Average - 4.19 µm ± 1.1

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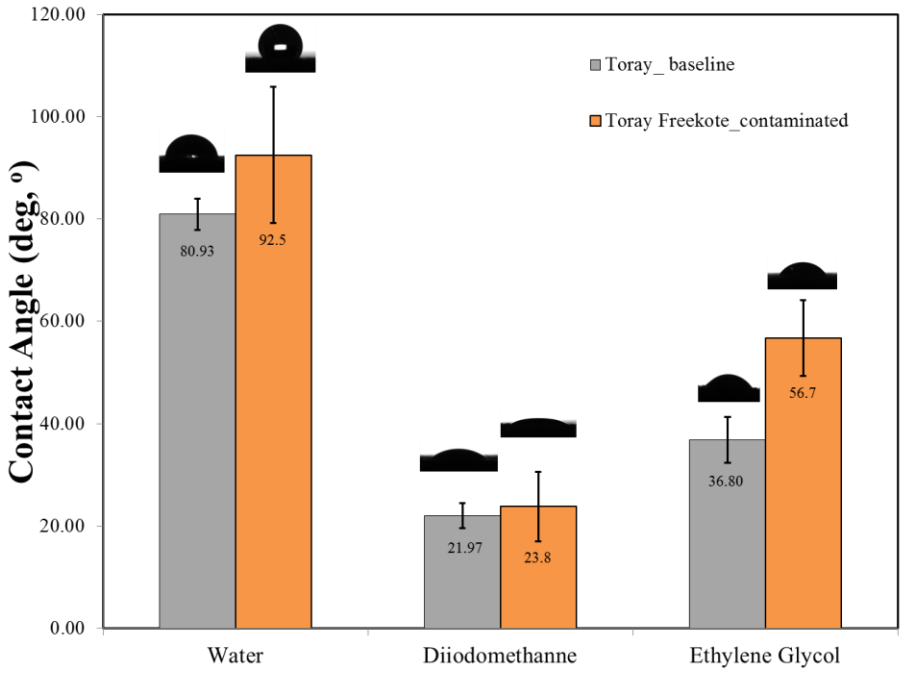
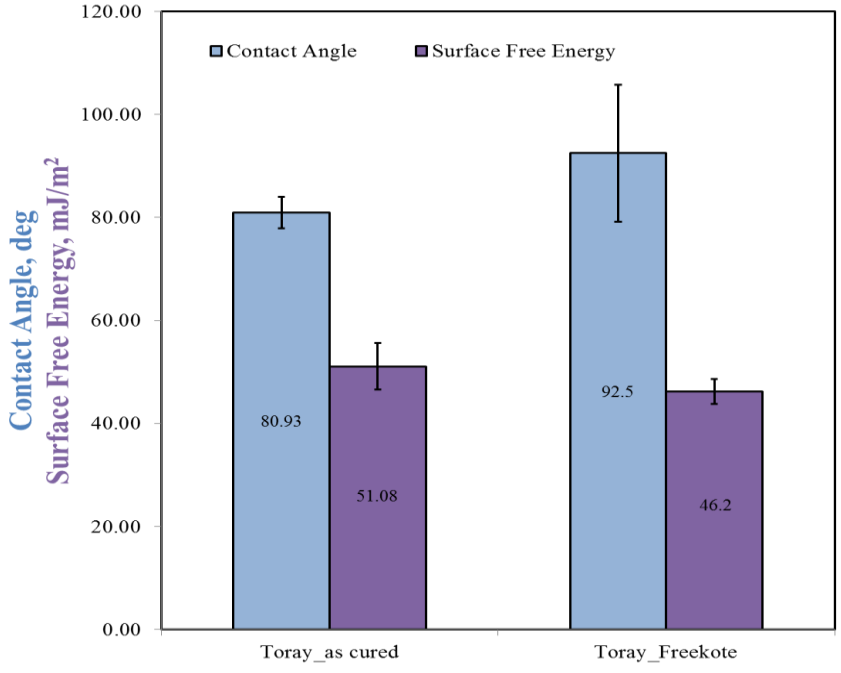
Surface Characterization Methods

Contact angle measurements

3 Fluids - DI water, diiodomethane and ethylene glycol



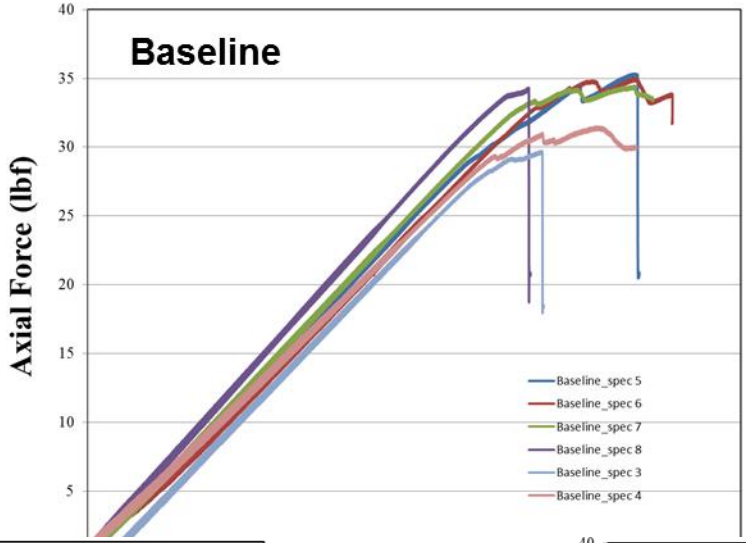


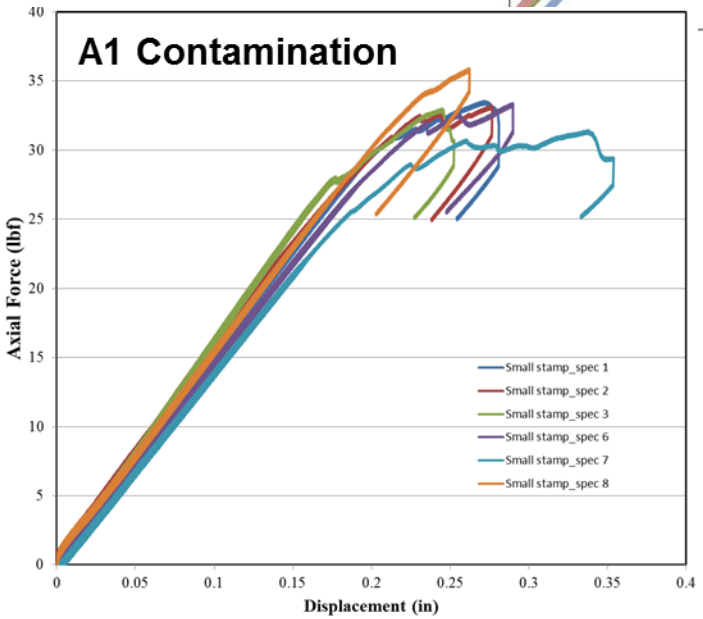
Contaminated specimens

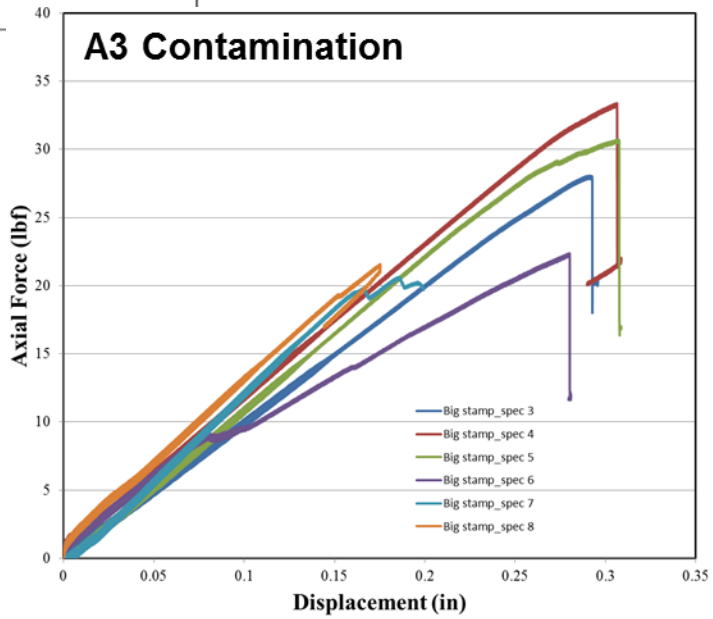
Higher contact angles

Lower Surface free energies = Poor adhesiveness → Less G_{1C}'s

Load-Displacement Curves







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