



Effect of Surface Contamination on Composite Bond Integrity and Durability

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Composite Bond Integrity/Long-Term Durability of Composite Bonds

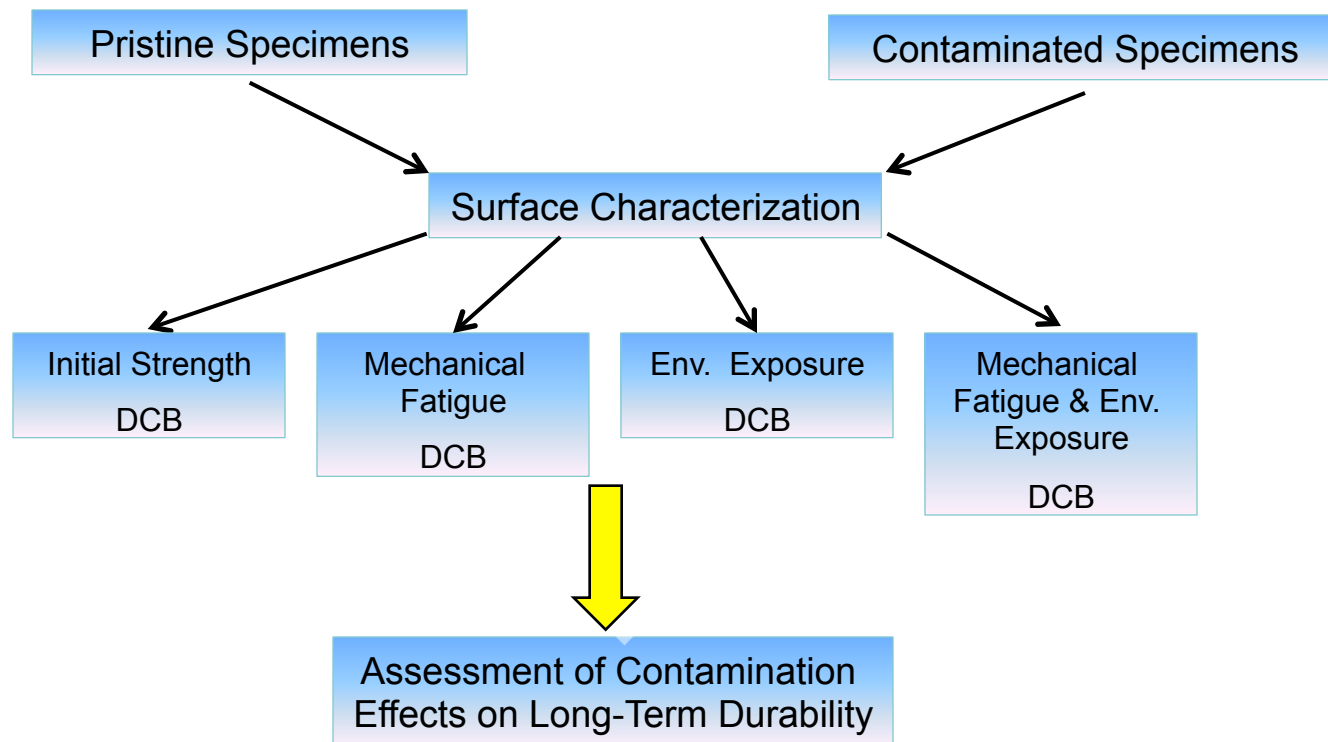
- **Motivation and Key Issues**

- 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

Durability Assessment Procedure



Bonding System Materials

Selection of materials and curing procedure for specimens: unidirectional carbon-epoxy system, film adhesive, secondary curing for bonding.

Material used on previous results:

- DA 411U 150 Uni-Carbon Epoxy Prepreg (350 F cure) from APCM
- 3M AF 163-2 adhesive film (9.5x2mills, 250 F cure)
- Peel plies : Polyester and Nylon from Fibreglast, Precision Fabric peel ply 60001
- 3M AF555U adhesive film (2.5X4 mills, 350 cure)

Current materials:

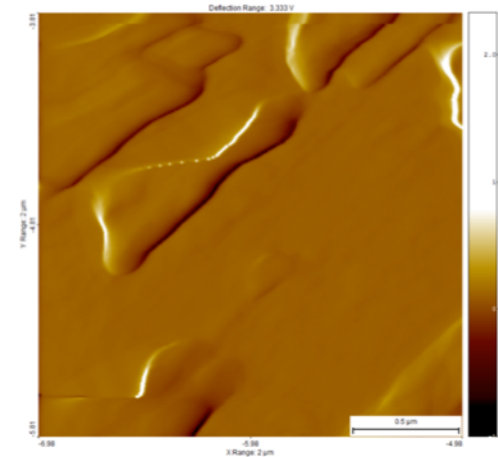
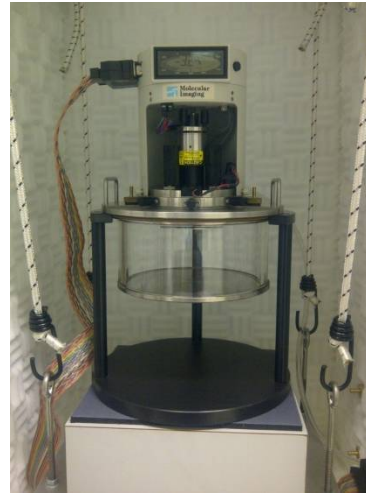
- Toray T800 unidirectional tape
- 3M AF555M adhesive film (8.5X2 mills, 350 cure)
- Precision Fabric peel ply 60001

Specimen conditioning:

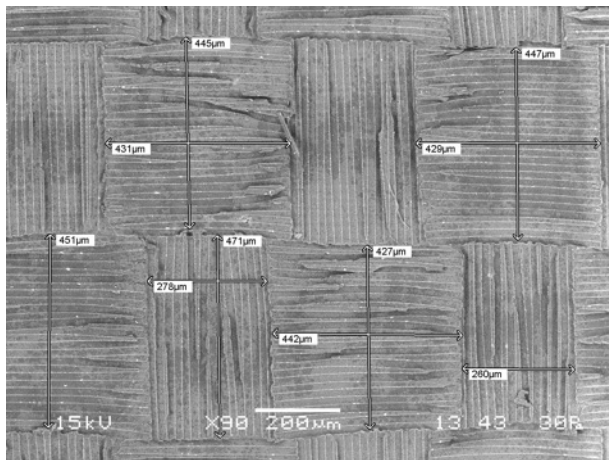
- Environmental Chamber 50°C, 95 % RH, for two months
- Specimens were fatigued for 2.8 million cycles

Surface Characterization

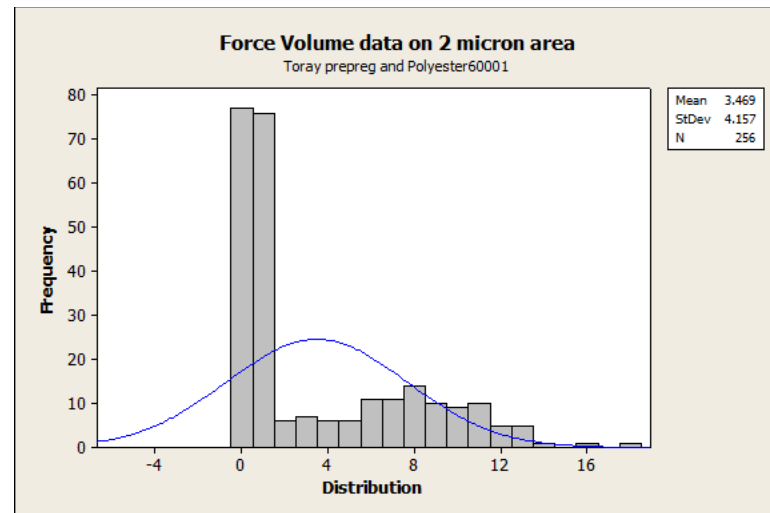
- FIU and UM has conducted research utilizing Atomic Force Microscopy and epoxy-modified probes tip to characterize surfaces prior to bonding.
- AFM can record the attraction/repulsion forces between the AFM probe and the surface.
- AFM data is used to generate topography and force volume measurements to quantify changes in adhesion forces.



Deflection image_2μ



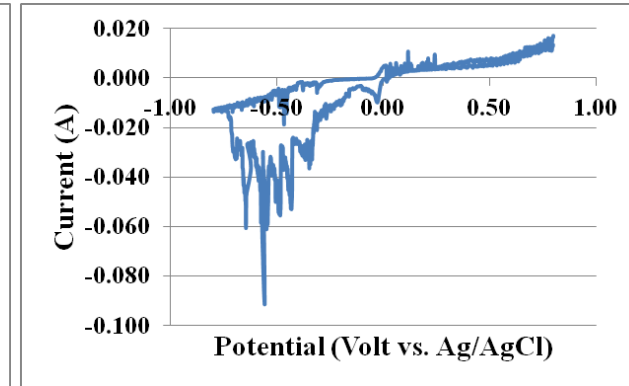
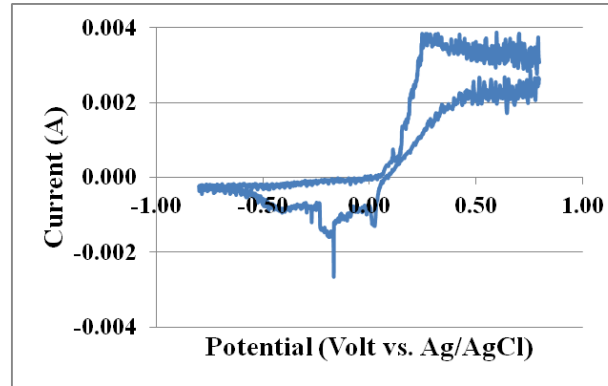
SEM image



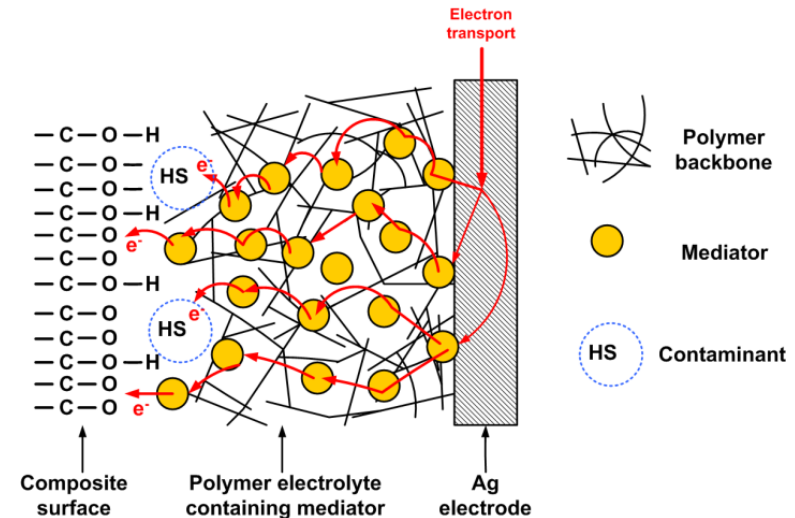
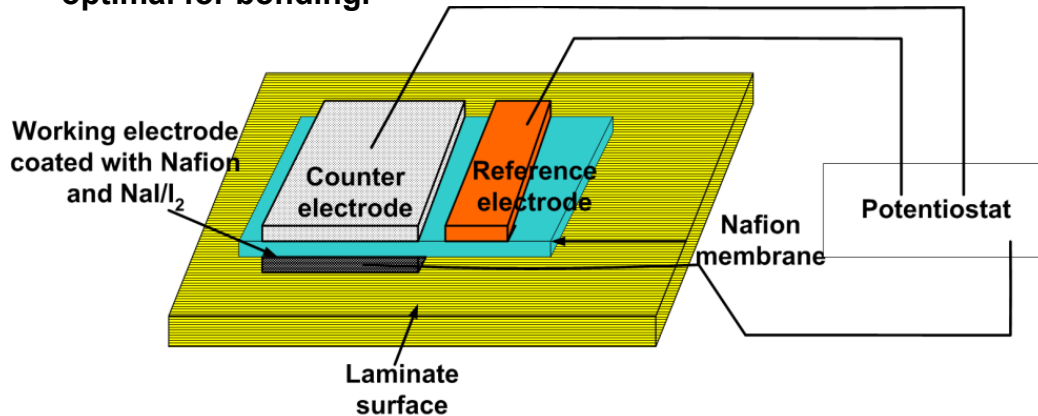
Surface Characterization

A all solid-state electrochemical sensor is being investigated to detect surface contamination.

The signatures obtained from the sensor have the potential to be associated with the presence of contamination on the surface. The active surface consists of dense dangling bonds (unsatisfied bonds) that tend to form a stronger interface between adherend and adhesive. The negative peak can correspond to an oxidizing surface, which is optimal for bonding.



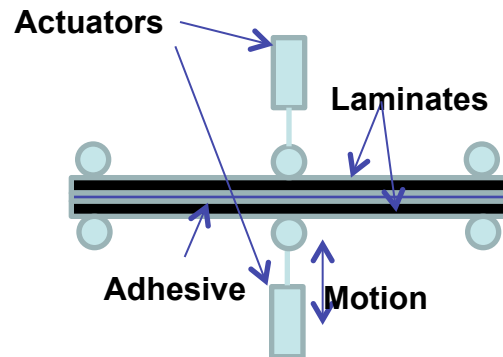
CV measurements for specimens manufactured with 60001 Poly peel ply.
 As tooled and sanded with 60 grid sandpaper



Fatigue Loading Procedure

Specimens are first mechanically fatigued and/or exposed to an accelerated aging environment. They are then tested for degradation in fracture toughness.

1- Mechanical loading



2- Durability evaluation using double cantilever beam test

Loading at constant displacement



Advantages

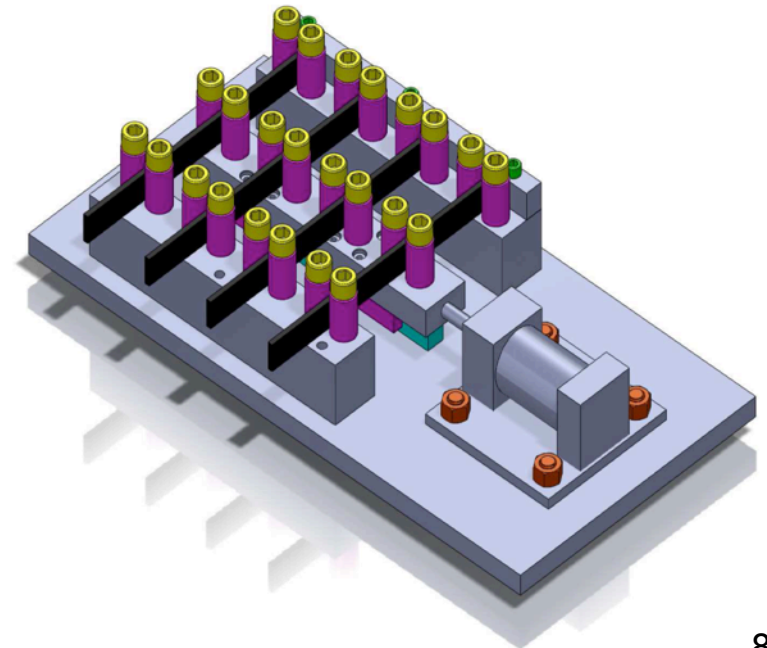
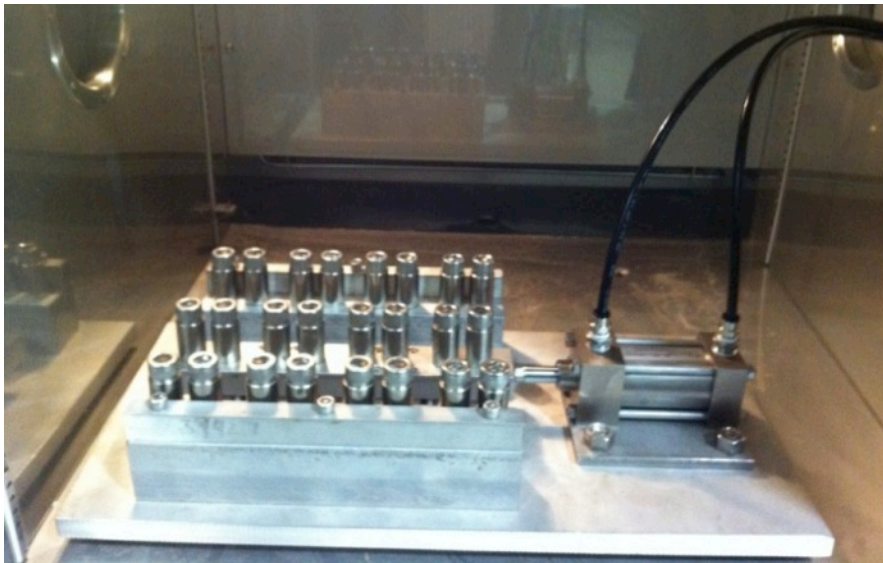
- Apply uniform shear stress at bondline
- Simple to set up – potential to enclose in an environmental chamber
- Can use DCB (ASTM 5528) or wedge specimens (ASTM 3762)

Disadvantages

- Specimen geometry needs to be adjusted to limit fatigue in adherend/adhesive
- Need to consider surface stress effects resulting from contact points

Fatigue Fixture

- Manufactured using stainless steel materials
- Center section slides on a ball bearing carriage
- Designed to load up to four 9 in specimens (later changed to 11.5 in) with a deflection up of 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



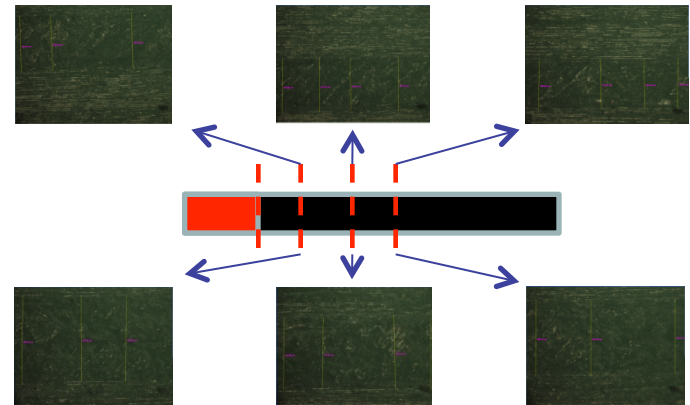
Analysis of Specimens

Recorded parameters:

- Weight measurement
- Bondline thickness measurements
- Fracture toughness measurements
- Mode of failure observations

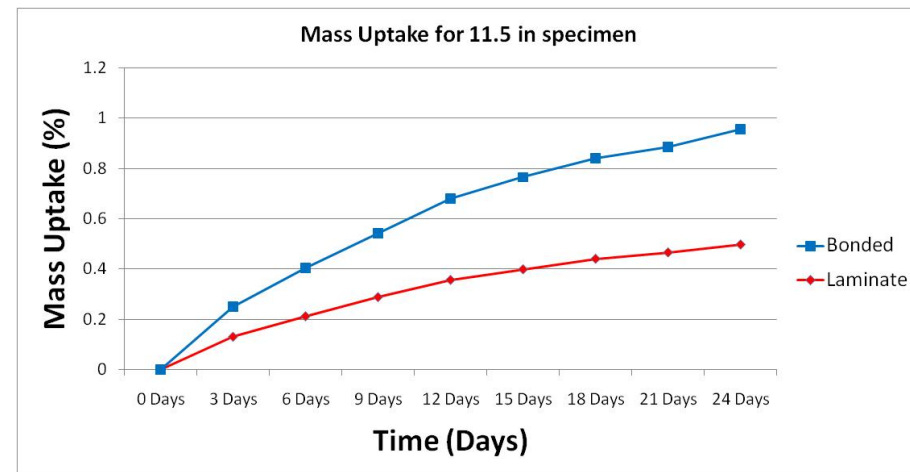
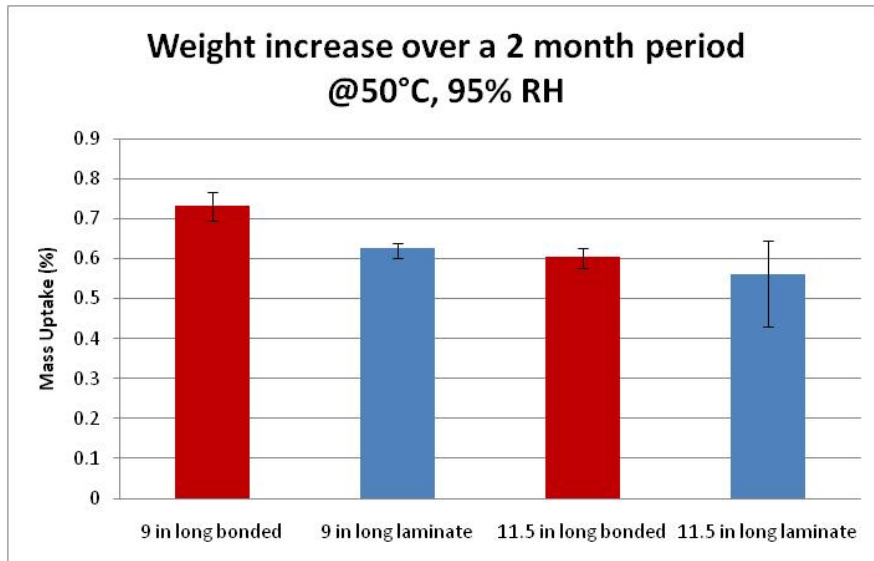
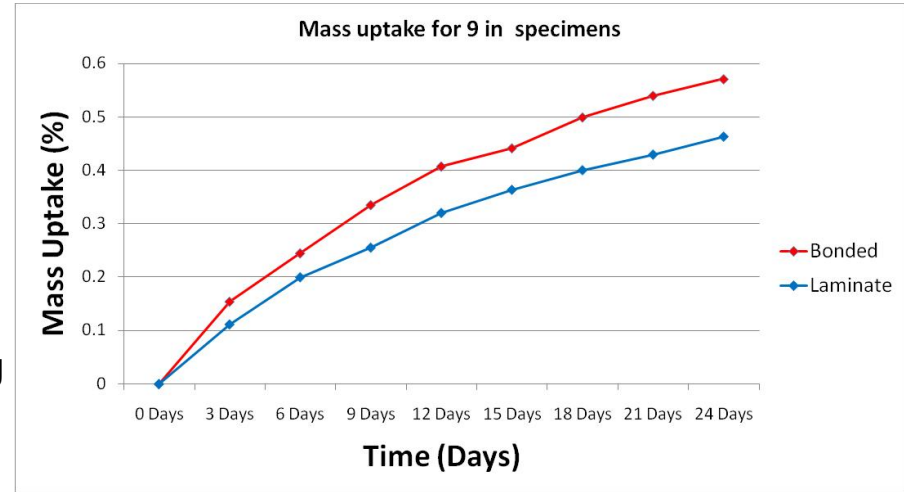
Specimens evaluated:

- 12 Bonded DCB specimens 9 in long
 - 4 Base line specimens
 - 4 Environmentally conditioned
 - 4 Fatigued loaded (failed)
- 8 Bonded DCB specimens 11.5 in long
 - 4 Environmentally conditioned
 - 4 Fatigued loaded
- 8 “laminates only” DCB specimens : 9 and 11.5 in long all environmentally conditioned
- 4 specimens (2 bonded, 2 “laminates only”) progressive weight measurements (24 days).



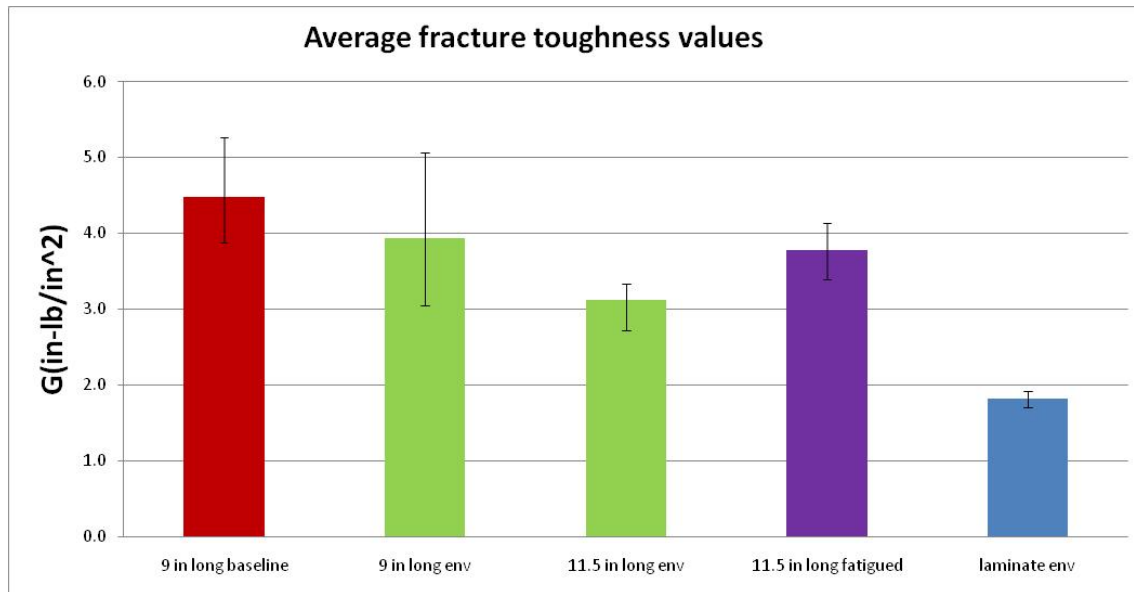
Weight Measurements

- Weight measurements were used to determine the water sorption of the laminate and adhesive
- “Laminate only” and bonded DCB specimens were environmentally conditioned to estimate the water sorption of the laminate vs. the water sorption of the adhesive
- Flattening of the weight curve can assist in determining the time required for exposure



Fracture Toughness

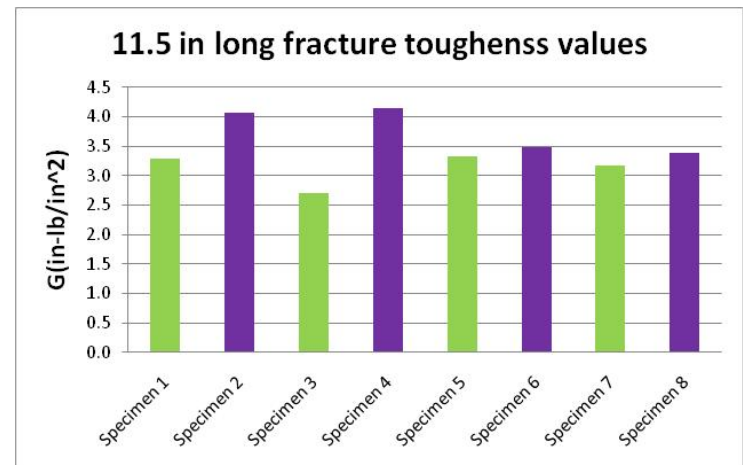
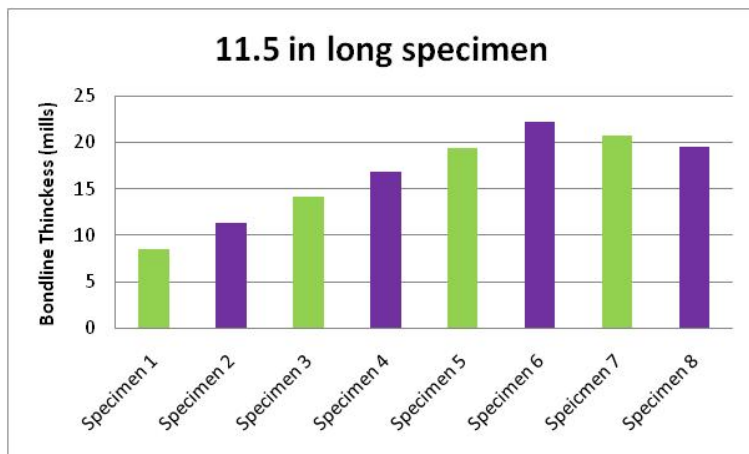
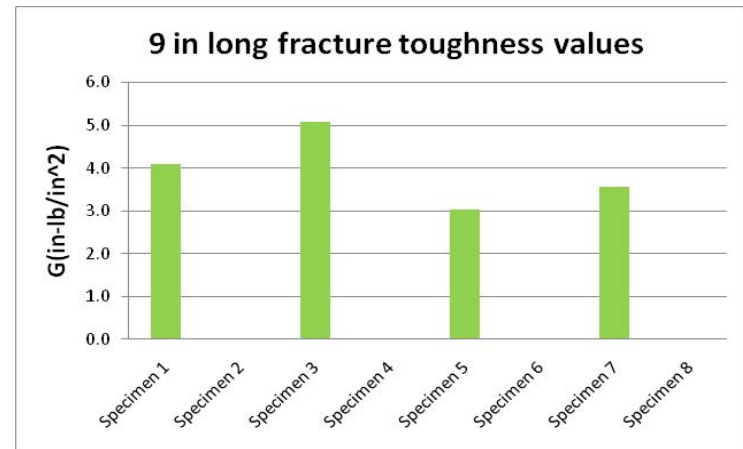
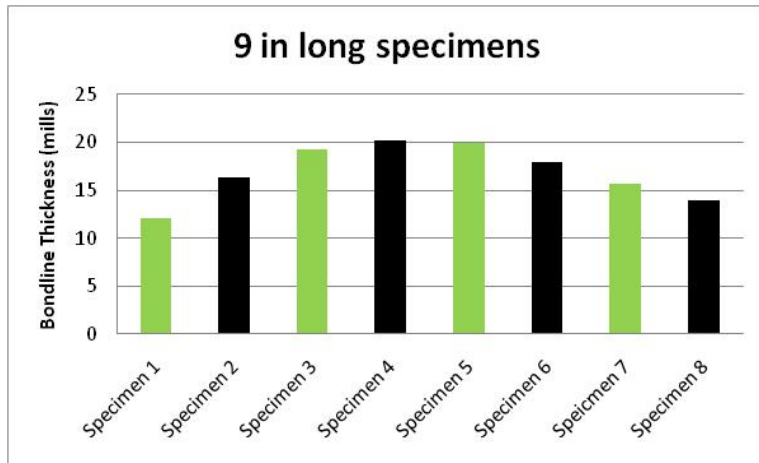
- Specimens environmentally conditioned exhibit the larger degree of degradation
- Specimens fatigued loaded exhibited some degree of degradation



	Average	Max	Min
	(in-lb/in ²)	(in-lb/in ²)	(in-lb/in ²)
9 in baseline	4.48	5.26	3.88
9 in env. conditioned	3.94	5.07	3.04
11.5 in env. Conditioned	3.12	3.34	2.71
11.5 in fatigued	3.77	4.14	3.39
laminate only	1.81	1.92	1.70

- Baseline
- Fatigue loaded
- Environmentally exposed
- Laminate only env.

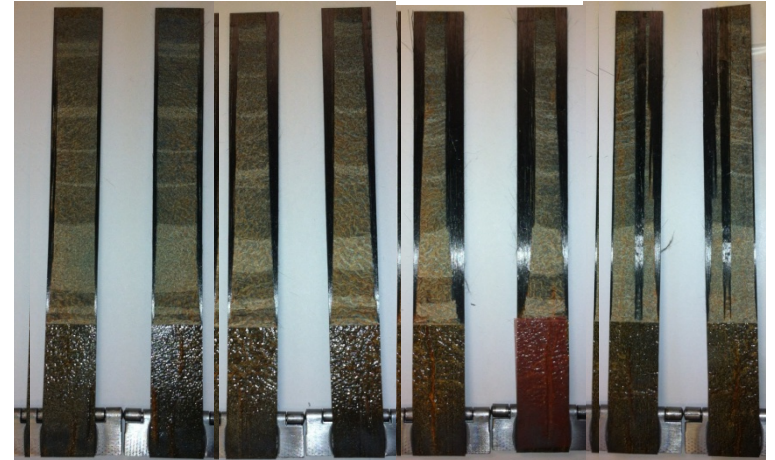
Bondline Thickness Measurements



Mode of Failure Observations



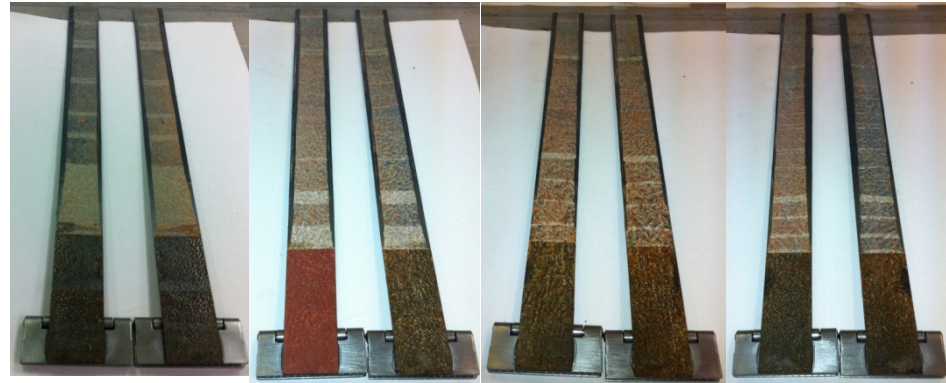
9 in long baseline specimens



9 in long environmentally conditioned specimens



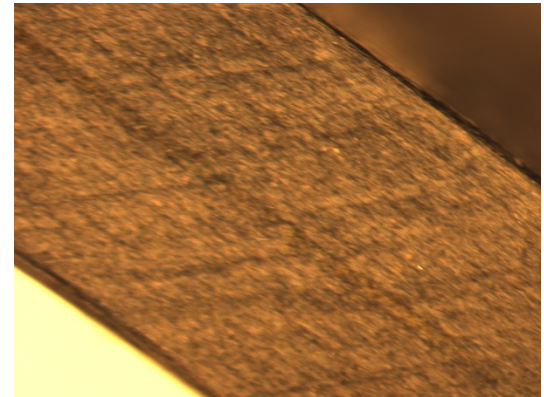
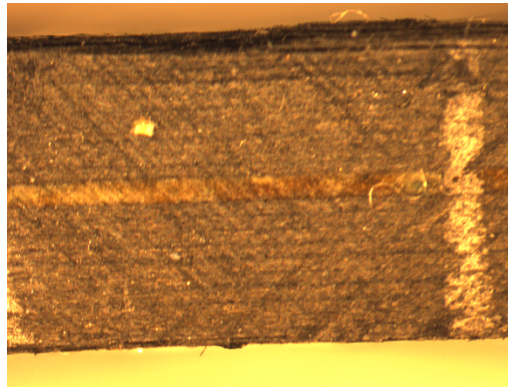
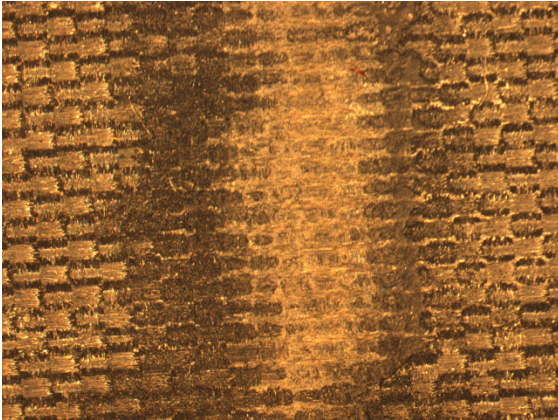
11.5 in long environmentally conditioned specimens



11.5 in long fatigue loaded specimens

Fatigue Fixture Observations

- Specimens were subjected to 2.8 million cycles
- Minor damage on outermost ply was observed due to roller contact
- No crack growth during fatigue loading
- No interlaminar failure resulting from fatigue loading occurred during DCB testing



Typical optical microscope images of fatigues loaded specimens (16 X magnification)

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Future work:

- Subject specimens to environmental aging and fatigue loading (progressive testing).
- Investigate and apply a procedure to quantify surface contamination prior to bonding.
- Fabricate, test and analyze data with less than ideal bonding conditions specimens.
- Implement means for measuring surface contamination/energy (AFM, ECM, water contact angle, FTIR, electrochemical sensor, etc).
- Fabricate fatigue fixture that will allow to load 8 specimens simultaneously.
- Increase the number of specimens for each case.

Benefit to Aviation:

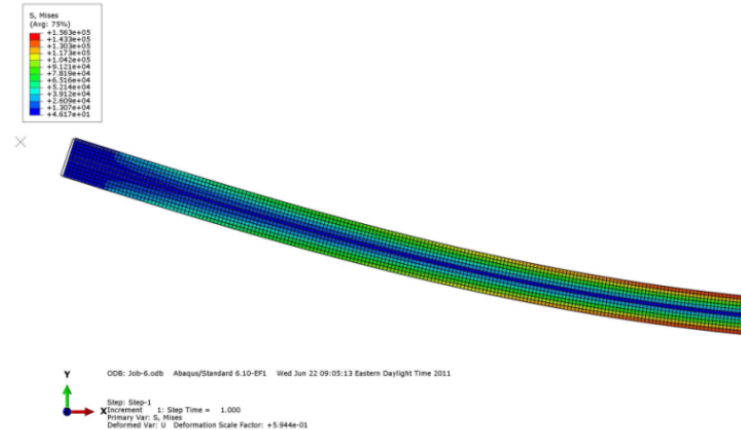
- Better understanding of durability assessment for adhesively bonded composite joints.
- Assisting in the development of bonding quality assurance procedures.

Composite Bond Integrity/Long-Term Durability of Composite Bonds

Questions?

Specimen Design

Max Stress (psi)	Avg. Life (Cycles)
4500	1.58×10^4
4000	5.28×10^4
3500	4.75×10^5
3000	2.67×10^6
2200	$1.03 \times 10^7 +$ (No failure)

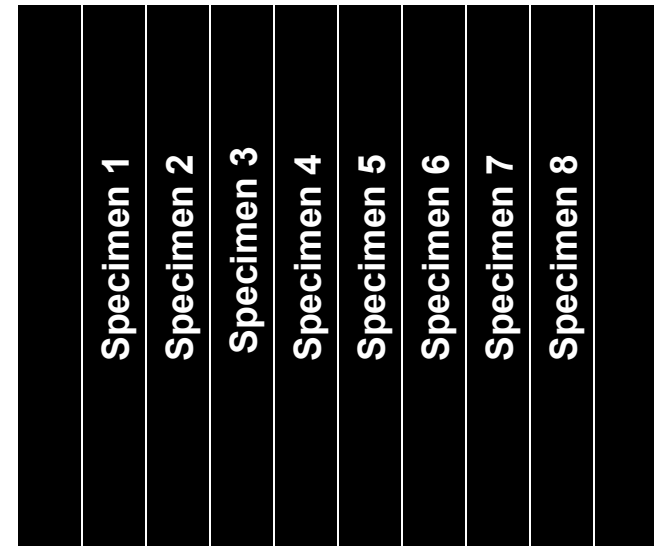
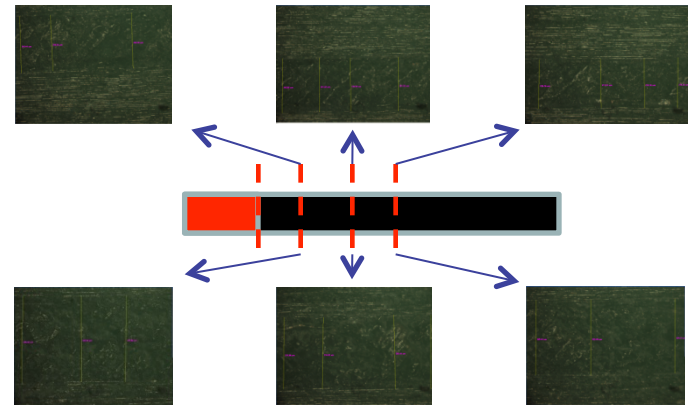
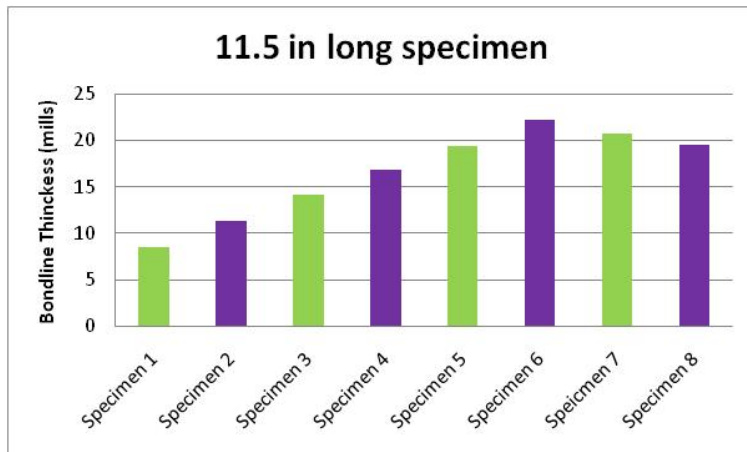
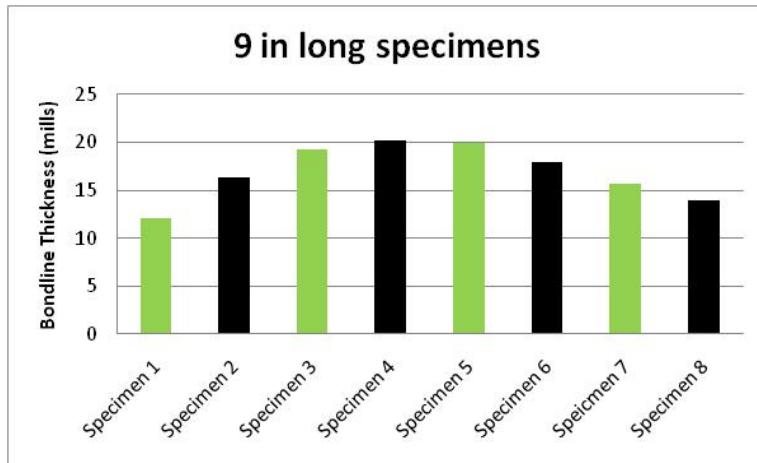


# Plies	Thickness (inches)	Force required by piston (lb)	Stress at Surface (ksi)
16	0.120	240	225
18	0.135	270	200
20	0.150	300	180
22	0.165	330	164
24	0.180	360	150

Selected laminate configuration:

- Specimen dimensions: 11.5 in long x 1 in wide
- 20 ply unidirectional laminate (**0.15 thick** + adhesive)

Bondline thickness measurements



Surface Stresses

- Specimens were tested to deflections of 0.25 in, 0.5 in, and 0.75 in
- 80% of tested specimens to 0.75 in of deflection fractured
- Toray800 material ultimate strength is 212×10^3 psi > 1 in specimen deflection
- Specimens have an effective length of 8 in
- Surface effect of 1 inch diameter rollers lowered Su to 129×10^3 psi -> 0.6 in deflection
- Conditioned specimens will be deflected to 0.5 inches (2,300 psi of transverse shear).

