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Effect of Contamination on Composite Bond Integrity and Durability

2012 Technical Review

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Effect of 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

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Effect of Contamination on Composite Bond Integrity and Durability

- **Principal Investigators & Researchers**
  - Dwayne McDaniel, Xiangyang Zhou, Tomas Pribanic
- **Students**
  - Vishal Musaramthota, Juanjuan Zhou, Sirui Cai
- **FAA Technical Monitor**
  - David Westlund
- **Industry Participation**
  - Boeing, Exponent

Literature Review

“A recent survey of organizations that use adhesive bonding indicated that 77 percent of designers use lap-shear test results to establish design allowable.” (Davis & Tomblin, ‘07)

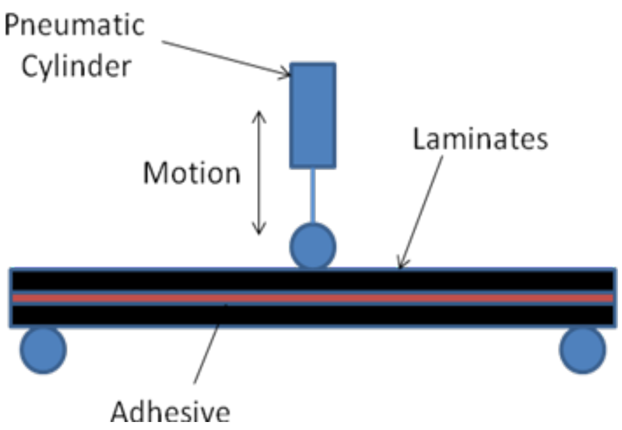
“The most important thing to note about durability testing of adhesively bonded joints is that the *MODE* of failure is more important than the failing load.” (Hart-Smith, ’99)

Specimen conditioning using mechanical loading in harsh environments

- Lloyd Smith – utilized customized load frames to apply static and cyclic loads on various types of adhesively bonded composite specimens. The frames could be placed in hot/wet baths for simultaneous loading.
    - Used thick wide lap-shear coupons with constant loads in a hot/wet environment – effects of moisture and peel ply.
    - Used DCB coupons with constant and cyclic loading in a hot/wet environment – effects of surface treatment.
- Briskham and Smith – used a string of lap shear joints in a hot/wet bath under cyclic loading.
- Ashcroft, et al. – used double lap joints with composite and metal adherends to study environmental effects on fatigue behavior.

Mechanical Conditioning

Modified Three Point Bending Test



Advantages

- Apply uniform shear stress at bondline
- Simple to set up – potential to include in an environmental chamber
- Can use DCB or wedge specimens

Disadvantages

- Specimen geometry made need to be altered to limit fatigue in adherend

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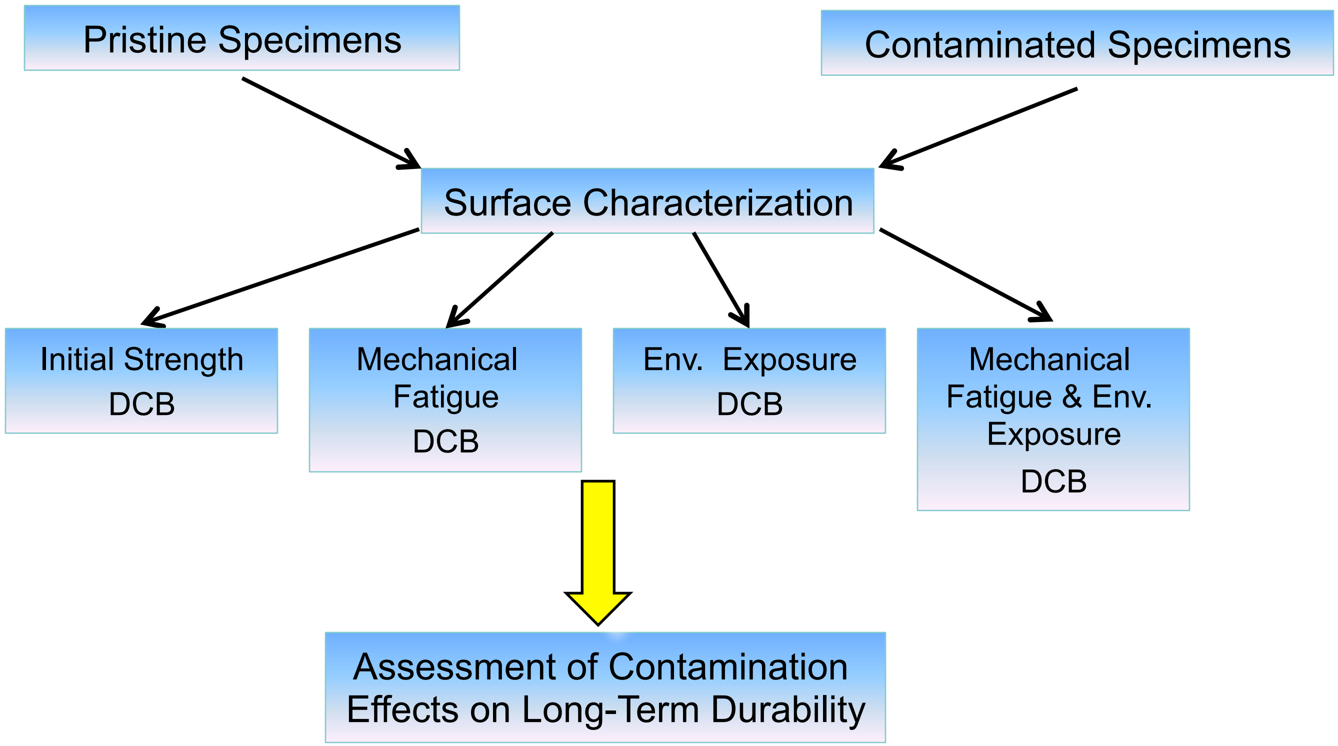
Durability Assessment Procedure



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Materials and Preparation

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

Current materials:

- DA 411U 150 Uni-Carbon Epoxy Prepreg System (350 F cure) from APCM
- AF 163-2 Scotch Weld Structural Adhesive Film (250 F cure) from 3M
- Polyester Release peel ply from Fibreglast
- Nylon Release peel ply from Fibreglast
- Precision Fabric peel ply 60001

Future materials will include:

- 3M AF555 adhesive film
- Toray T800 unidirectional tape

Surface Preparation:

- As tooled
- Hand sanded with 60 grit Al_2O_3 sand paper

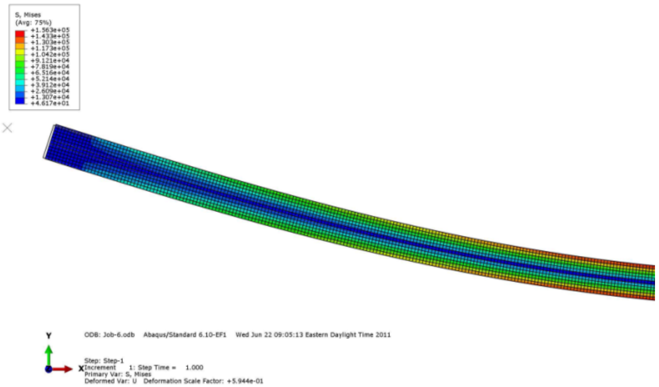
Specimen Conditioning:

- Environmental Chamber - 50°C, 95 % RH, for three weeks

Specimen Design

Known fatigue properties AF 163-2 (Metal to Metal for Double Lab Strap)

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	1.03×10^7 + (No failure)



Values of some of the evaluated laminate configurations

# Plies	Stress at surface (psi)	Force require by piston (lb)	Displacement (in)	70% displacement (in)
22	136.36	264	1.53	1.07
24	125.00	288	1.28	0.90
26	115.38	312	1.09	0.76
28	107.14	336	0.94	0.66
30	100.00	360	0.82	0.57

Selected laminate configuration:

- Specimen dimensions: 9 in long x 1 in wide
- 24 ply unidirectional laminate (0.14 in thick)
- 0.019 in bond line thickness

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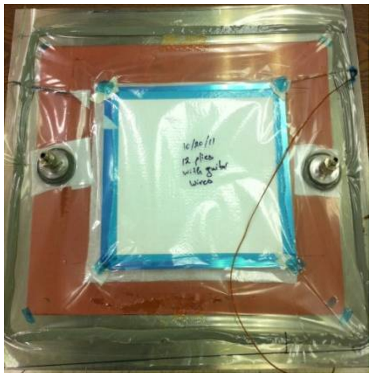
Manufacturing Procedure

Fabrication of laminates



Cure cycle @350F

Bonding of laminates

Secondary cure @250F

Variation of ASTM D5528

Surface characterization, testing, and data analysis

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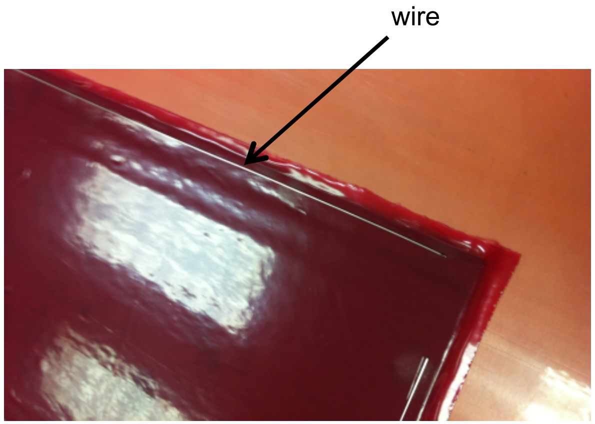
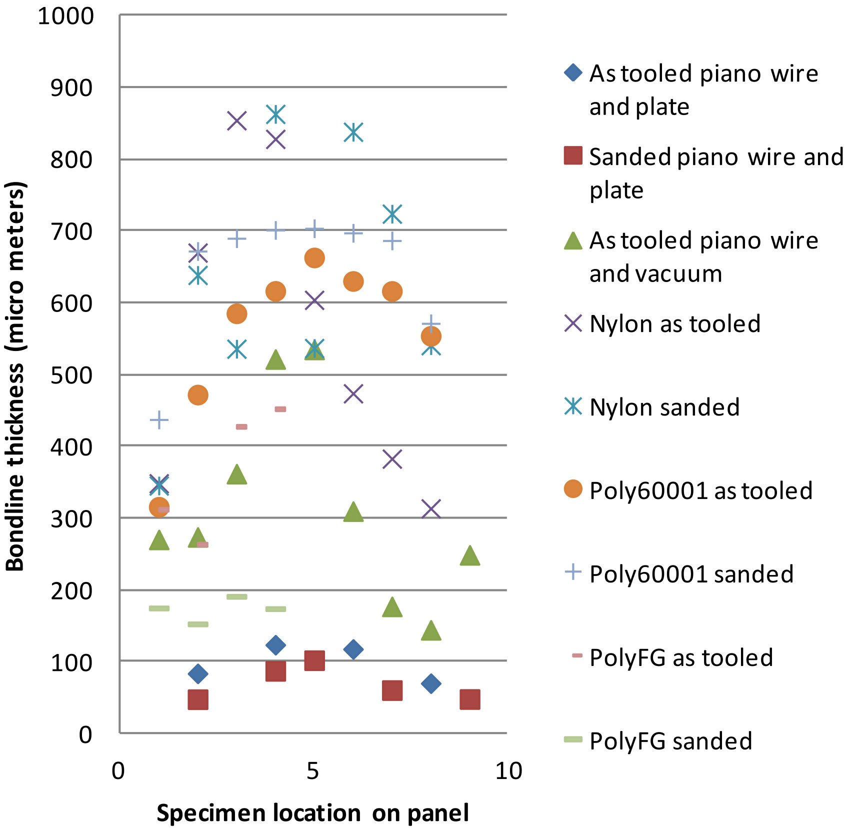
Bondline Thickness Control

- Two high finish plates for tooling
- Two layer of adhesive film (per 3M recommendation)
- Wire having a diameter of desired bondline thickness around perimeter
- Tested vacuum pressure over entire curing cycle





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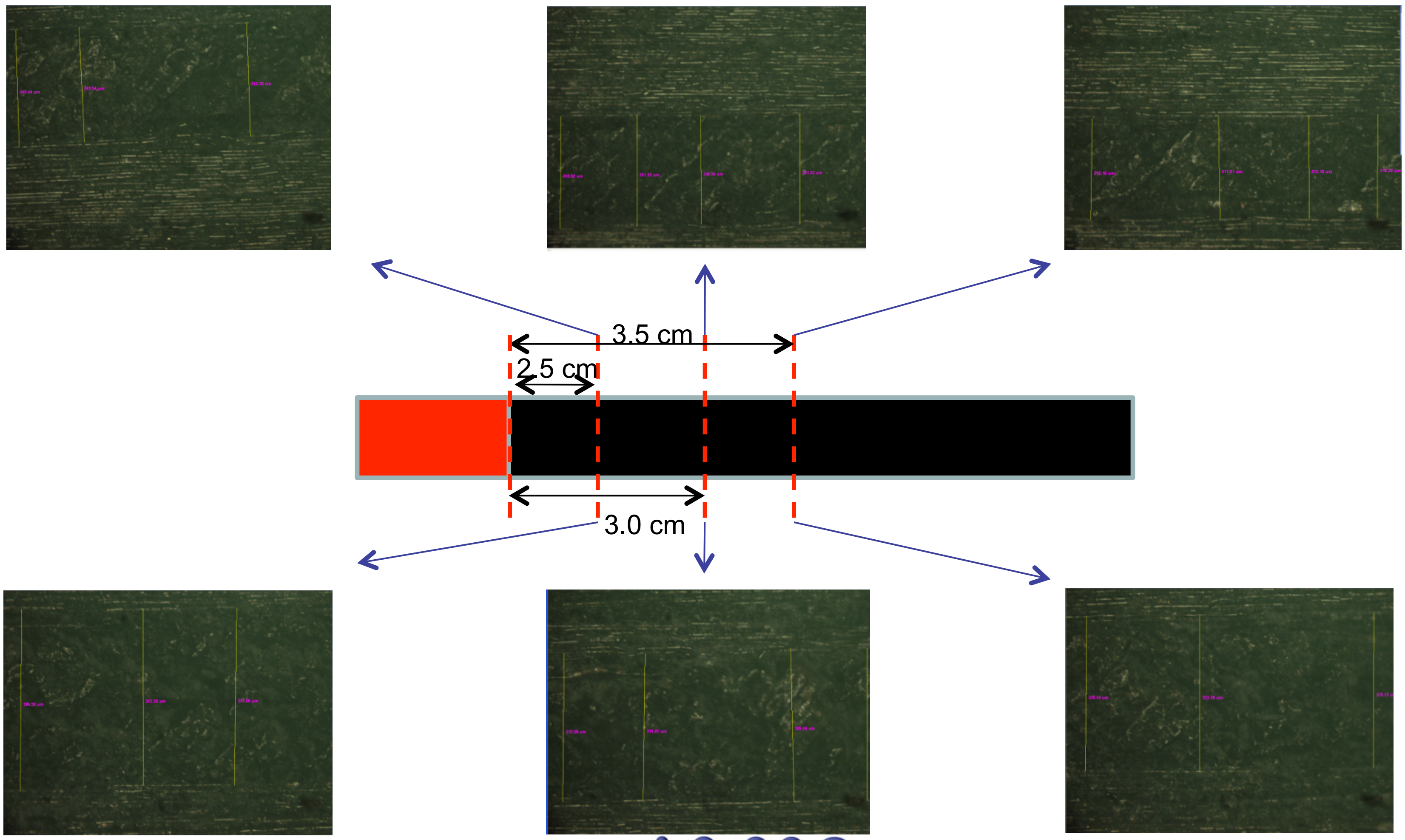
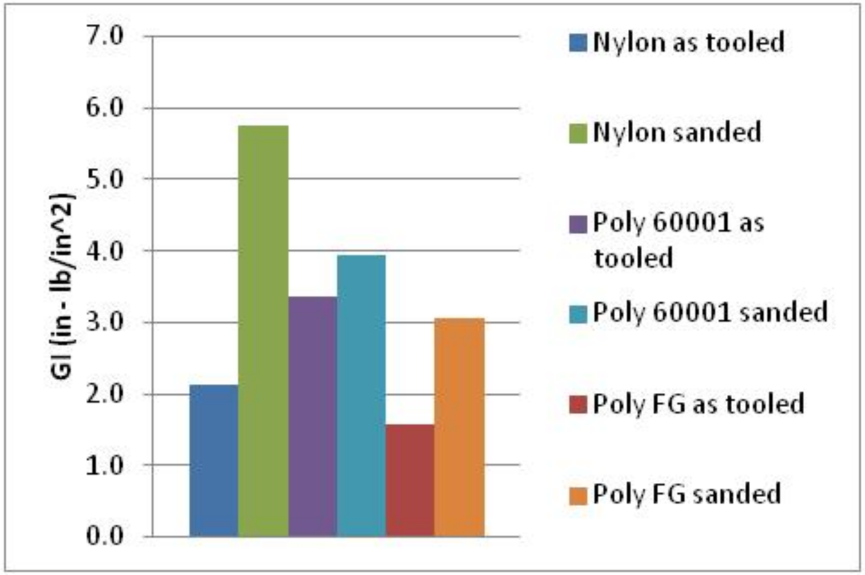
Bondline Thickness Control

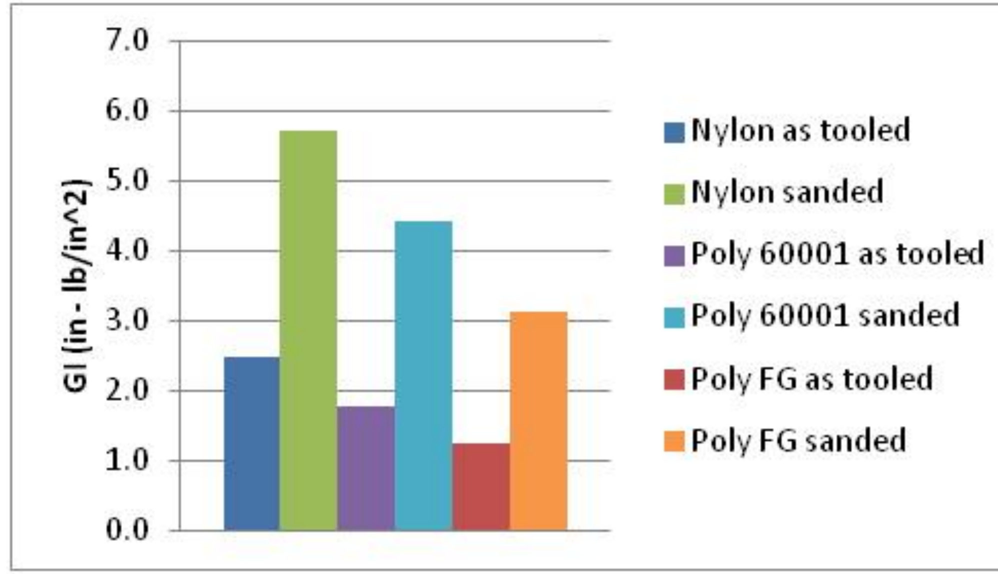
Optical microscopy measurements were taken at three locations (2.5, 3, 3.5 cm)



Fracture Toughness Results



Baseline specimens



Environmentally aged specimens

- FG polyester and nylon, PF60001 polyester
- As tooled and sanded with 60 grit Al_2O_3 sandpaper
- Aged for three weeks at 95% RH and 50°C
- Sanded nylon surface had highest G_{1C}
- Sanded surfaces had higher G_{1C}'s than counterparts
- For two of the six surfaces, there was a noticeable reduction in G_{1C} with environmental aging

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Fracture Surfaces

- The majority of specimens what were bonded “as tooled” resulted in adhesion failure
- A few manufactured with the polyester PF60001 peel ply showed a mix mode failure (adhesion/interlaminar)
- All specimens that were sanded resulted in 100% interlaminar or a mix of interlaminar/adhesion failures
- No cohesion failures were obtained for the combination of materials used - likely due to low interlaminar fracture toughness of the DA 411U 150 prepreg

Nylon peel ply, as tooled
no environmental aging

Nylon peel ply, sanded
no environmental aging

60001 polyester peel ply, as tooled
with environmental aging

60001 polyester peel ply, sanded
with environmental aging

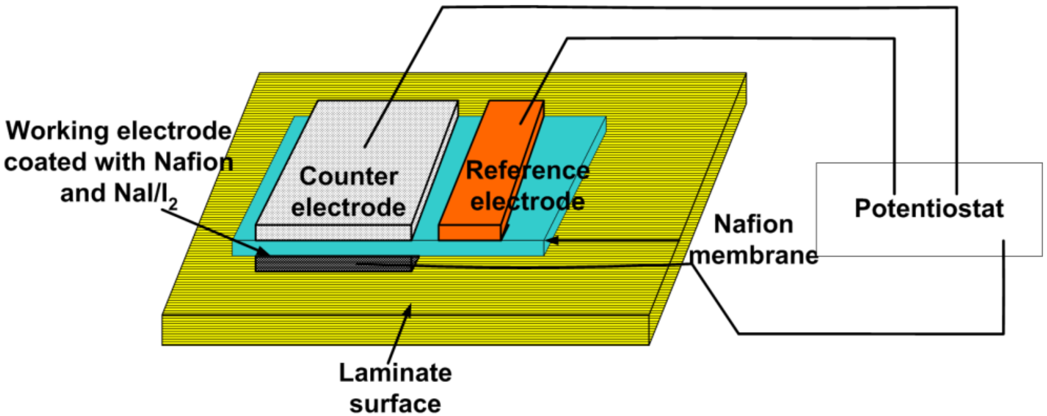
Electrochemical Sensor (ECS) Analysis

Solid-State electrochemical sensor – Methodology

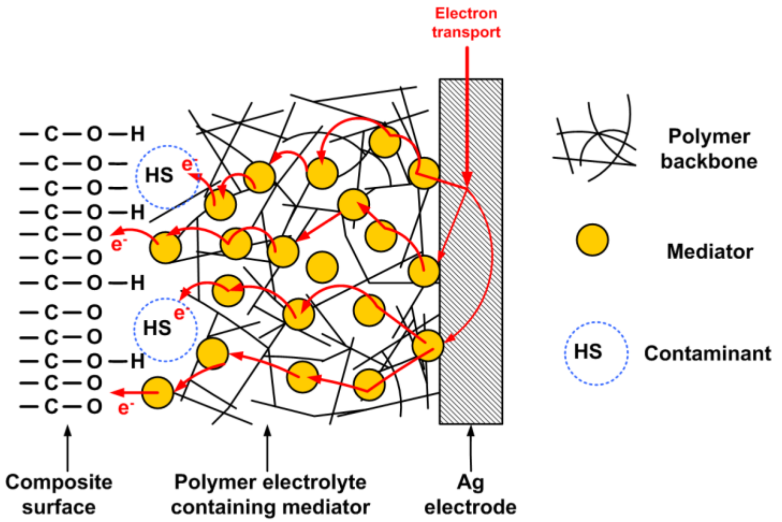
- Obtain maximum current peak from CV plots – corresponds to maximum electrochemical activity and potential contamination
- Establish trends and correlations with mechanical data and other surface analysis tools

Sensor design was modified and manufactured with a new configuration

- Reduction in internal resistance - improves CV response
- Working electrode is coated with the mediators



Schematic of the solid-state ECS



Operation principle of the solid-state ECS

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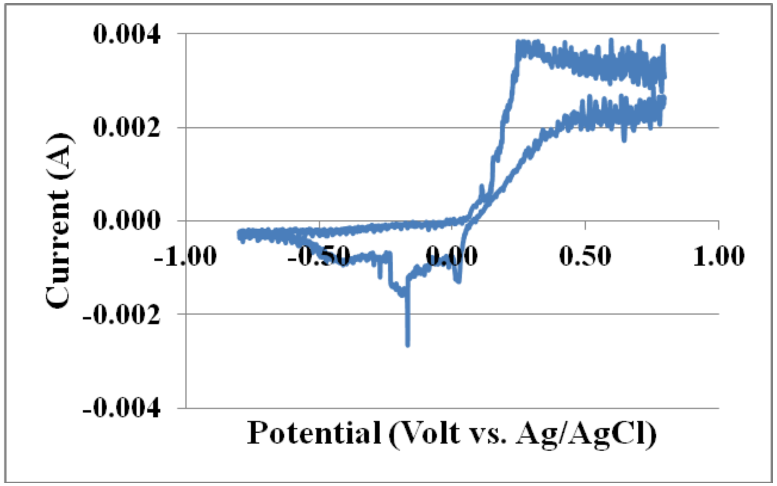
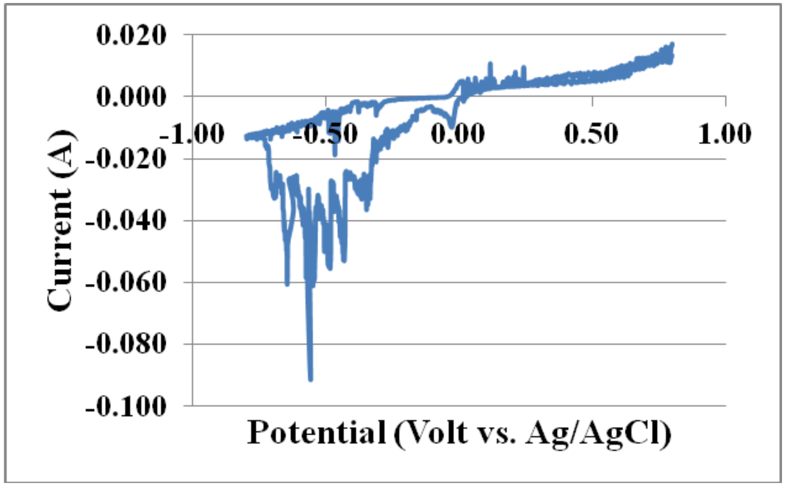
ECS Analysis

Solid-State electrochemical sensor – Cyclic Voltammetry results using specimens prepared with PF60001

- As tooled samples - a number of small peaks at positive polarization, and one large peak at the negative polarization. Peaks reveal the activity or contaminations on sample surface.
- Sanded samples - a number of peaks at negative polarization. The magnitude of the negative peaks is much greater than for the tooled samples. This indicates that the sample surface is an electron acceptor with a relative high oxidizing activity – indicates that the sanded surface is more active than as tooled surface.



CV for 60001 polyester peel ply samples – as tooled

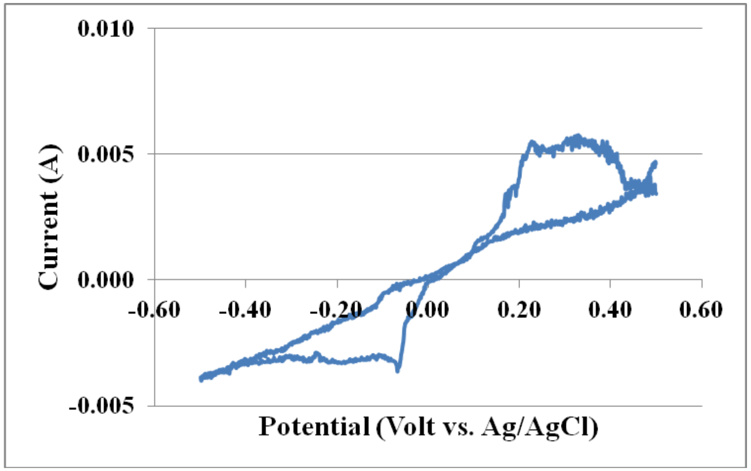


CV for 60001 polyester peel ply samples – sanded

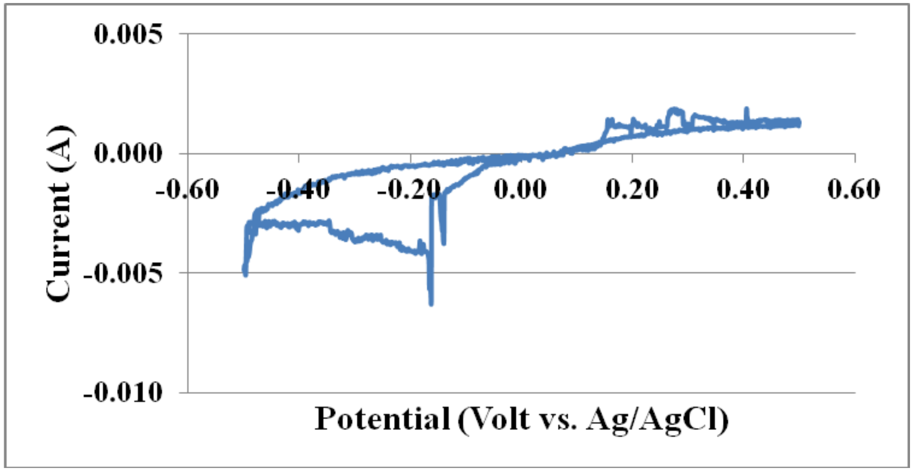
ECS Analysis

Solid-State electrochemical sensor – Cyclic Voltammetry results using specimens prepared with Fiberglast polyester peel ply

- As tooled samples – few small peaks at positive and negative polarization.
- Sanded samples - no peaks at positive polarization and few larger peaks at negative polarization. Peak magnitudes indicate activity is less than values obtained for the 60001 polyester samples.



CV for Fiberglast polyester peel ply samples – as tooled

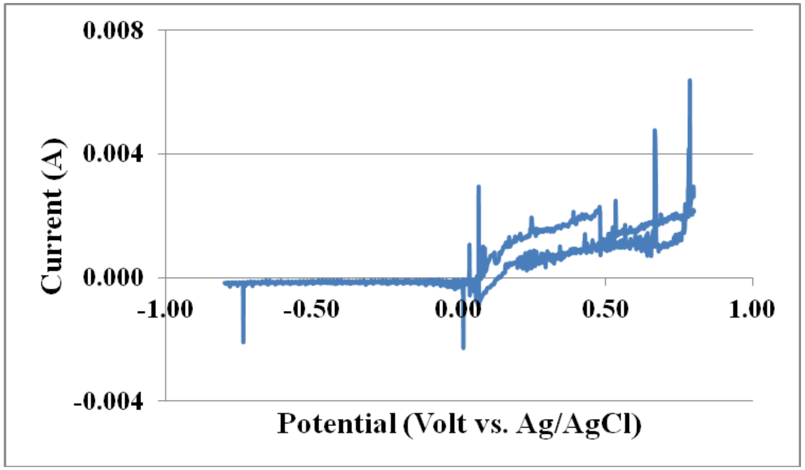


CV for Fiberglast polyester peel ply samples – sanded

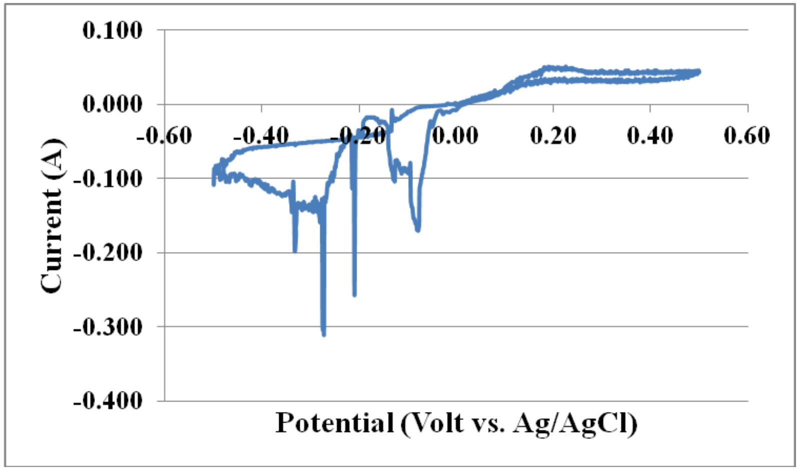
ECS Analysis

Solid-State electrochemical sensor – Cyclic Voltammetry results using specimens prepared with Fiberglast nylon peel ply

- As tooled samples – a few high peaks at the positive polarization and two peaks at the negative polarization.
- Sanded samples - no peaks at positive polarization and few larger peaks at negative polarization. Peak magnitudes indicate activity is larger than values obtained for the 60001 and Fiberglast polyester samples.



CV for Fiberglast nylon peel ply samples – as tooled



CV for Fiberglast nylon peel ply samples – sanded

CV results correlated well with the G_{1C} values – sanded nylon samples providing the best bonding surface for samples tested.

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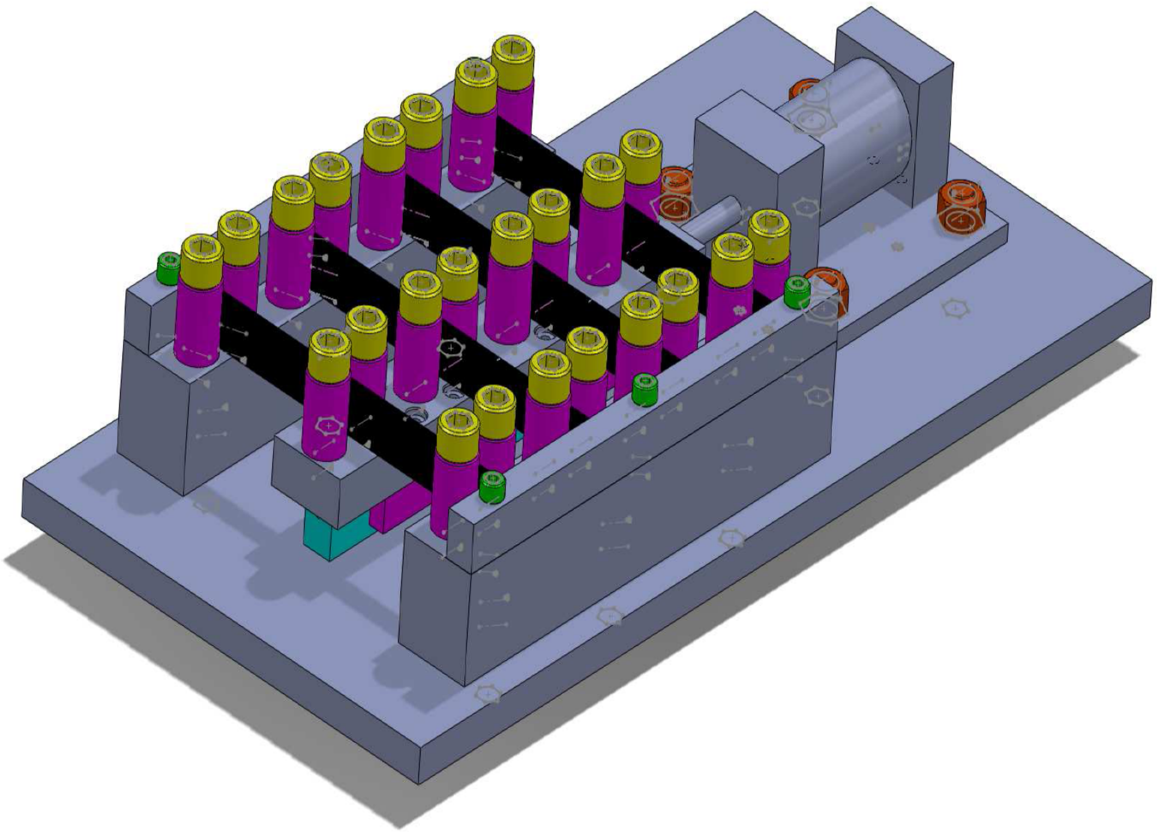
Fatigue Fixture

- Manufactured using stainless steel materials
- Center section slides on a ball bearing carriage
- Designed to load up to four 9 inch specimens with a deflection of 2 inches DA
- Current stainless steel pneumatic / hydraulic actuator is rated to 400 psi with a 1 inch bore diameter – can load only one 20 ply specimen
- Pneumatic controller can operate up to 2 Hz at 150 psi



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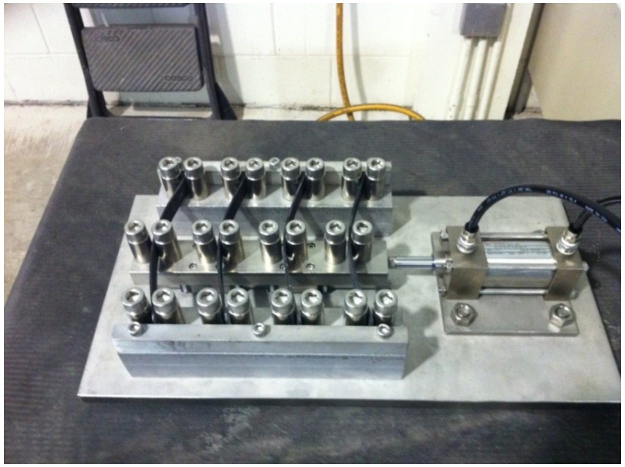
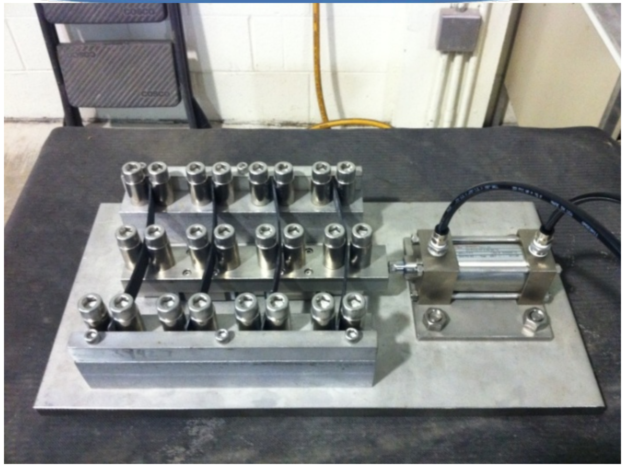
Fatigue Fixture

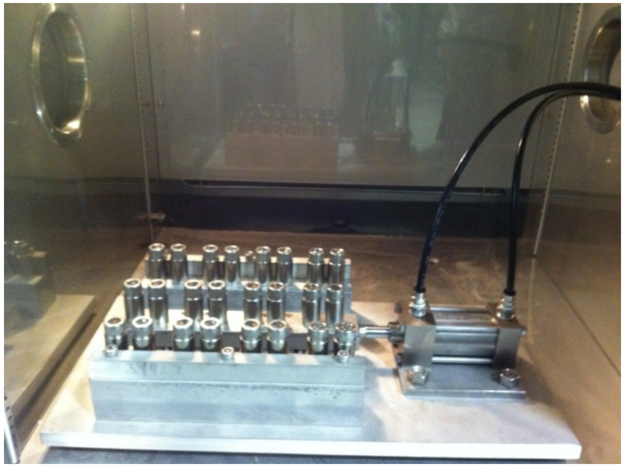


Fixture at 1 inch deflection – four 10 ply specimens



Fixture at 1 inch deflection – four 10 ply specimens

Thermatron environmental chamber



Fixture in environmental chamber

Fatigue Fixture

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Looking Forward

- Future work
  - Validate that the bondline is fatigued using current actuator
  - Procure larger diameter actuator to fatigue four 24 ply specimens simultaneously
  - Obtain baseline data using 3M AF555 adhesive and T800 prepreg system
  - Condition specimens manufactured with new material
    - Mechanical loading
    - Environmental exposure (increase duration)
    - Mechanical loading and environmental exposure
  - Repeat testing with specimens manufactured with undesirable bonding conditions
- Benefit to Aviation
  - Better understanding of durability assessment for adhesively bonded composite joints
  - Assisting in the development of bonding quality assurance procedures

End of Presentation.

Thank you.

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