Effect of Surface Contamination on Composite Bond Integrity and Durability

2013 Technical Review

Dwayne McDaniel
Florida International University

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 by conditioning the specimens prior to testing

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Effect of Surface Contamination on Composite Bond Integrity and Durability

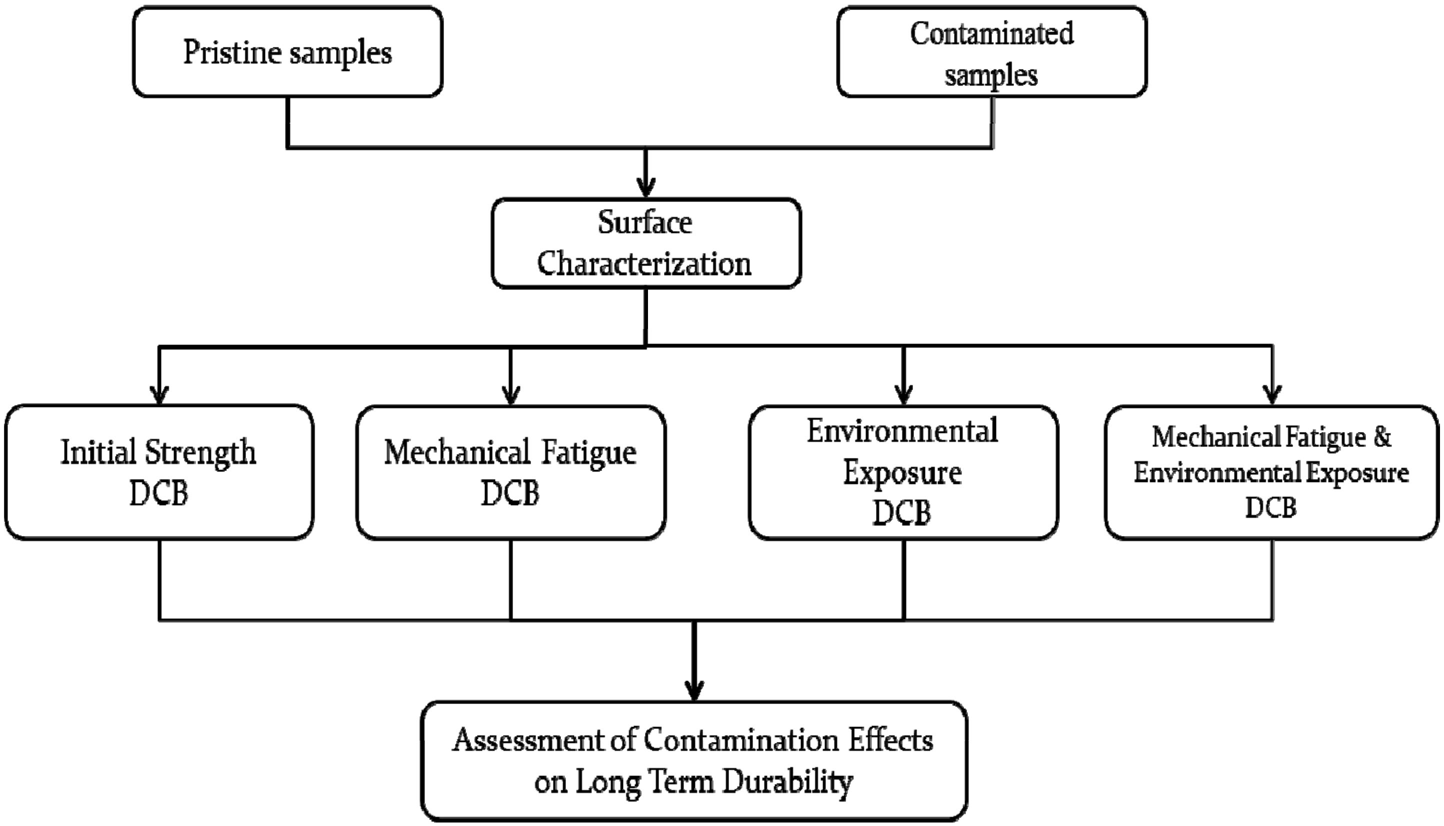
- **Principal Investigators & Researchers**
  - Dwayne McDaniel, Xiangyang Zhou, Tomas Pribanic

- **Students**
  - Vishal Musaramthota, Juanjuan Zhou, Sirui Cai

- **FAA Technical Monitor**
  - David Westlund

- **Industry Participation**
  - NRC, Boeing, Exponent

Durability Assessment Procedure



Bonding Material System

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 (350F cure) from APCM
- 3M AF 163-2 adhesive film (9.5x2mills, 250F cure)
- 3M AF555U adhesive film (2.5x4 mills, 350F cure)
- Peel plies: Polyester and nylon from Fibreglast, Precision Fabric peel ply 60001

Current materials:

- Toray P 2362W-19U-304 T800 Unidirectional prepreg system (350F cure)
- 3M AF 555 adhesive film (7.5x2 mills, 350F cure)
- Precision Fabric polyester peel ply 60001

Specimen Conditioning:

- Environmental Chamber : 50°C, 95% RH, for 8 weeks

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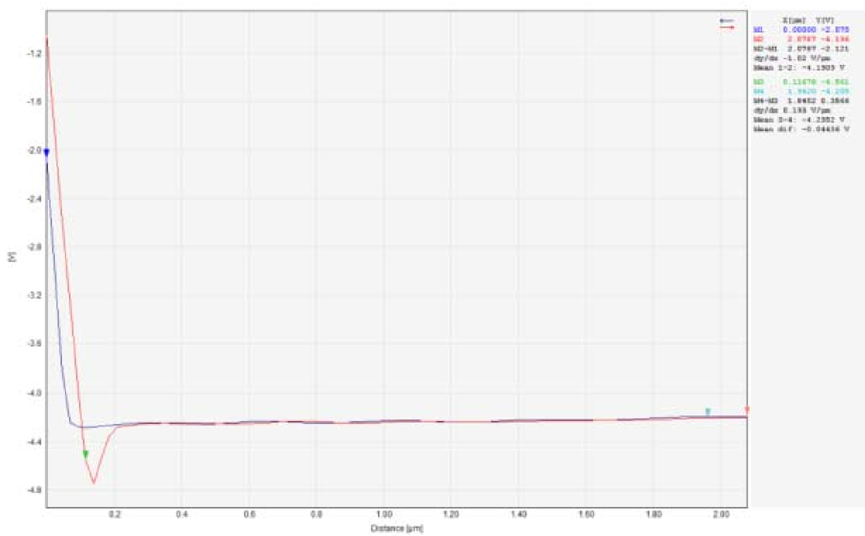
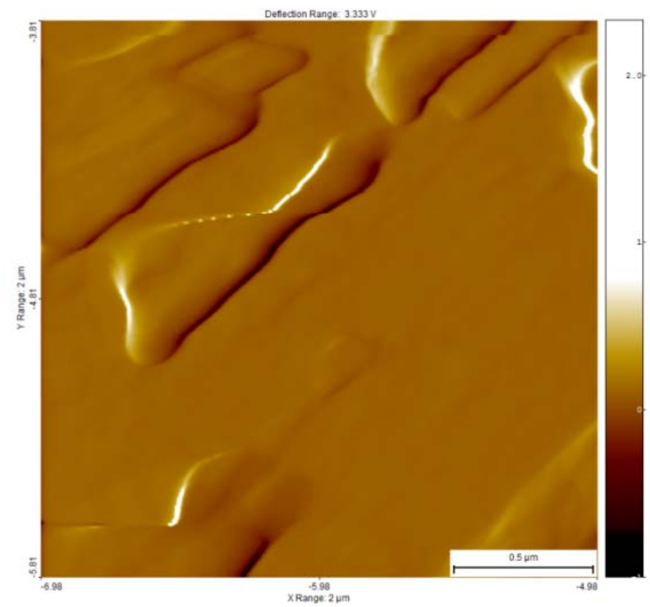
Surface Characterization Methods

<u>Atomic Force Microscopy</u>

- FIU 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.



Typical force deflection curve



Topography image of peel ply imprint peak

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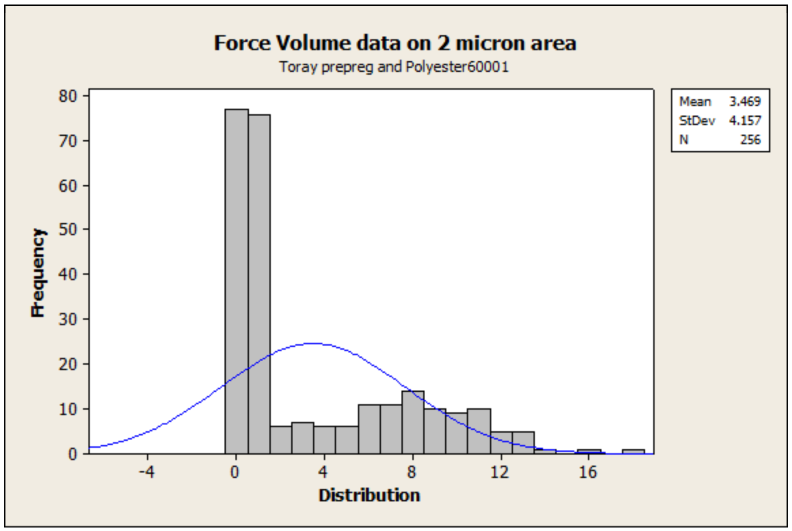
Surface Characterization Methods

Atomic Force Microscopy

- A 16 x16 grid is used to generate 256 adhesion force measurements using a force volume approach
- Force volume data was collected in a controlled environment (~0% humidity) and in ambient air



Histogram of adhesion force measurements

AFM Force-Volume data	*Detach Force (nN)*	
	Ambient Air	*Controlled Environment*
Mean	1.35	3.47
Standard Deviation	0.98	4.16
Maximum	6.03	18.44
Minimum	0	0.20

Adhesion force measurement data

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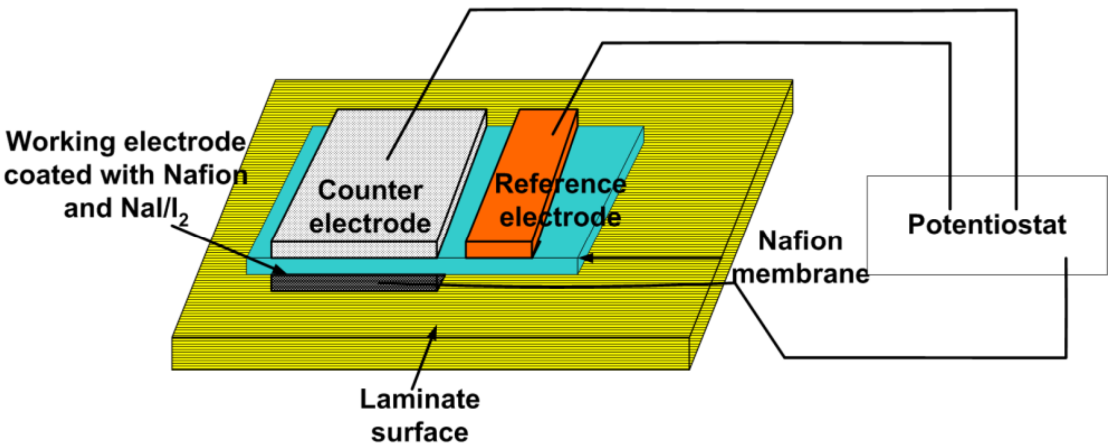
Surface Characterization Methods

Electrochemical Sensor

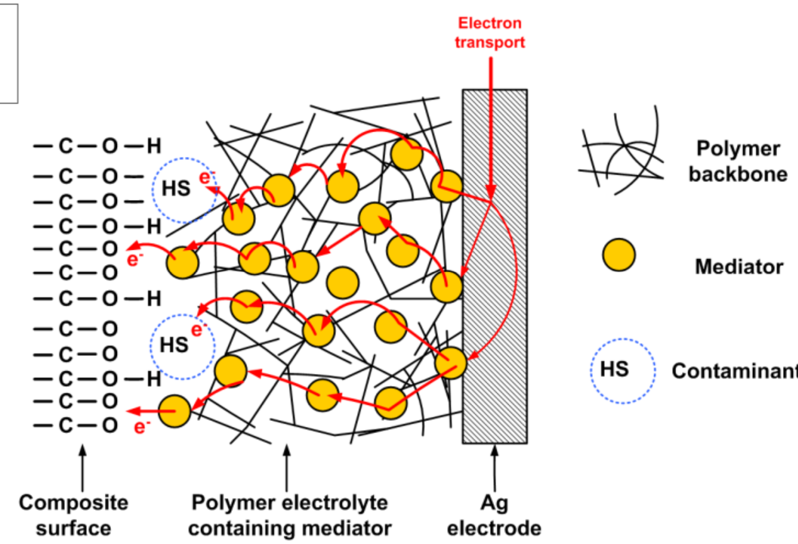
- An all solid-state electrochemical sensor is being investigated to detect variations in surface electrochemical activity.

- CV and EIS signatures obtained from the sensor correlate with the surface electrochemical activity which can be affected by contamination.



Principle of operation

Schematic of ECS



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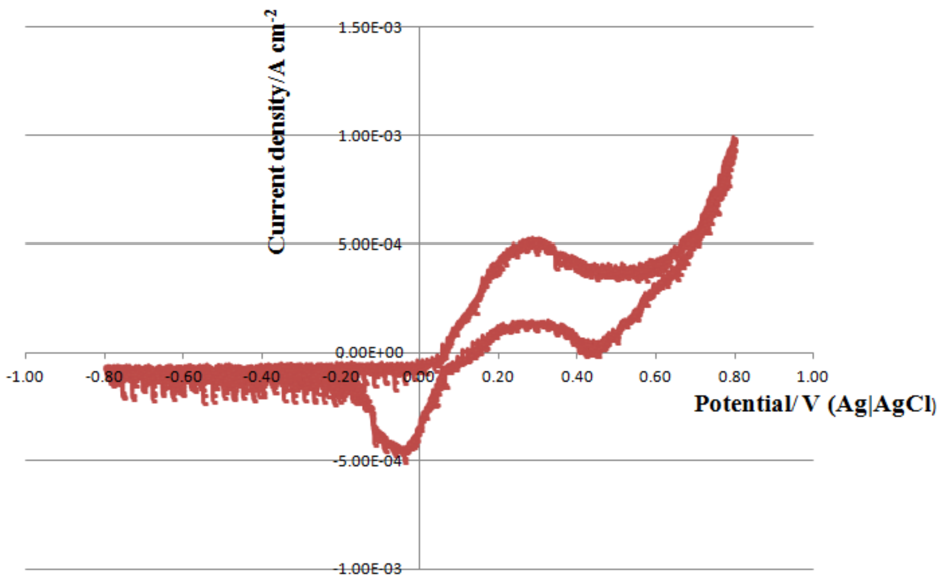
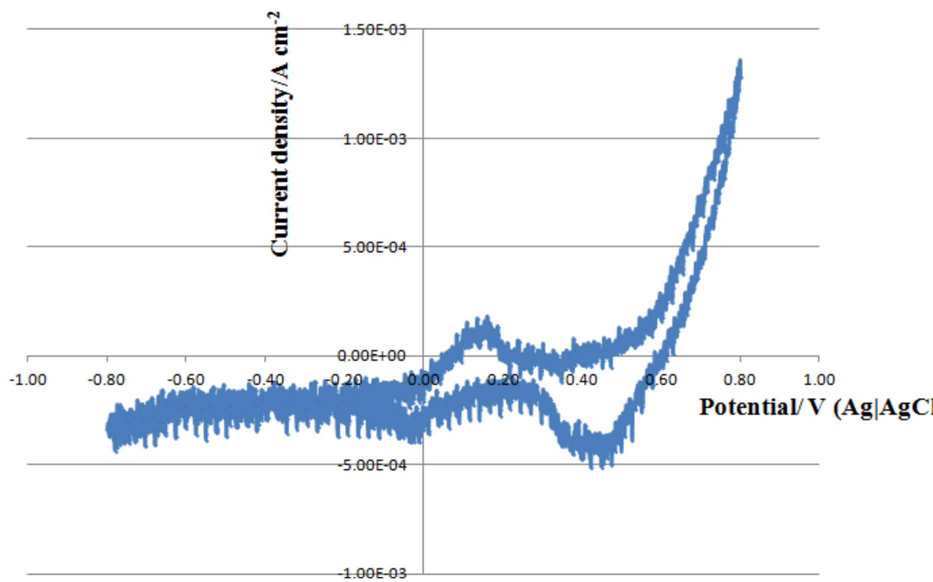
Surface Characterization Methods

Electrochemical Sensor

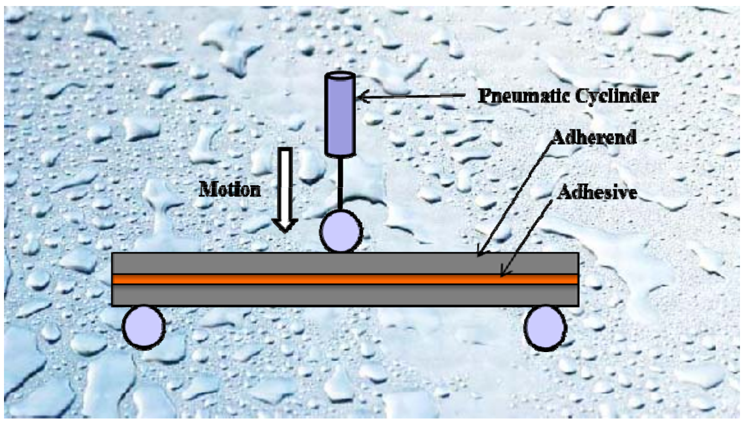


Typical CV scans using the ECS on Toray prepreg and PF 60001 polyester peel ply



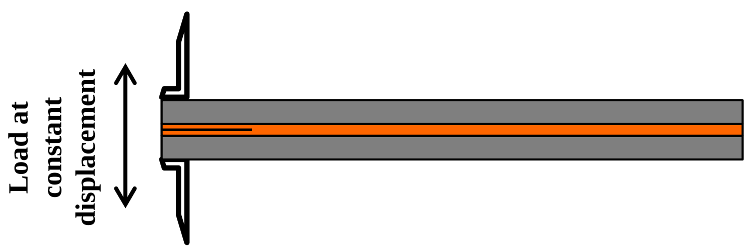
Fatigue Loading

DCB specimens are conditioned by mechanically fatiguing and/or exposure to an accelerated aging environment. A fatigue structure was manufactured that loads the specimens in three point bending.



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)



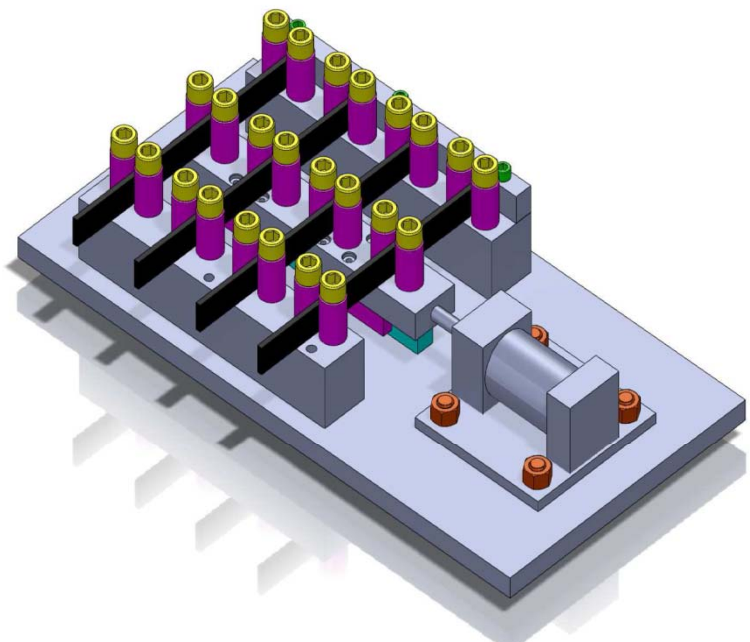
Disadvantages:

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

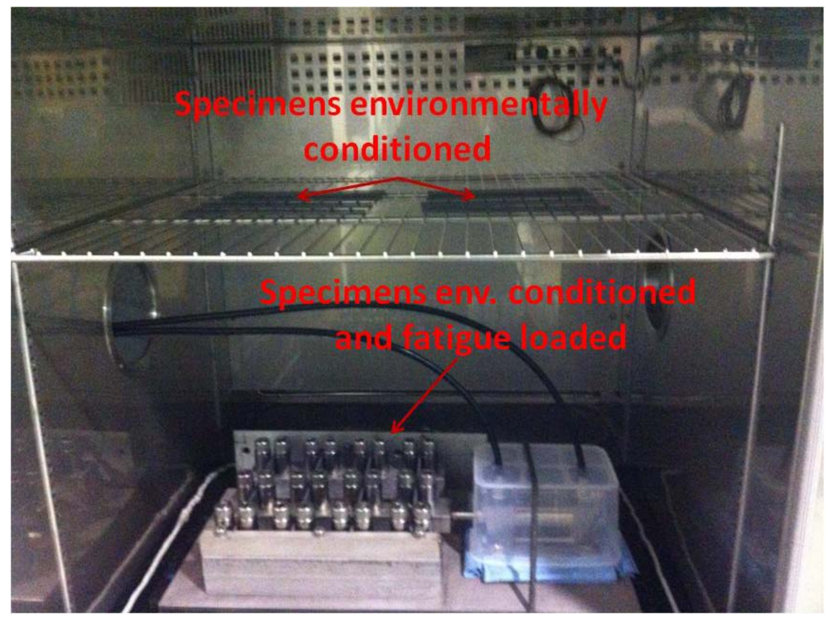
Aging of Specimens

The fatigue fixture was manufactured so it 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

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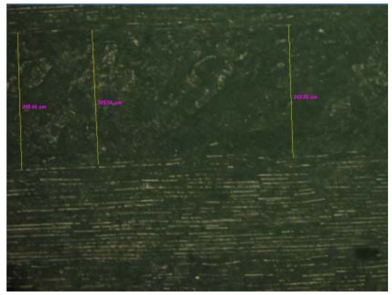
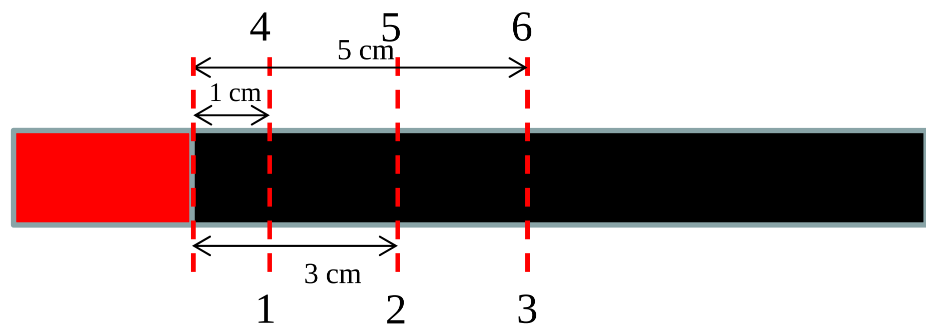
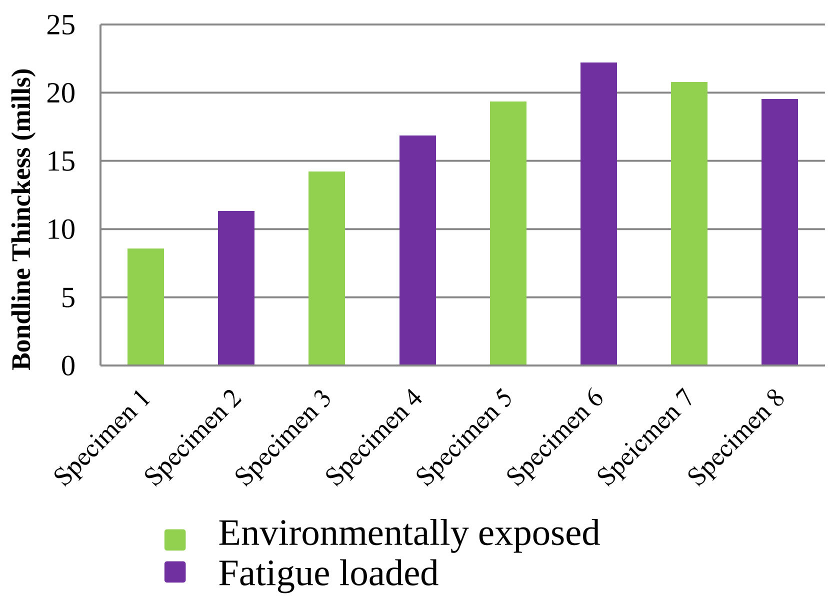
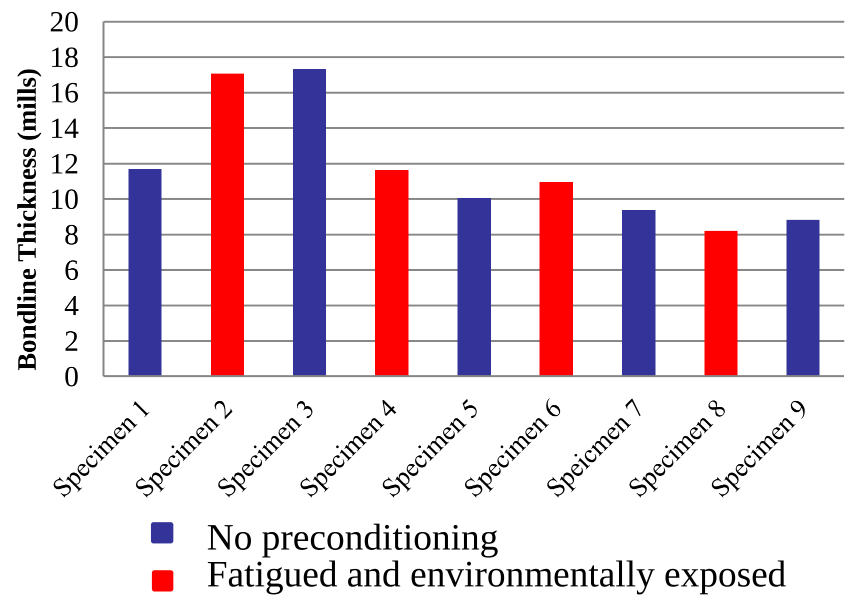
Bondline Thickness

- Bondline thickness measurements of all specimens were taken
- Average values of 6 measurements are reported









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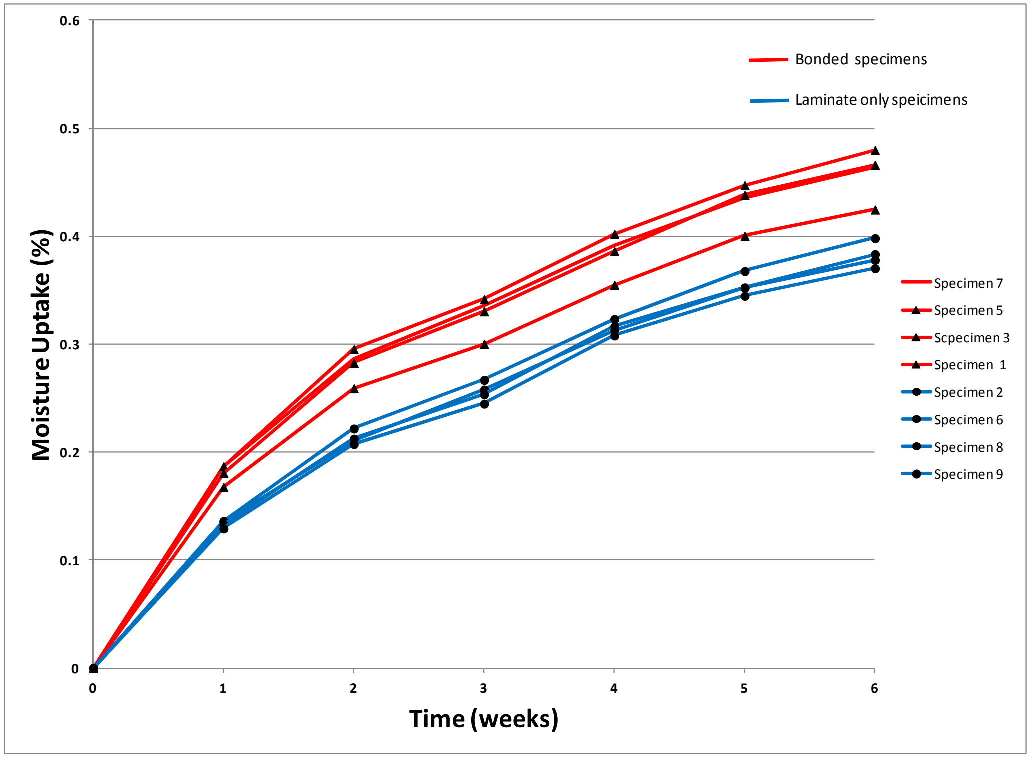
Moisture Saturation

Moisture uptake of the specimens placed in the environmental chamber were monitored to project the saturation point.

Specimens with and without adhesive were evaluated.



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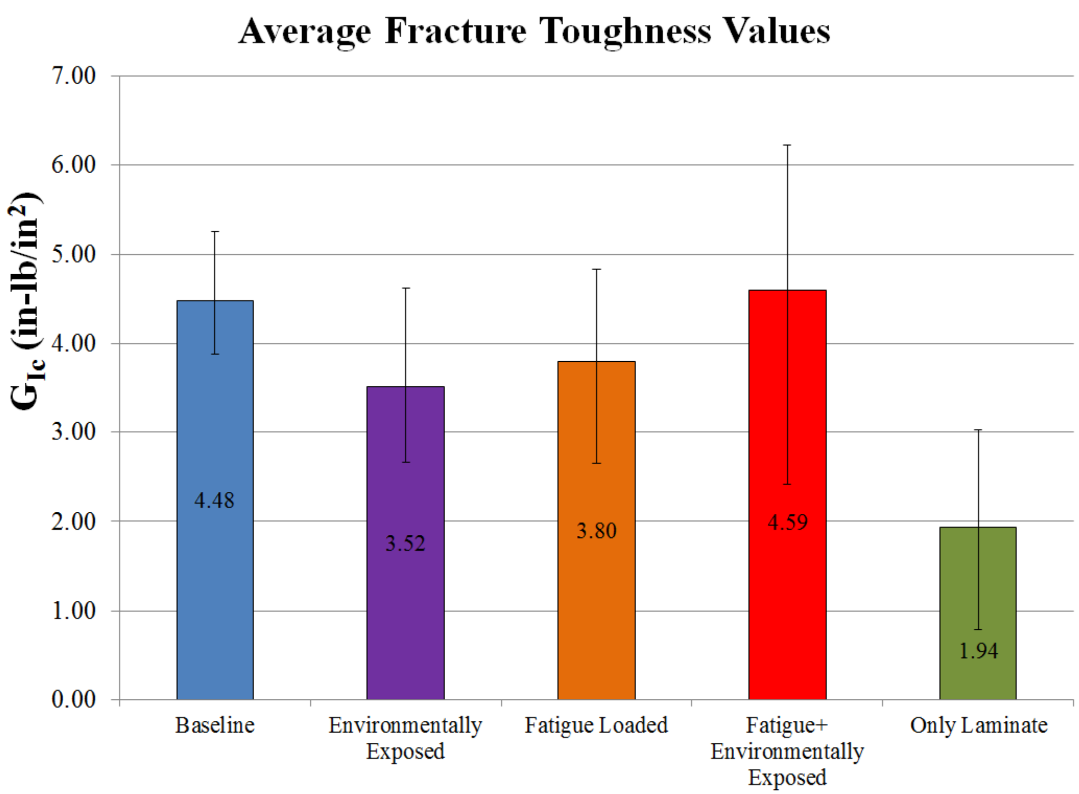
Fracture Toughness Results

Strain energy release rates were obtained for baseline and conditioned DCB specimens



G_{IC}	Average (in-lb)/in²	Max (in-lb)/in²	Min (in-lb)/in²
Baseline	*4.48*	*5.26*	*3.88*
Environmentally Exposed	*3.52*	*4.63*	*2.67*
Fatigue loaded	*3.80*	*4.83*	*2.65*
Fatigue+ Environmentally Exposed	*4.59*	*6.23*	*2.41*
Only Laminate	*1.94*	*3.02*	*0.79*

- Environmentally exposed - aged for 8 weeks
- Mechanically fatigue loaded in ambient air for 2.8 million cycles
- Combined fatigue loading and environmental exposure - aged for 8 weeks

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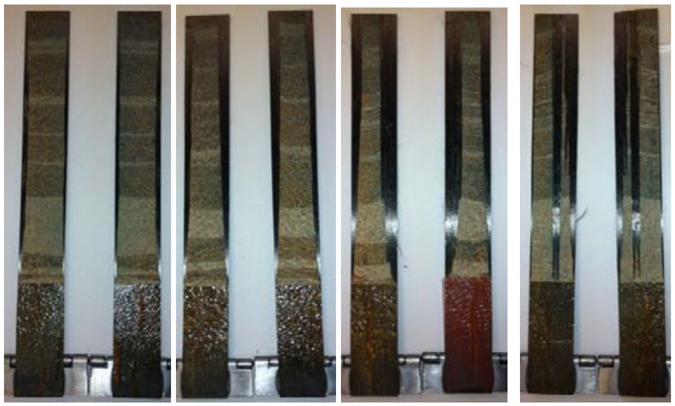
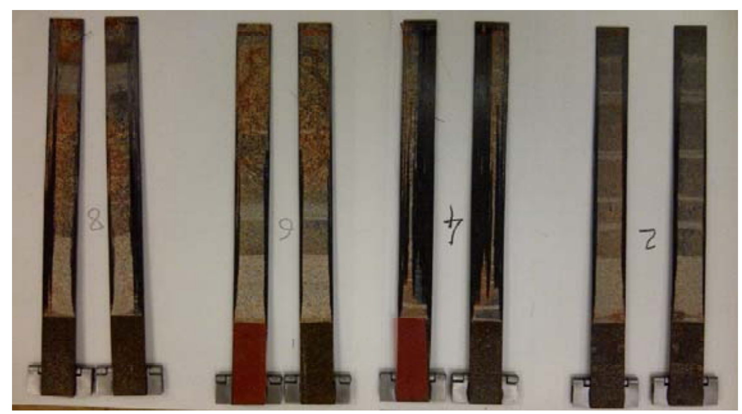
Mode of Failure



Baseline

Environmentally exposed

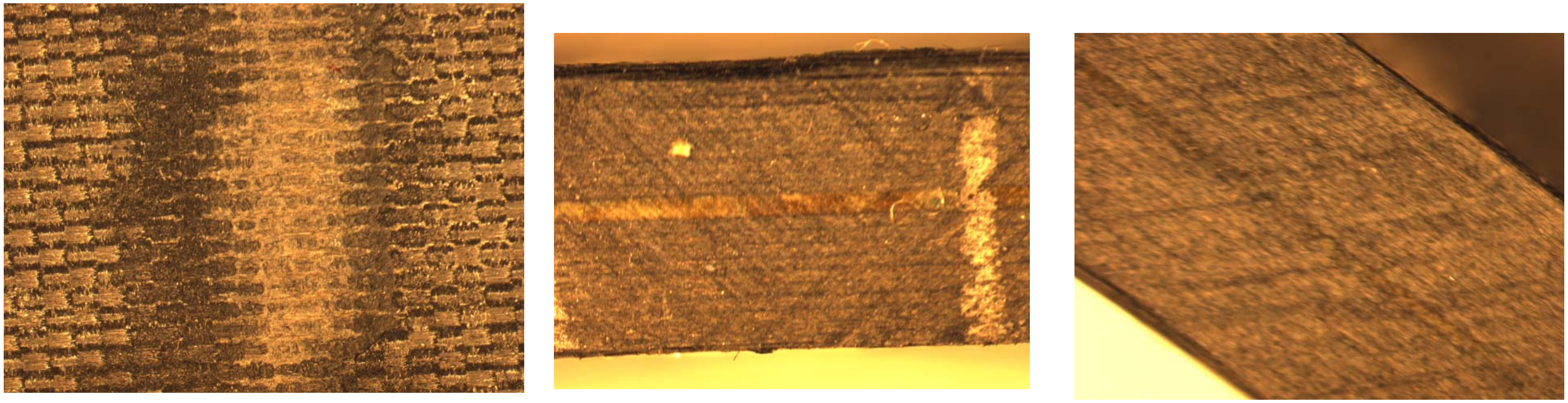
Mechanically fatigued



Fatigue and environmentally exposed

Fatigue Specimens 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 resulted from fatigue loading



Typical optical microscope images of fatigued specimens (16 x magnification)

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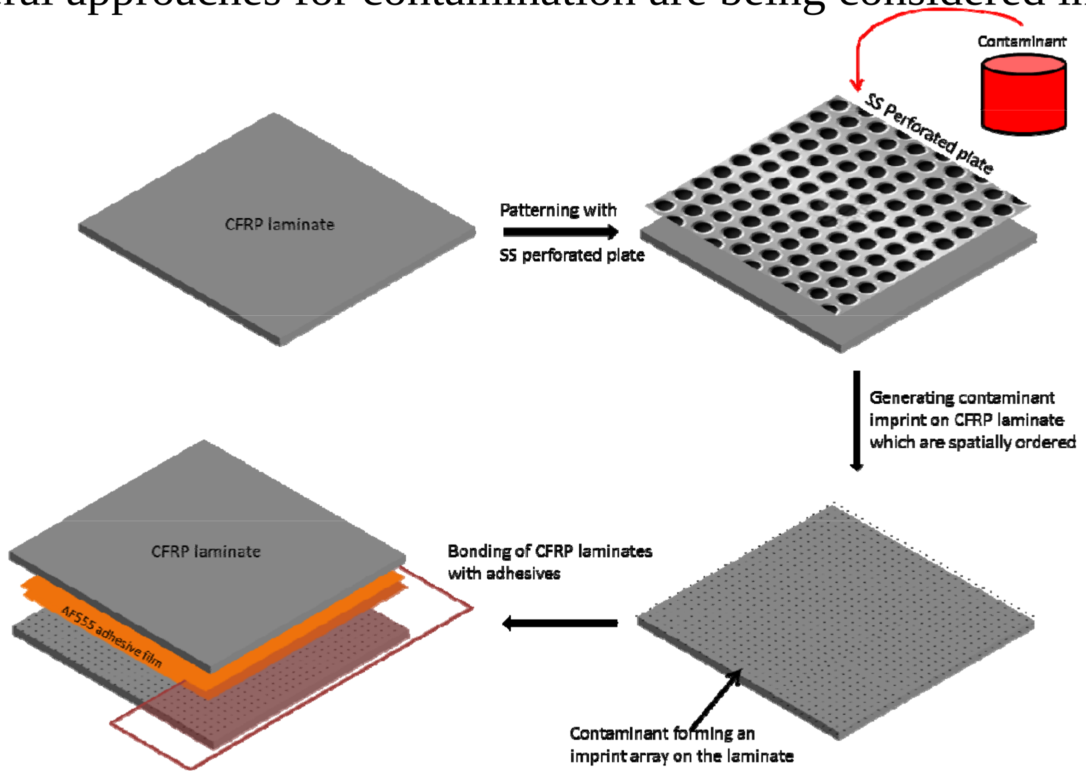
Contamination Procedure

Undesirable bonding conditions will be used to evaluate how these conditions can effect durability. Several approaches for contamination are being considered including



- Spatially ordered array of contaminant over the entire substrate
- Perforated stainless steel mesh with various hole diameters can be used to vary the degree of contamination
- Provide a high concentration of contaminant at predetermined sites

Conclusions

- A general procedure has been developed to test the durability of adhesively bonded joints. This involves the conditioning of specimens using a 3 point bending fixture for mechanical loading in an environmental chamber.
- Specimens that have been environmentally aged and mechanically fatigued showed slight reductions in bond strength.
- Specimens subjected to the combined loading showed approximately the same bond strength.
- Adhesion force measurements and ECS measurements from the baseline specimens showed differences from the previously tested material sets. This data will be evaluated against measurements from contaminated specimens.
- Contamination procedures are being investigated that will allow for a controlled amount of contaminant at specified locations.

Path Forward

Future Work:

- Develop and validate controlled procedure to contaminate surfaces prior to bonding
  - including fabrication, testing and data analysis
- Investigate the fabrication or modification of the fatigue fixture that will increase the number of specimens
- Characterize contaminated surfaces prior to bonding (AFM, ECS, water contact angle, etc)
- Repeat conditioning on contaminated specimens
- Measure bond degradation of contaminated specimens (DCB testing) to determine effects on durability and assess the

Benefit to Aviation:

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

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End of Presentation.

Thank you.

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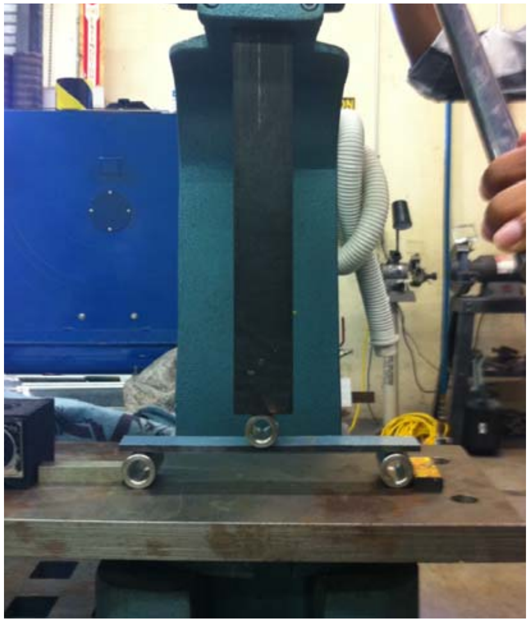
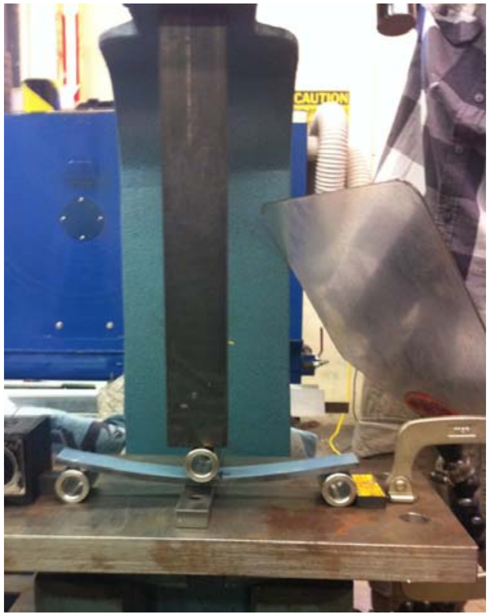
Back up

Surface Effects on Allowable Deflection

- 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 $212x10^3$ psi > 1 in specimen deflection
- Specimens have an effective length of 8 in
- Surface effect of 1 inch diameter rollers lowered Su to $129x10^3$ psi -> 0.6 in deflection
- Conditioned specimens will be deflected to 0.5 inches (2,300 psi of transverse shear).
- Frequency will be increased accordingly





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Literature Review

Adhesive bonding community relies largely on usage of Lap Shear joints to establish design allowables (Davis & Tomblin, ‘07) but to establish the G_{Ic} of a composite material and to access the durability- DCB tests have to be considered.

“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)

Joannie, et. al., – studied the sorption behavior of water in composite matrices at elevated temperatures

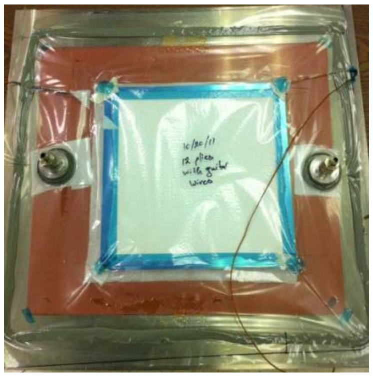
Stress ratio introduced into the composites will have an influence on its fatigue life (Agarwal and James, ‘75)

Specimen conditioning using mechanical loading in harsh environments

- Service environments can significantly affect the joint types and materials of adhesively bonded composite joints (Ashcroft, et. al,. ‘00)
- Knight, et. al. – hygrothermally aged SLS specimen for longer durations and observed a decrease in shear strengths and change in failure modes.

Manufacturing Procedure

Fabrication of laminates



Cure cycle @350F

Bonding of laminates

Secondary cure @350F

Variation of ASTM D5528

Surface characterization, testing, and data analysis

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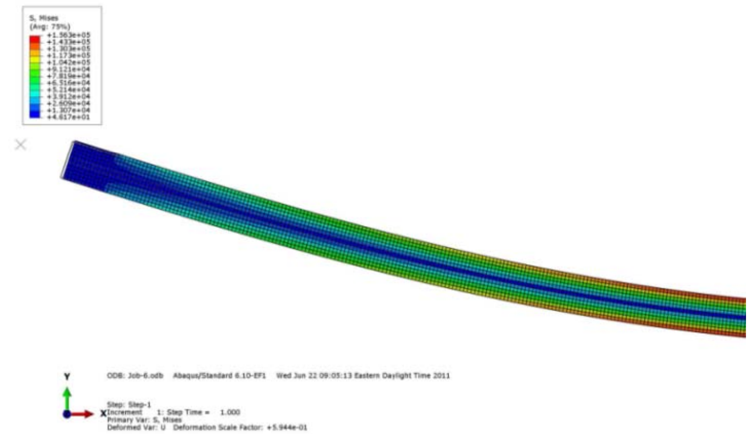
Specimen Design

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	1.03×10^7 + (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)

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