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Effect of Surface Contamination on Composite Bond Integrity and Durability

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JAMS 2014 Technical Review
March 25-26, 2014

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.

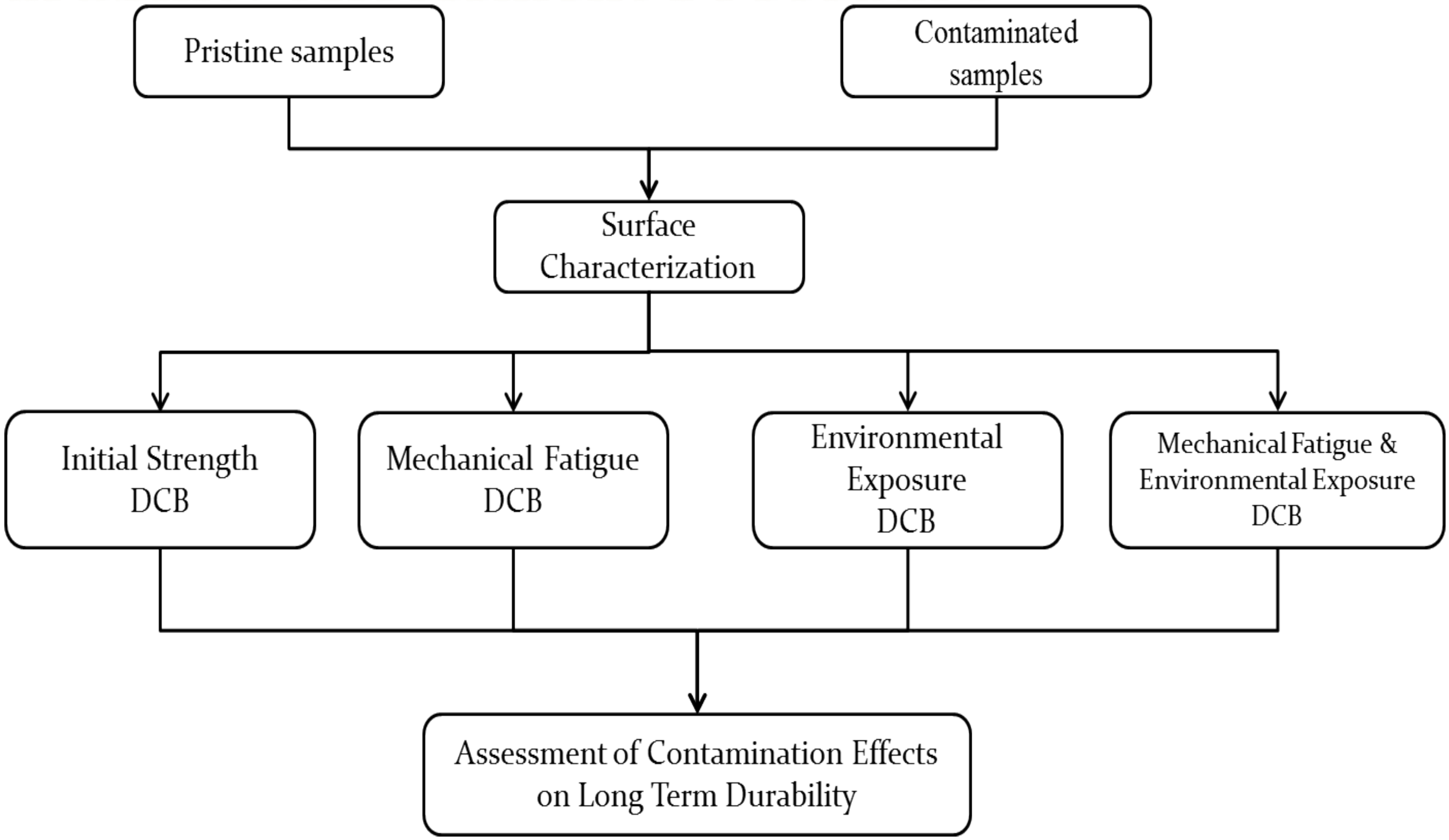
Objective

- ✓ 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.

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**
  - Curt Davies, David Westlund
- **Industry Participation**
  - Exponent, NRC

Durability Assessment Procedure



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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 mills, 350F cure)
  - Precision Fabric polyester peel ply 60001

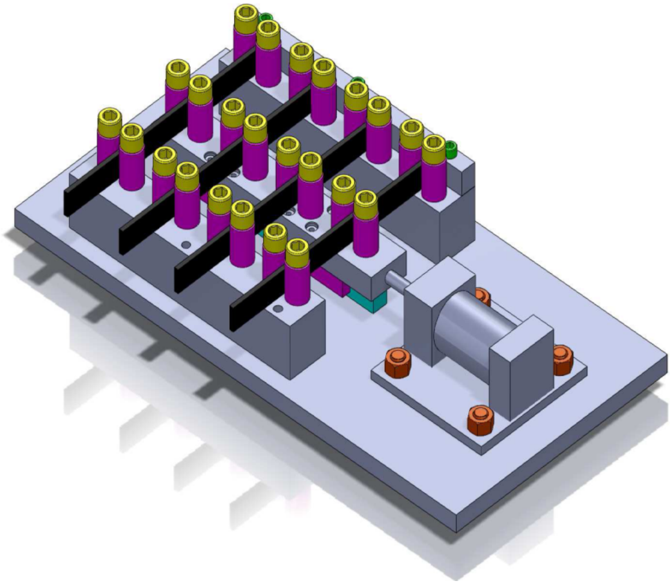
- Contaminants:
  - Silicone Spray from CBS Aerosol & Paint, Inc
  - Freekote 700-NC from Henkel Corporation

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

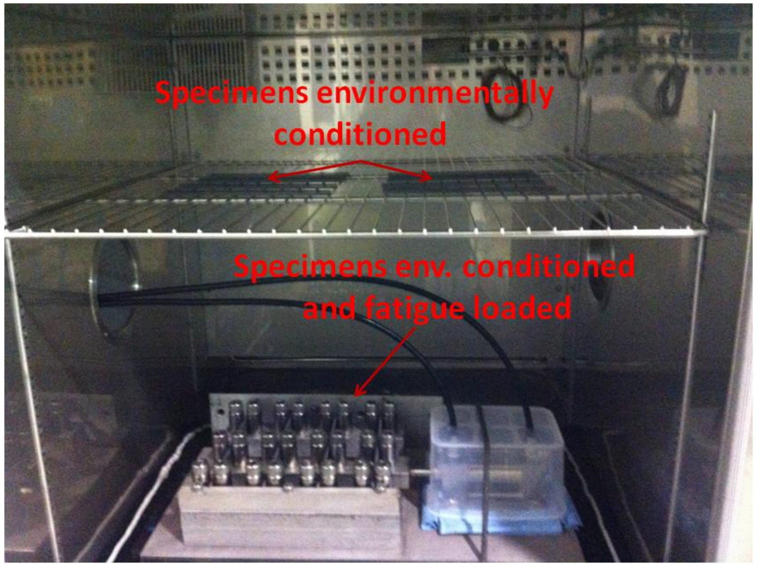
Accelerated Aging Procedure

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

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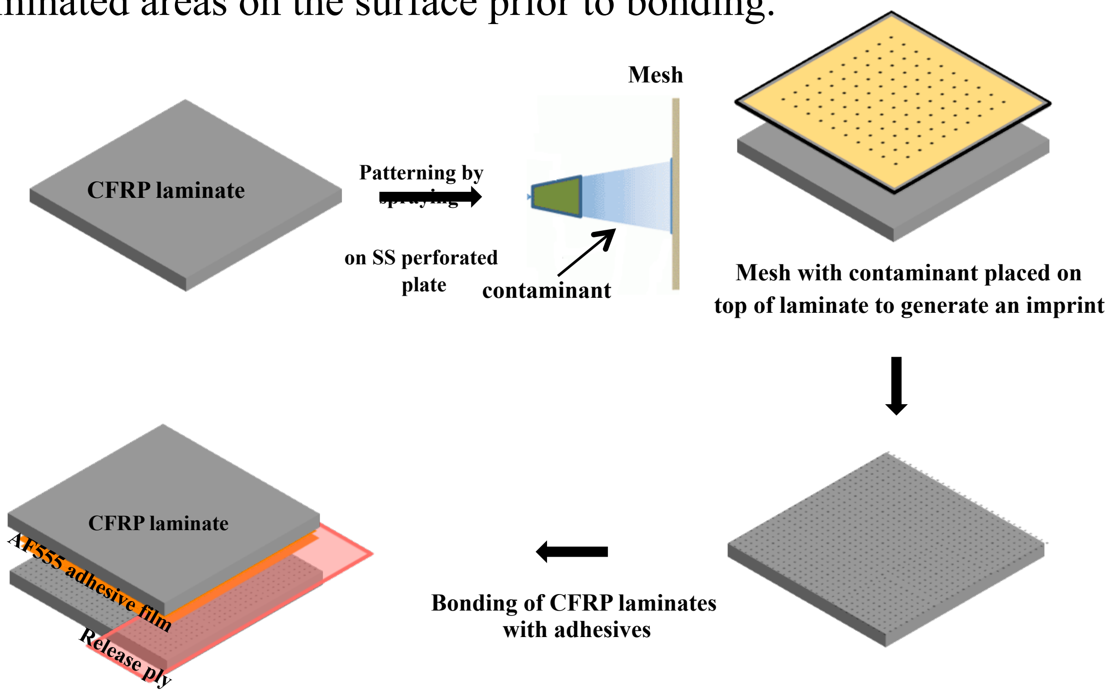
Contamination Procedure

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

- Mesh approach
- Stamped approach

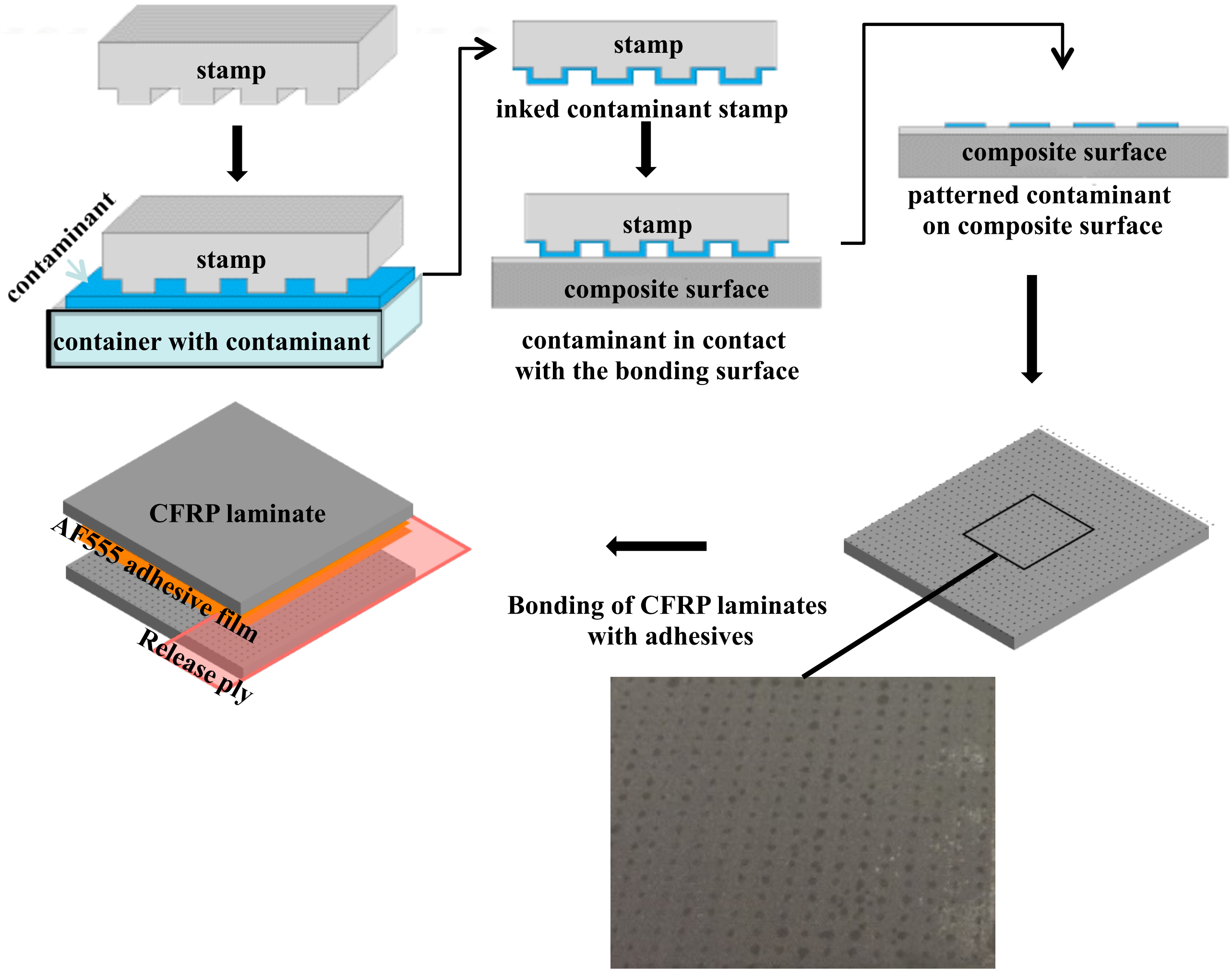
For both cases, the contamination procedure aims to create weak bonds by placing a spatially ordered array of contaminated areas on the surface prior to bonding.

Mesh Contamination Approach



Contamination Procedure

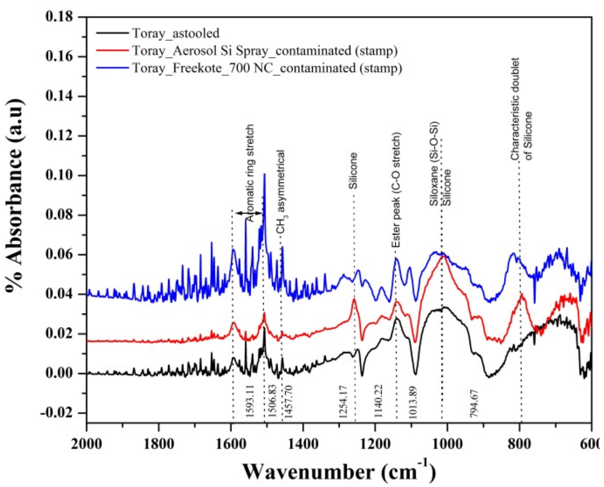
Stamped Approach



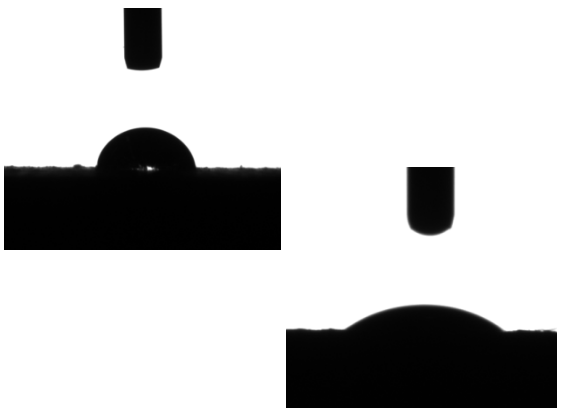
Surface Characterization Methods

<u>Contaminated Specimens</u>

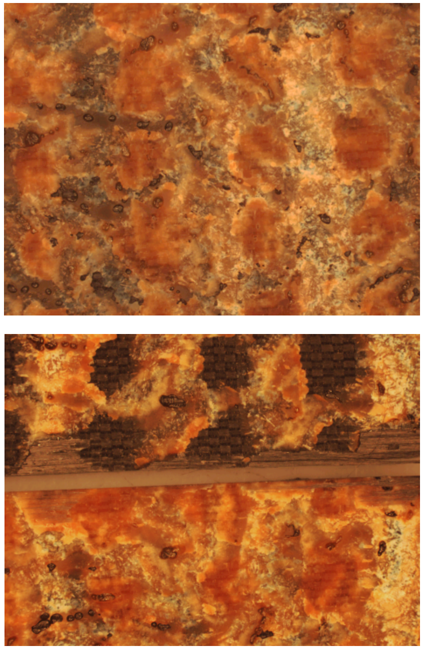
- FTIR data can be used to identify surface molecular bonds – identify contamination.
- Water contact angle - Determines the wetting characteristic behavior of various liquids on the composite surface.
- Optical Microscopy- Inspecting fracture surfaces, Line profile analysis.



FTIR spectrum on CFRP substrate with contaminant



Contact Angle image of CFRP substrate



Optical Microscopy image of failed surfaces

Gravimetric Analysis

- A Metler Toledo AB 304-S weighing system utilized
- Establishing the weight gains before and after contamination
- Differences in weight gains helps in quantifying the contamination
- Moisture uptake % is also determined

Fracture Toughness/DCB Testing

- DCB tests are established to determine the interlaminar fracture toughness as per ASTM D5528
- Reveals data for the energy release rate, crack growth length, and also provides the dominant mode of failure

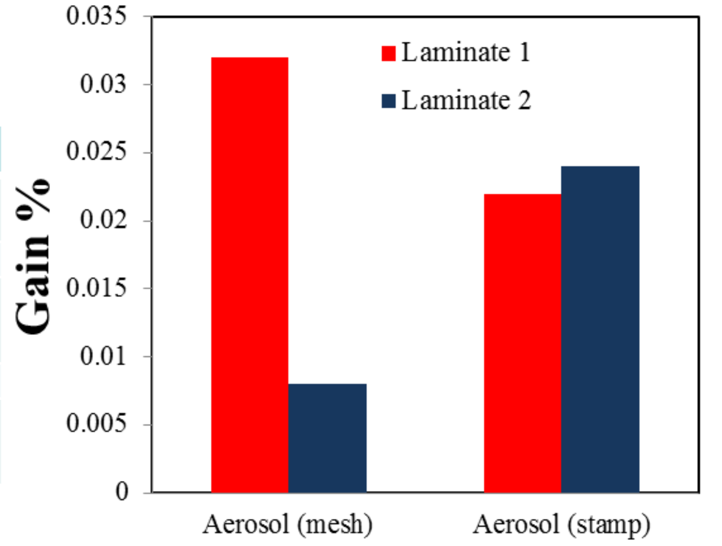
Configuration: Loading rate - 2.5 to 5.0 mm/min in the direction perpendicular to the specimen from one of the edges

RESULTS

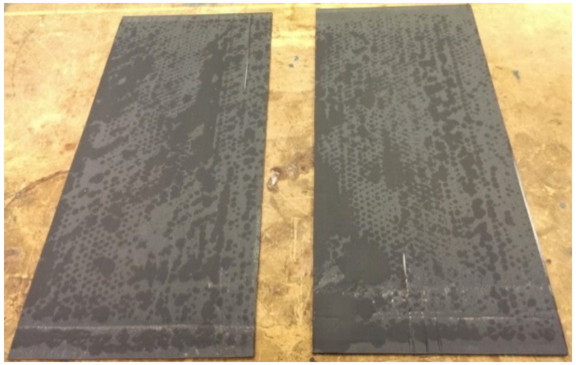
Gravimetric Analysis

Mass changes between Mesh and Stamp approach

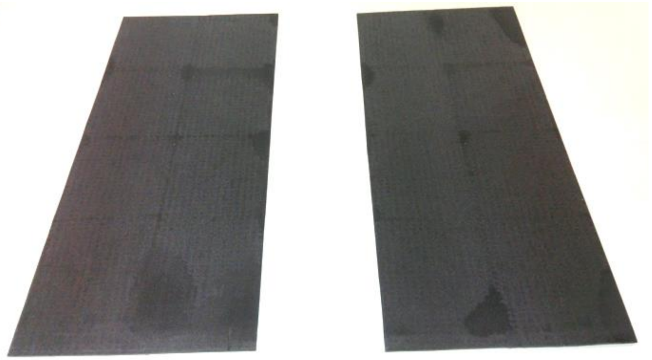
Contaminant deposited-Aerosol				
		Before (gms)	**After (gms)**	**Gain %**
Aerosol Si spray (mesh)	Laminate 1	123.59	123.63	0.032
	Laminate 2	122.52	122.53	0.008
Aerosol Si spray (stamp)	Laminate 1	123.04	123.06	0.022
	Laminate 2	124.18	124.21	0.024



Mesh Approach



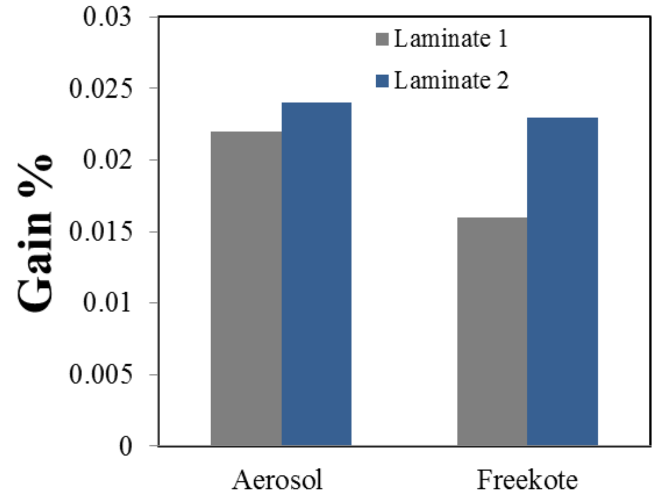
Stamp Approach



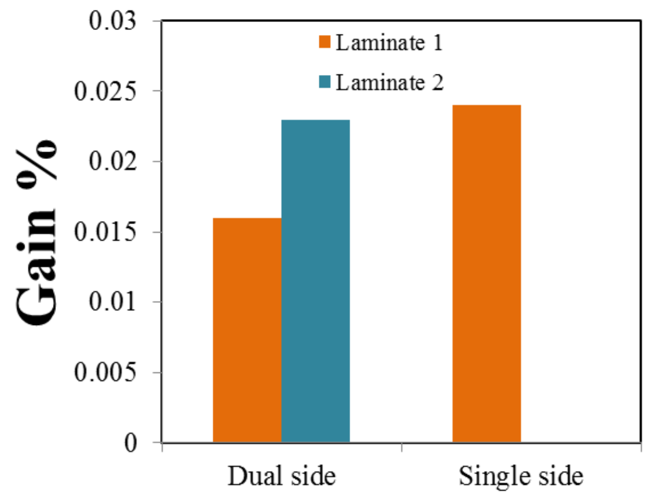
Stamp approach- promising and consistent

Gravimetric Analysis

Contaminant deposited-Aerosol & Freekote				
		Before (gms)	**After (gms)**	**Gain %**
Aerosol Si spray (stamp)	Laminate 1	123.04	123.06	0.022
	Laminate 2	124.18	124.21	0.024
Freekote 700NC (stamp)	Laminate 1	121.35	121.37	0.016
	Laminate 2	125.46	125.49	0.023



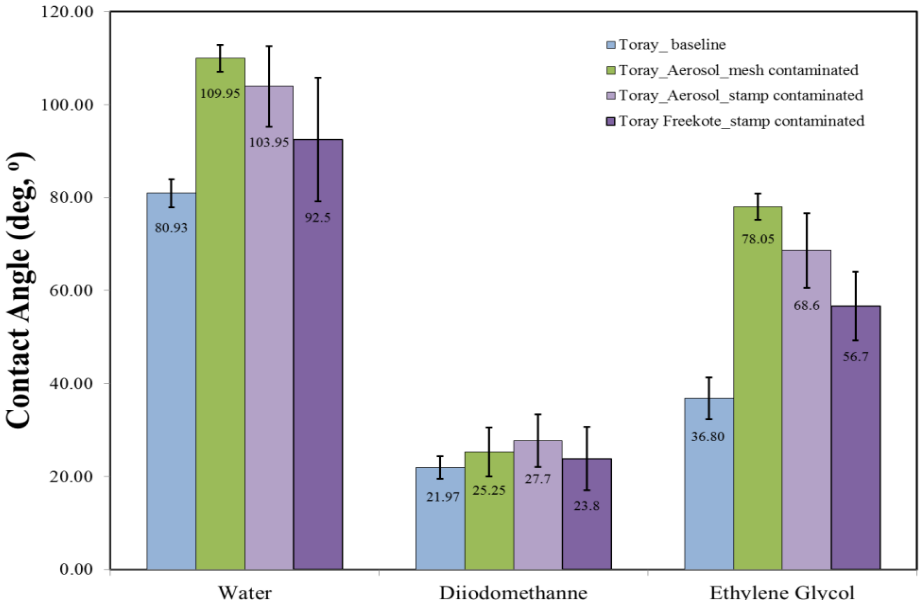
Contaminant deposited-Freekote				
		Before (gms)	**After (gms)**	**Gain %**
Freekote 700NC (Dual side)	Laminate 1	121.35	121.37	0.016
	Laminate 2	125.46	125.49	0.023
Freekote 700NC (Single side)	Laminate 1	122.41	122.44	0.024
	Laminate 2	NA	NA	NA

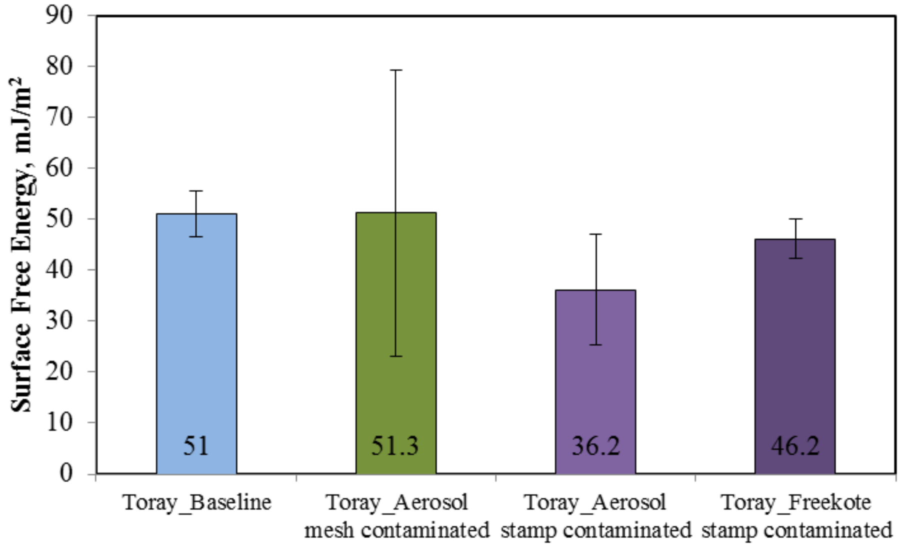


Only one laminate is contaminated

Surface Characterization Methods

Water Contact Angle measurements





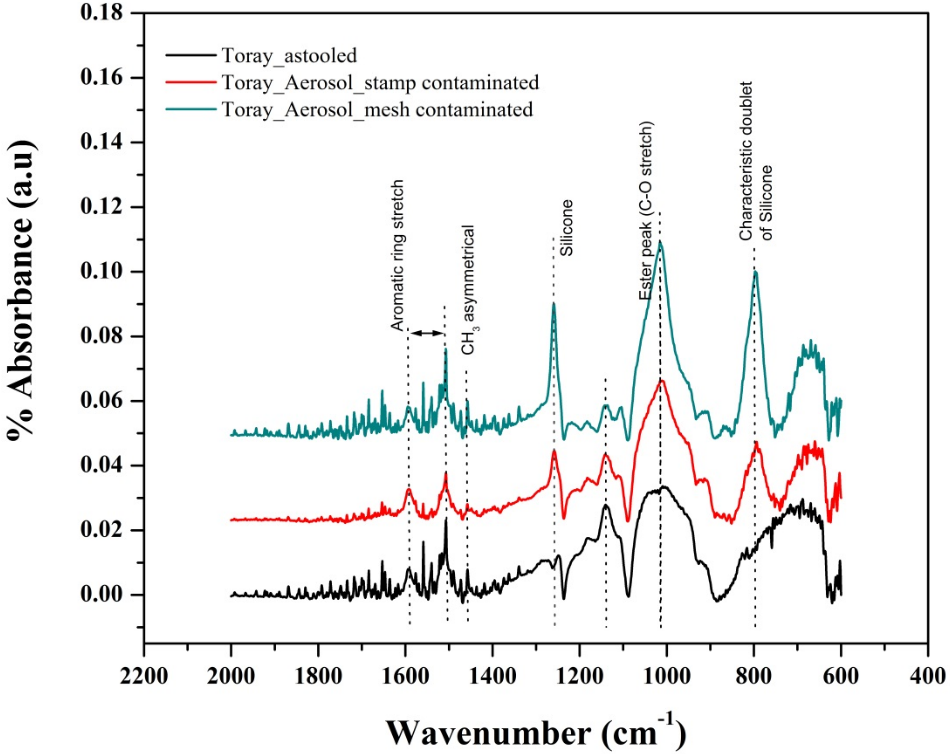
Higher contact angles for contaminated specimen when compared with baseline

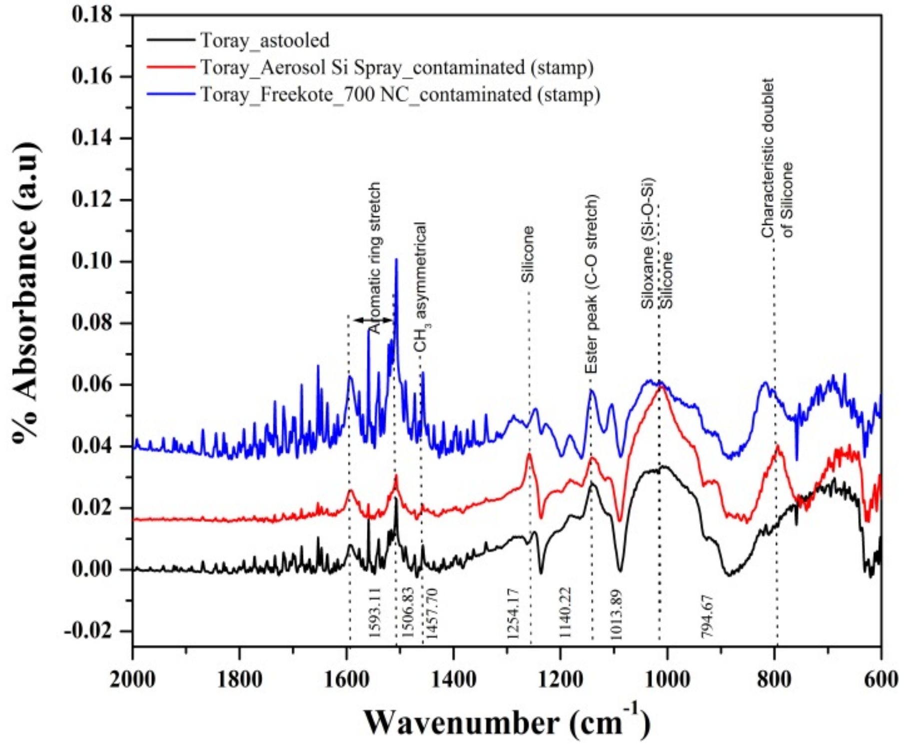
Low Surface Free Energy (SFE) => Poor Wettability

Contaminated set has low SFE when compared to baseline SFE

Surface Characterization Methods

Fourier Transformed Infrared Spectroscopy





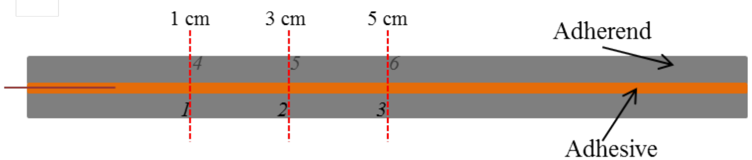
Ester (C-O stretch) peak resulting from Polyester peel ply

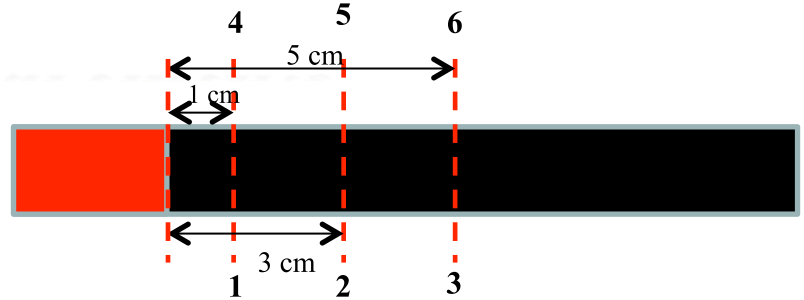
CH_3 asymmetrical resulting from prepreg (Toray)

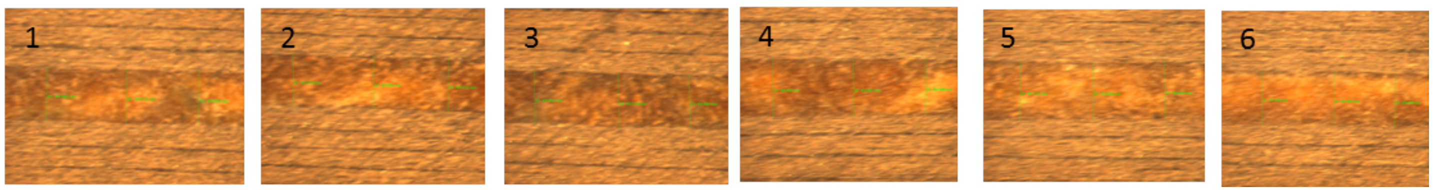
Characteristic Silicone peaks identified in Aerosol but no Silicone in Freekote

Freekote majorly comprised of heavy Naphtha

Bondline Thickness Measurements

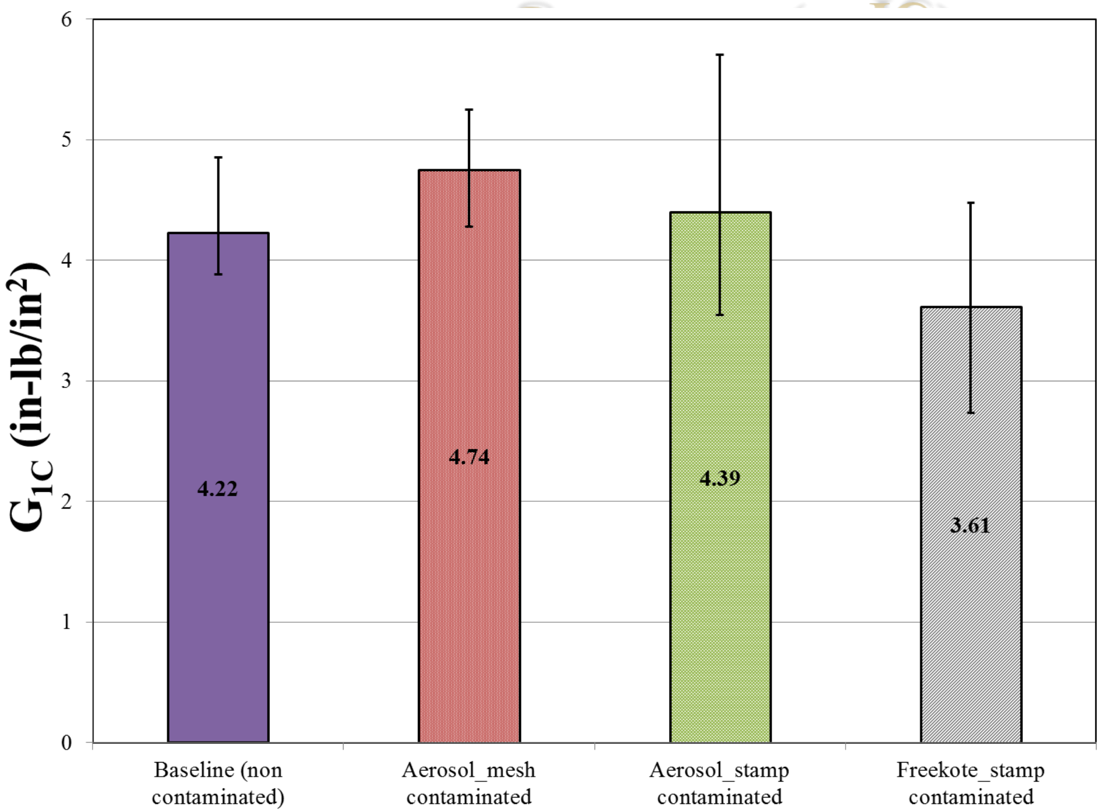






Specimen	Non Contaminated	Contaminated
Baseline	9.29 mils	12.99 mils
Environmentally Exposed	15.72 mils	13.86 mils
Fatigue	17.47 mils	13.50 mils
Combined Env Exposed+ Fatigued	11.96 mils	TBD mils

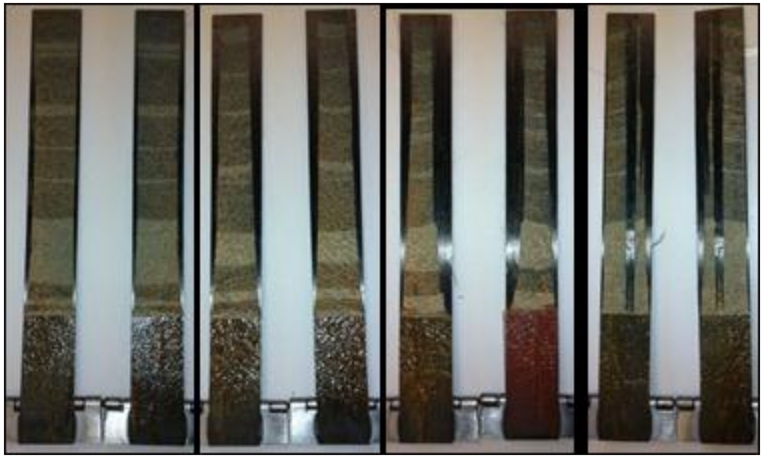
Fracture Toughness (G_{IC})



G_{IC}	*Average*	*Max*	*Min*
	(in-lb)/in²	*(in-lb)/in²*	*(in-lb)/in²*
Baseline	*4.22*	*4.85*	*3.88*
Contaminated-Aerosol (mesh)	*4.74*	*5.24*	*4.27*
Contaminated-Aerosol (stamp)	*4.39*	*5.70*	*3.54*
Contaminated-Freekote 700 NC	*3.61*	*4.47*	*2.73*

Freekote specimens shows significant reduction in fracture toughness

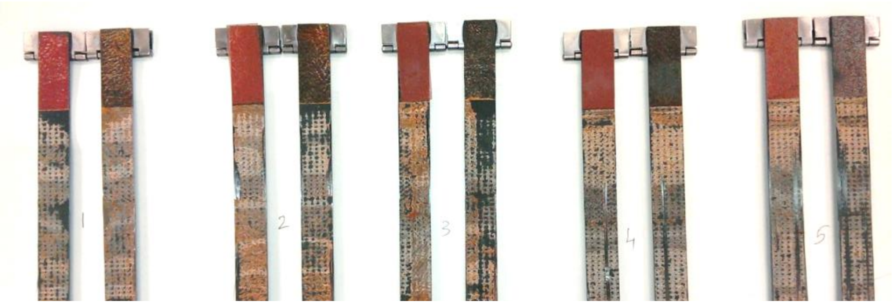
Mode of Failures



Baseline

Aerosol_mesh contaminated

Aerosol_stamp contaminated



Freekote_stamp contaminated

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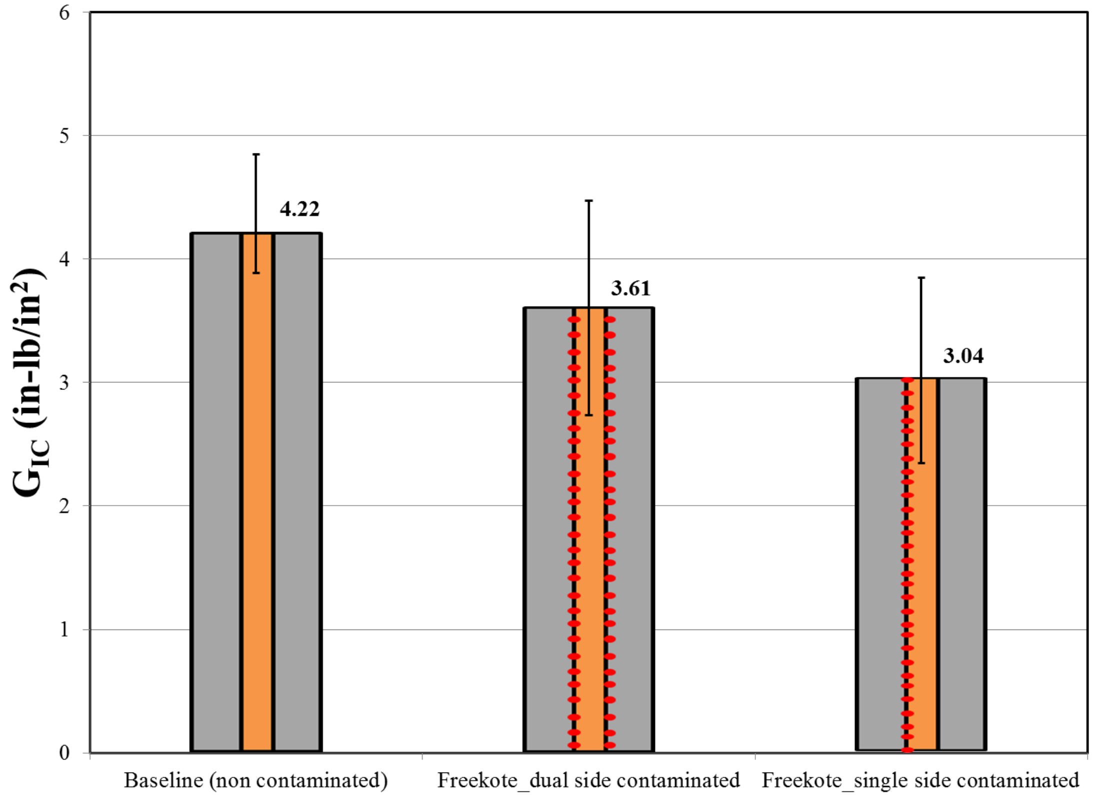
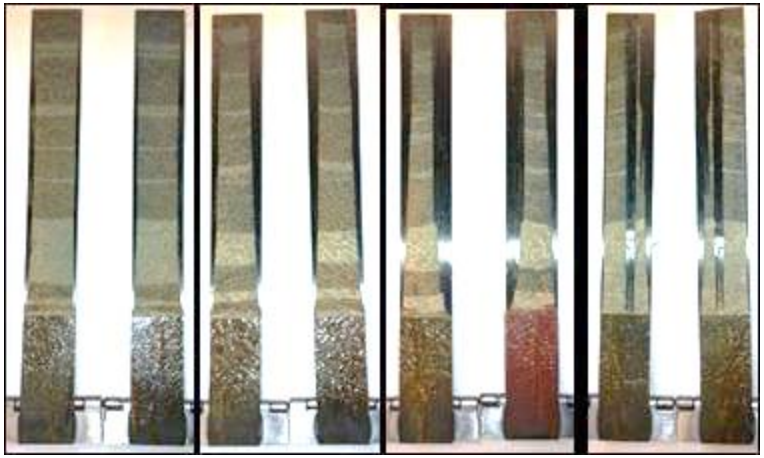
Fracture Toughness (G_{IC})



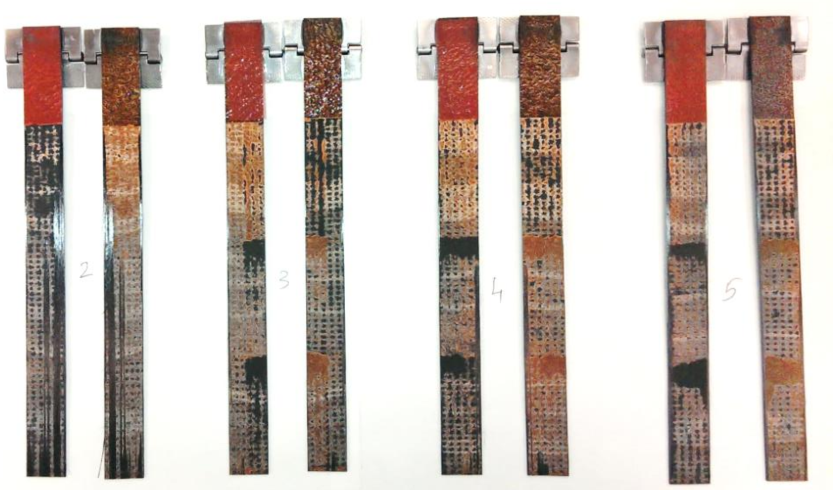
G_{IC}	*Average* (in-lb)/in²	*Max* (in-lb)/in²	*Min* (in-lb)/in²
Baseline (no contamination)	*4.22*	*4.85*	*3.88*
Freekote (dual side contaminated)	*3.61*	*4.47*	*2.73*
Freekote (single side contaminated)	*3.04*	*3.84*	*2.34*

Freekote contaminated on single side showed reduction in fracture toughness when compared to dual side

Mode of Failures



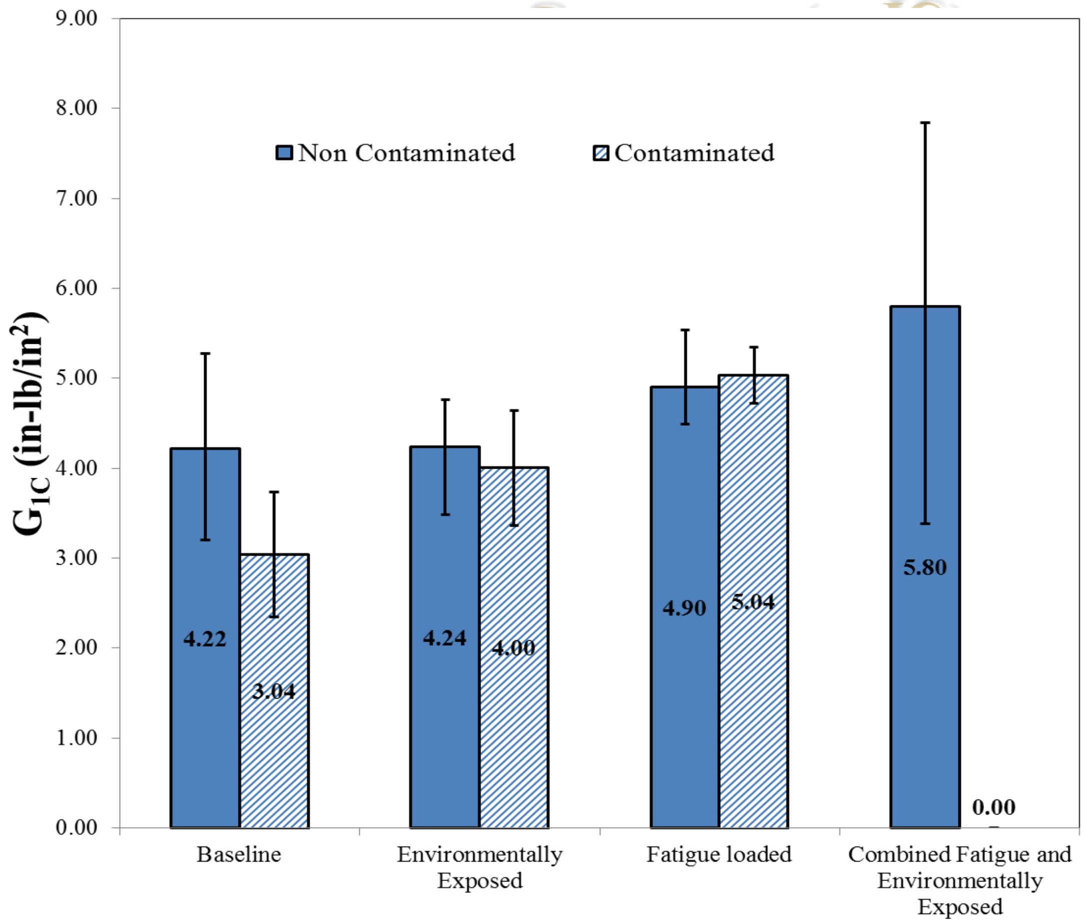
Baseline



Freekote_dual side contamination

Freekote_single side contamination

Fracture Toughness (G_{IC})



G_{IC}		Average (in-lb)/in^2	Max (in-lb)/in^2	Min (in-lb)/in^2
Baseline	*Non contaminated*	*4.22*	*5.27*	*3.20*
	Contaminated	*3.04*	*3.85*	*2.35*
Environmentally Exposed	*Non contaminated*	*4.24*	*4.76*	*3.48*
	Contaminated	*4.00*	*4.61*	*3.37*
Fatigue	*Non contaminated*	*4.90*	*5.54*	*4.49*
	Contaminated	*5.04*	*5.47*	*4.72*
Combined Fatigue and Environmentally exposure	*Non contaminated*	*5.80*	*7.84*	*3.38*
	Contaminated	*TBD*		

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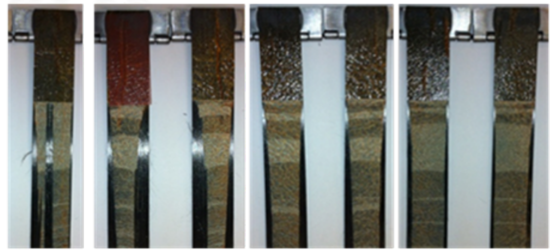
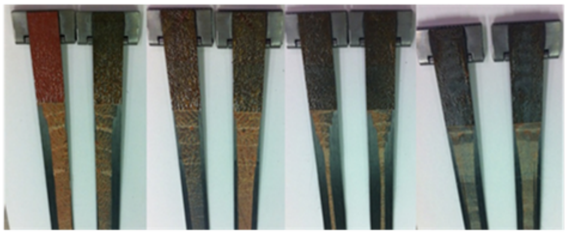
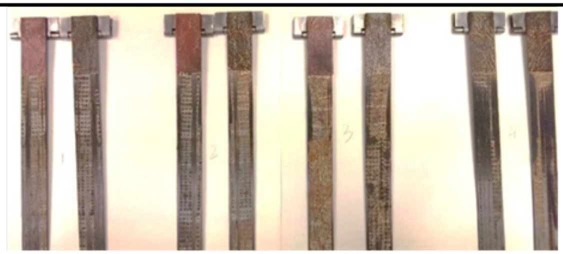
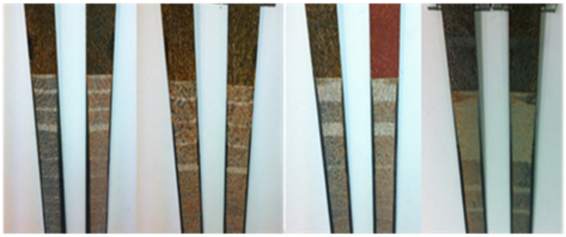
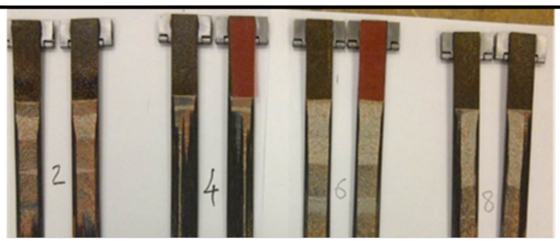
Mode of Failures

	Non Contaminated	Contaminated
A) BASELINE		
B) ENVIRONMENTALLY EXPOSED		
C) FATIGUED		
D) ENVIRONMENTAL EXPOSED AND FATIGUED		TBD

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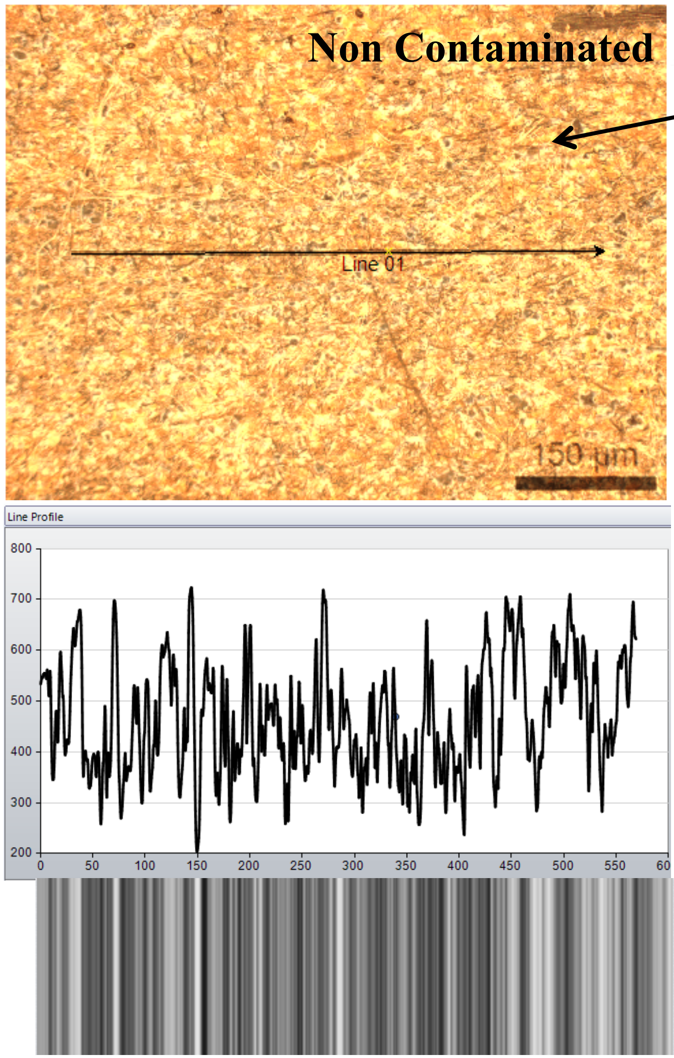
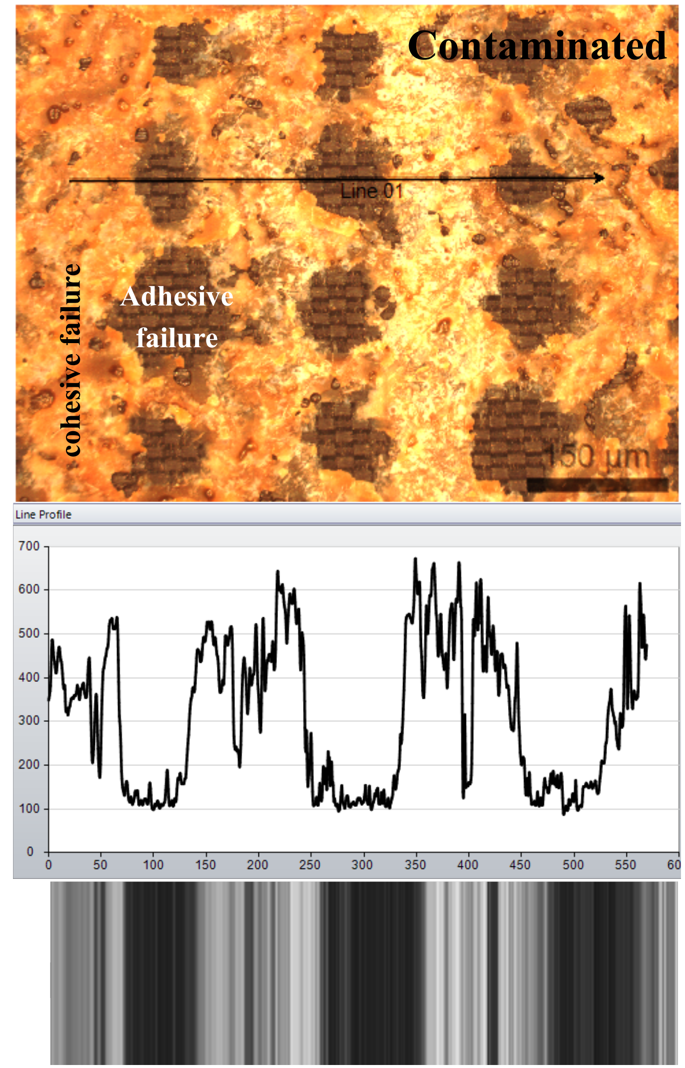
Fracture Surface Inspection



Pure cohesive failure



Line Profiles

Spectral Mapping

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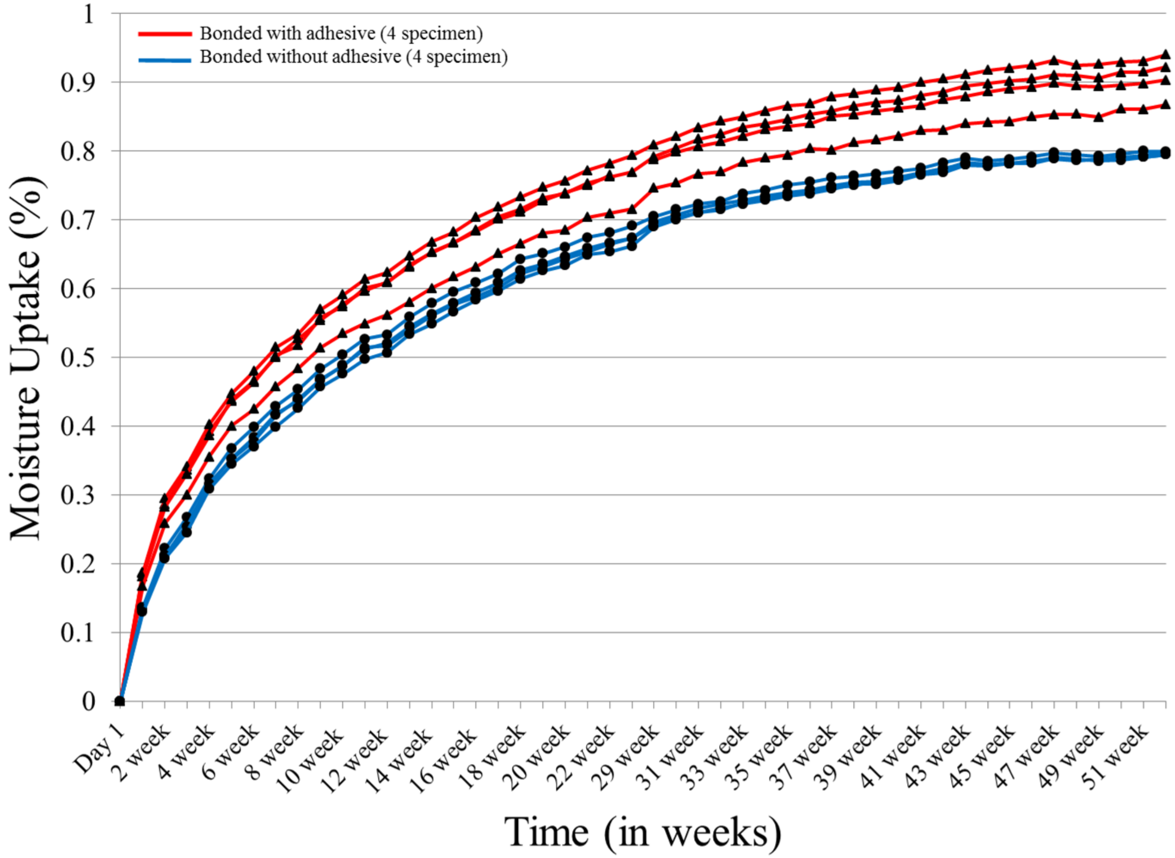
Moisture Uptake Analysis

- 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



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Conclusions

- A general contamination procedure has been developed to evaluate the effect of contamination on adhesively bonded joints.
- A durability assessment was performed of contaminated specimens by conditioning using a 3 point bending fixture for mechanical fatiguing and accelerated environmental aging using an environmental chamber.
- Surface characterization (Contact Angle and FTIR) showed a different surface chemistry on contaminated specimens when compared to pristine specimens.
- Line Profile analysis revealed the mode of failure in pristine and contaminated specimens.
- The G_{IC} values of contaminated specimens needs to be investigated for compliance values related to changes in the material properties of the laminate.
- Adhesion failure modes were observed with the Freekote contamination. Mixed cohesive and interlaminar failure modes were observed in others specimens.
- Moisture uptake curve continues to asymptotically increase - equilibrium saturation limit will continue to be monitored.

Future Work

- Characterize contaminated surfaces prior to bonding (AFM, ECS)
- Repeat conditioning on contaminated specimens (environmentally aging and mechanical fatiguing)
- Measure bond degradation of new conditioned contaminated specimens – DCB testing
- Investigate compliance of fracture toughness values.
- Quantify the contaminated areas to establish correlations between the fracture surfaces and the surface properties in accordance with bond strength.

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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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)

-Hygrothermal aging, Durability of adhesive joints in water at elevated temperatures investigated for lap shear specimen (Knight et.al. (2012), Bowditch (96))- DCB specimen have to be considered

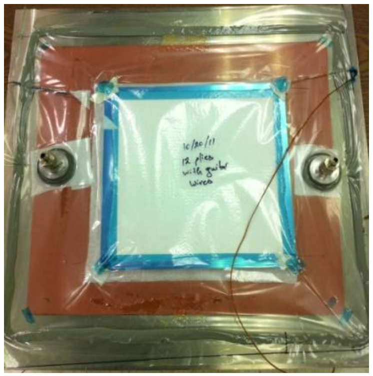
-Fatigue life of CFRP in relation to crack propagation rate have been studied (Ishii et.al (2007), Goto et. al (2003), Li et. al (2000)).

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)
- Development of unique fixtures for mechanical loading simultaneously subjecting to environmental degradation- bidirectional laminates (A. K. Singh et. al,. '06) – Unidirectional DCB specimen to be considered.

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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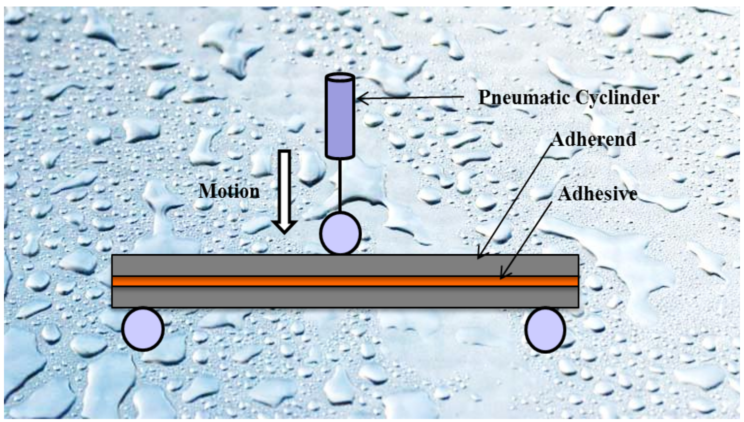
Fatigue Loading Procedure

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