Effect of Surface Contamination on Composite Bond Integrity and Durability

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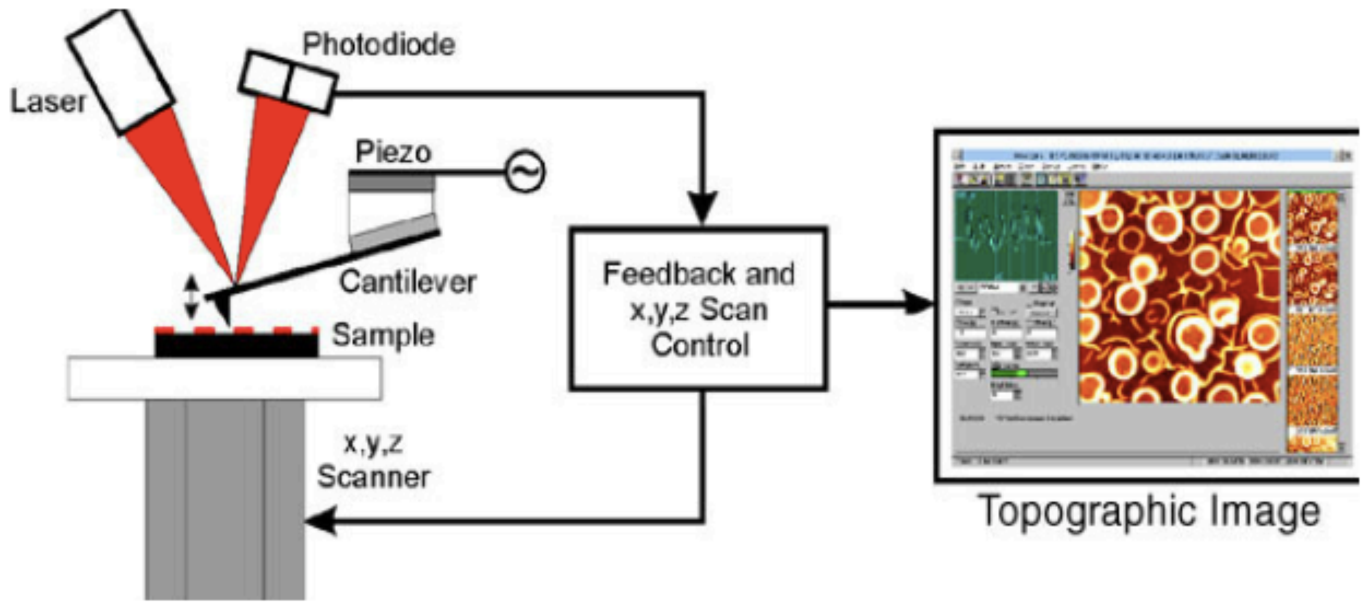
Tasks Overview

- Previous tasks:
  - Advancement of Electrochemical Sensor
  - Evaluation of Chemical Force Microscopy

- New tasks:
  - Composite Bond Integrity/Long-Term Durability Testing of Composite Bonds
  - Composite Bond Surface Characterization (Flinn – Lead)
  - Revising ASTM 3762 (Adams – Lead)

AFM Principles



Schematic representation of an atomic force microscopy (AFM) showing the force sensing cantilever.

CFM Principles

CHEMICAL FORCE MICROSCOPY 411

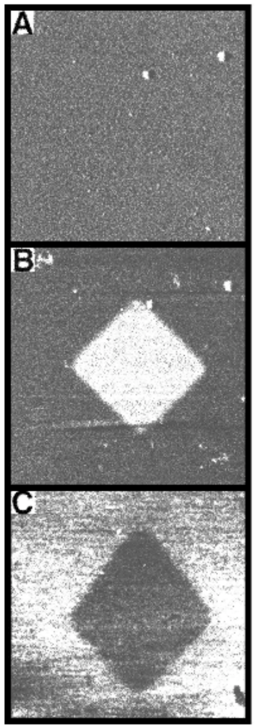


Figure 16 Force microscopy images of a photopatterned SAM sample. The 10 × 10 μm square region terminates in COOH, and the surrounding region terminates in CH_3. The images are of (*A*) topography, (*B*) friction force using a tip modified with a COOH-terminated SAM, and (*C*) friction force using a tip modified with a CH_3-terminated SAM. Light regions in (*B*) and (*C*) indicate high friction; dark regions indicate low friction (reproduced from Reference 33).

Force microscopy images of a photopatterned SAM sample. The 10 × 10 µm square region terminates in COOH, and the surrounding region terminates in CH3.

(A) Topography,

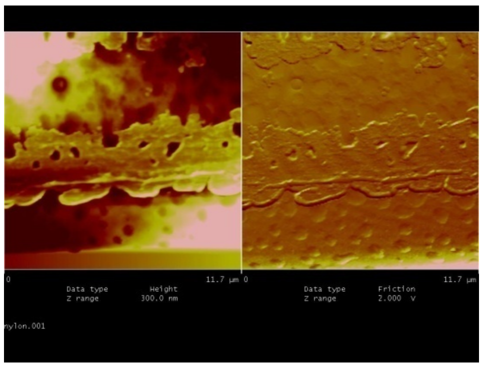
(B) friction force using a tip modified with a COOH-terminated SAM,

(C) friction force using a tip modified with a CH3-terminated SAM.

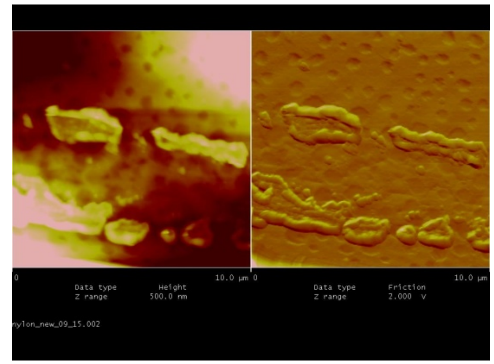
NOTE: Light regions in (B) and (C) indicate high friction; dark regions indicate low friction.

AFM/CFM Results

Nylon prepared samples

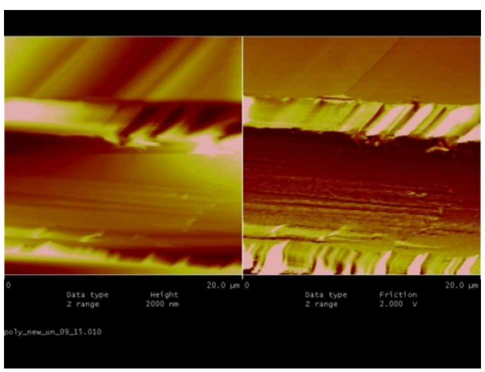


Unmodified tip

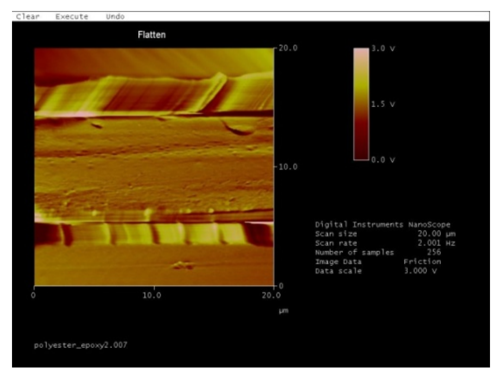


Epoxy modified tip

Polyester prepared samples

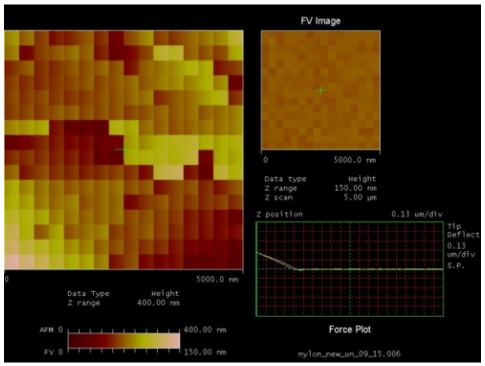


Unmodified tip



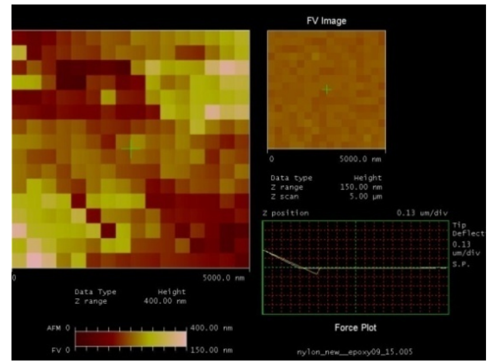
Epoxy modified tip

AFM/CFM Results - Force Volume

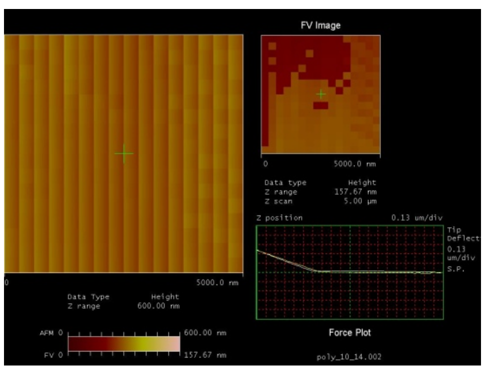


Unmodified tip
Ave Force - 11.3 nN

Nylon prepared samples

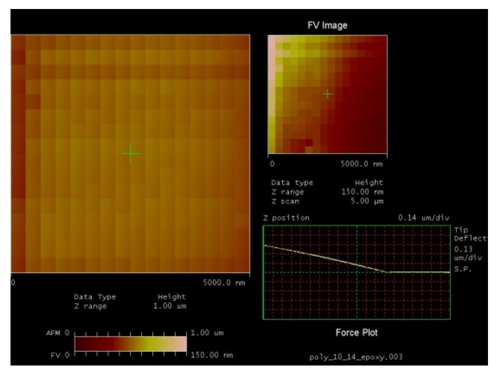


Epoxy modified tip
Ave Force - 29.5nN



Unmodified tip
Ave Force - 13.1 nN

Polyester prepared samples



Epoxy modified tip
Ave Force - 14.5nN

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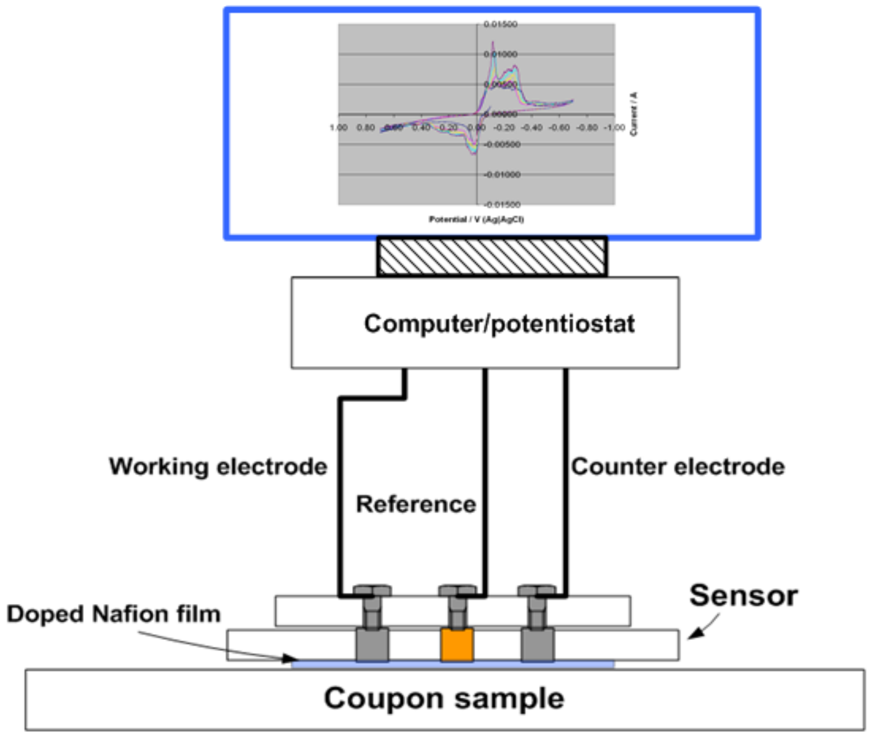
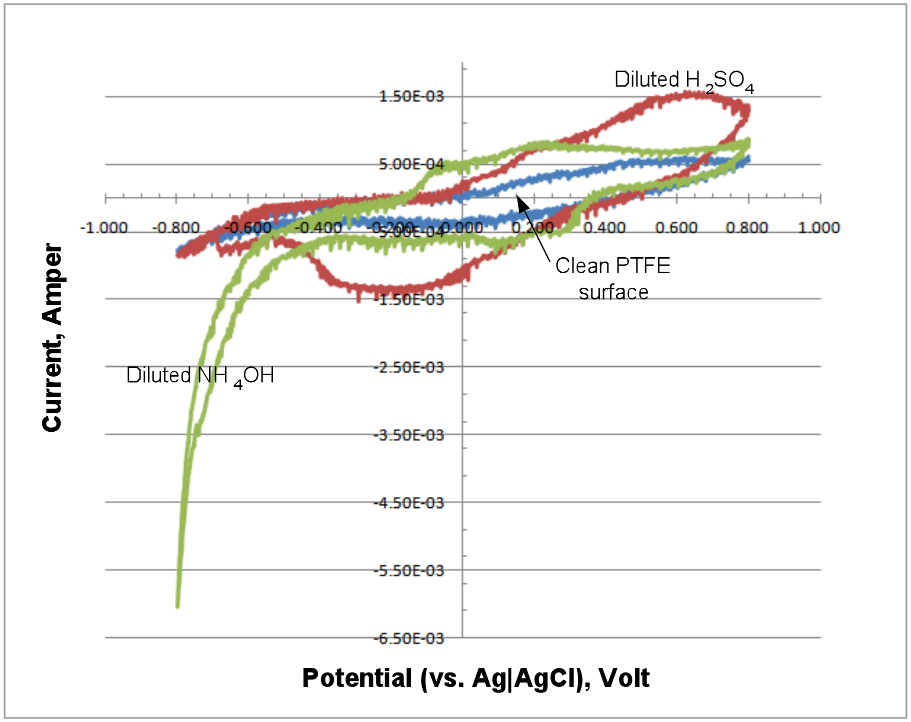
2nd Generation Solid-State Electrochemical Sensor-Experimental Setup



- Working electrode contains mediators (NaI/I_2) that can undergo redox reactions with the surface contaminants.
- The rate of the reactions can be measured with the current at certain potentials.
- For clean surfaces, the current is typically low while for a contaminated surface the current is higher.
- The reactivity can also be measured with the electrochemical impedance spectroscopy (EIS) method.
- In the EIS method, usually a high charge transfer resistance corresponds to a low reactivity while a low charge transfer resistance corresponds to a high resistivity.

Electrochemical Sensor Improvements

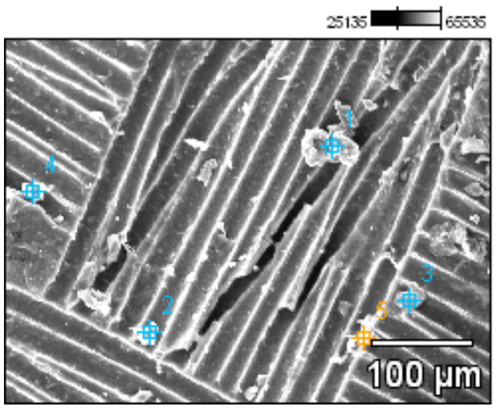
- The NaI/I_2 mediators that were used in the sensors were replaced with polyaniline - conducting polymer that can undergo highly reversible redox reactions at the active sites in the polymer.
- Provides a more sensitive and reusable sensor.
- Unlike NaI/I_2 mediators that may diffuse out and may contaminate the surface under inspection, the polymeric active material has little chance to diffuse out of the electrode.
- The use of the polymeric electrode may mitigate the contamination problem with repeated use.



SEM/EDAX

Bombardier Contaminated Samples

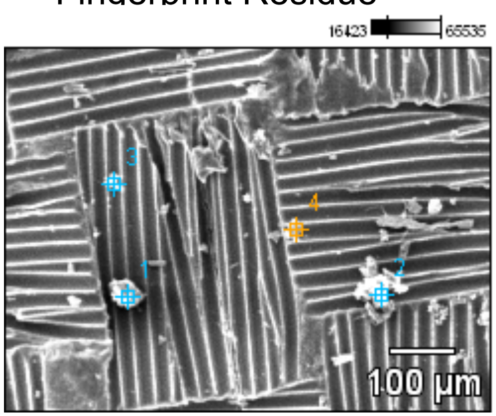
Breather Cloth



	C-K	N-K	O-K	F-K	Na-K	Al-K	Cl-K	K-K	Nb-L	Au-L
BC(2)_pt1	56.74		6.39	0.13	1.31	0.21	2.65	0.82	0.00	31.74
BC(2)_pt2	46.21	17.30	15.56	1.23		0.33			0.00	19.37
BC(2)_pt3	50.45	17.59	14.01	1.18		0.15			4.92	11.70
BC(2)_pt4	66.00		9.84	4.31		1.78			0.00	18.07

Point data

Fingerprint Residue



Element Line	Net Counts	K-Ratio	Weight %	Weight % Error	Atom %	Atom % Error
C K	260	0.71	67.02	+/- 3.61	78.75	+/- 4.24
O K	63	0.07	18.45	+/- 3.22	16.27	+/- 2.84
F K	16	0.02	4.25	+/- 2.39	3.15	+/- 1.77
Br K	18	0.00	---	---	---	---
Br L	158	0.21	10.29	+/- 1.89	1.82	+/- 0.33
Br M	0	0.00	---	---	---	---
Total			100.00		100.00	

Area data

EDAX vs. EIS Data

Sample	[O] wt%	[Al] wt%	[K] wt%	[Si] wt%	[Na] wt %	[S] wt%	[Fe] wt%	[Zn] wt%	Polarization Impedance (ohm)
Pristine	13.63	3.89	0	0	0	0	0	0	2.0×10^6
Cleanser HFP	15.89	6.47	0	0	0	0	0	1.53	1.8×10^5
UV dye	13.70	0.60	0.28	0	1.33	0	0	0	6.0×10^5
Ultrasonic Coupling gel	36.45	5.67	8.03	0	0	0	0	0	6.0×10^5
Silicone rubber glove residue	9.05	8.28	0	0	0	22.89	0	0	1.8×10^6
Solution from a marker	18.43	2.56	0	0	0	0	0	0	8.0×10^5
Tape Residue	11.28	3.04	0	0	0	0	0	0	1.7×10^6
Soda	26.57	0.57	0	0	0	0	12.16	0	6.5×10^5
Coffee	15.73	0.93	2.07	0	0	0	0	0	6.0×10^5
Protective Cream	6.39	0.13	0.82	0	1.31	0	0	0	1.2×10^3
Dust	18.54	5.80	3.47	2.07	1.64	2.47	8.90	0	2.0×10^4
Fingerprint Residue	13.1	3.25	0	0	0	5.56	0	0	2.5×10^4
Cleanser MEK	31.50	3.80	0	0	0	0	0	0	4.9×10^4
Breather Cloth	18.41	0.42	0	0	0	0	0	0	2.7×10^4

Composite Bond Integrity/Long-Term Durability
Testing of Composite Bonds

Adhesive Bonding Group:

University of Washington, University of Utah, Florida International University, Boeing

Background:

- A significant amount of research has been conducted and documented regarding surface preparation on the strength and durability of adhesively composite joints.
- Surface analysis techniques that can improve the quality management system for bonding continue to be of interest to the industry and the FAA.
- Identifying key process parameters and their effects on short and long term bond quality has led to an in interest in demonstrating the applicability of quality control methods including surface analysis techniques that could be integrated into a quality management system.

Tasks:

1) Composite Bond Surface Characterization (UW)
2) Composite Bond Integrity/Long-Term Durability Testing of Composite Bonds (FIU)
3) Revision of ASTM D3762 Metal Wedge Crack Durability Testing (UU)

Motivation Task 2:

Past research has focused on verifying the required quality of an initial composite bonded system to be assured good initial bond strength. Additionally, the effects of contaminants has also been established on the initial bond strength.

What is less understood is the effects of contaminants on durability.

Specific Requirements:

- 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.

Procedure:

- Conduct literature review for areas needed.
- Develop approach/test plan.
- Revise plan based on input from team members (FIU,UW, UU), industry (Boeing, Bombardier, AARCorp, Exponent, etc), FAA.

Issues to be discussed/evaluated

Initial bond strength characterization

- Lap shear test
- Peel test
- **DCB test**

Durability testing

- Wedge test
- Moving wedge test
- Variation of **DCB** and peel tests

Contamination of composite specimens

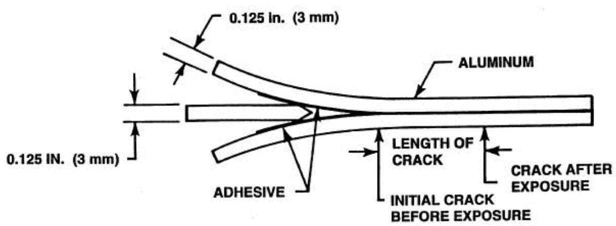
- Various contaminants (silicone, fluorine)
- Procedures/level of contamination
- Characterization of contamination

Manufacture and bonding of composite specimens

- Ply orientations
- Thickness, number of plies
- Surface prep
- Type of adhesive (**film** vs paste)
- Bondline thickness
- Bondline control (**scrim,** glass beads)

Composite Bond Integrity/Long-Term Durability
Testing of Composite Bonds

Wedge Test

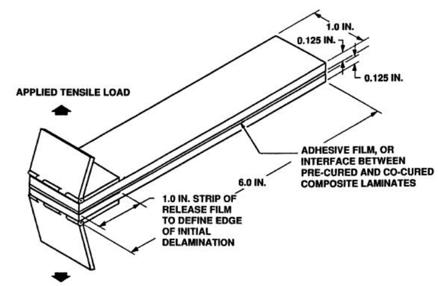


Hart-Smith, International Journal of Adhesion & Adhesives (1999)

“The reason that the wedge test is suited to evaluation of bond durability is that the adhesive and the interface are placed under extremely high tensile stresses. The initial crack arrests when the tensile stresses are just below tensile ultimate for the adhesive. That leaves the interface under extreme stresses so any degradation of the interface, such as by hydration, will result in interfacial failure.” - Davis and McGregor, 6/2010.

“The wedge-crack test is known not to work for composite laminates made from woven fabric layers, because of the tendency of any initial crack to be diverted into (bundles of) 90° fibers on the surface adjacent to the adhesive layer.” Hart-Smith , 1999.

Double Cantilever Beam Test



The DCB can be use for initial bond strength characterization and with modifications can provide characterization of long-term durability. DCB provides quantitative and qualitative information including mode of failure and a measure of the strain energy release rate.

For durability testing, the DCB requires use of an test machine and an environmental chamber for each coupon tested. With the Wedge test, multiple coupons can be tested at one time.

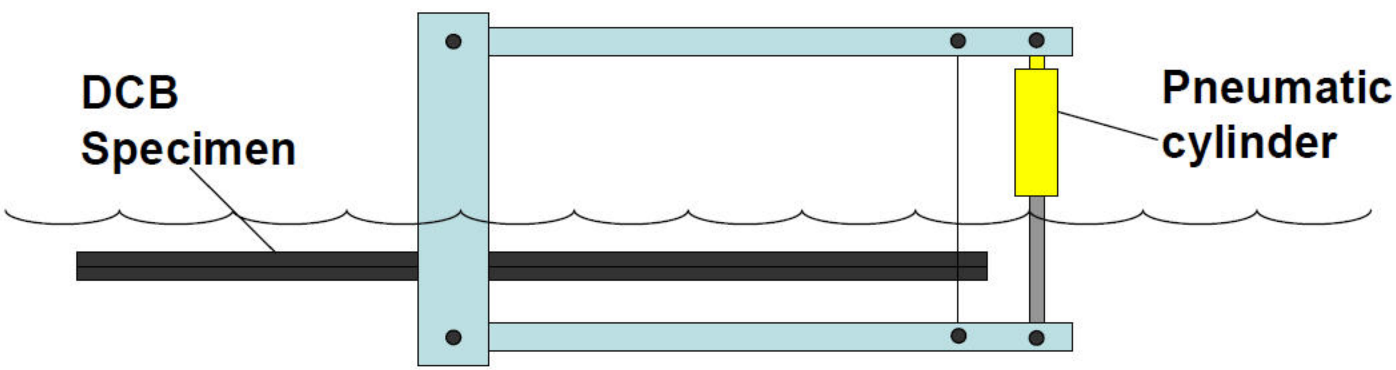
Composite Bond Integrity/Long-Term Durability
Testing of Composite Bonds

Durability Testing Options:

Compact Pneumatic Creep Frames

Adhesive bond durability testing by WSU – Lloyd Smith

- Develop test methods to accelerate adhesive degradation
- Accelerated degradation testing of adhesive bonds combining moisture, temperature and stress



Future Work

- Complete literature review
- Establish test plan
  - Initial bond strength characterization
  - Long term durability
  - Procedures/levels for contamination of coupons
- Work with team members, industry partners, and FAA to agree on path forward