Identification and Validation of Analytical Chemistry Methods for Detecting Composite Surface Contamination and Moisture

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The Joint Advanced Materials and Structures Center of Excellence

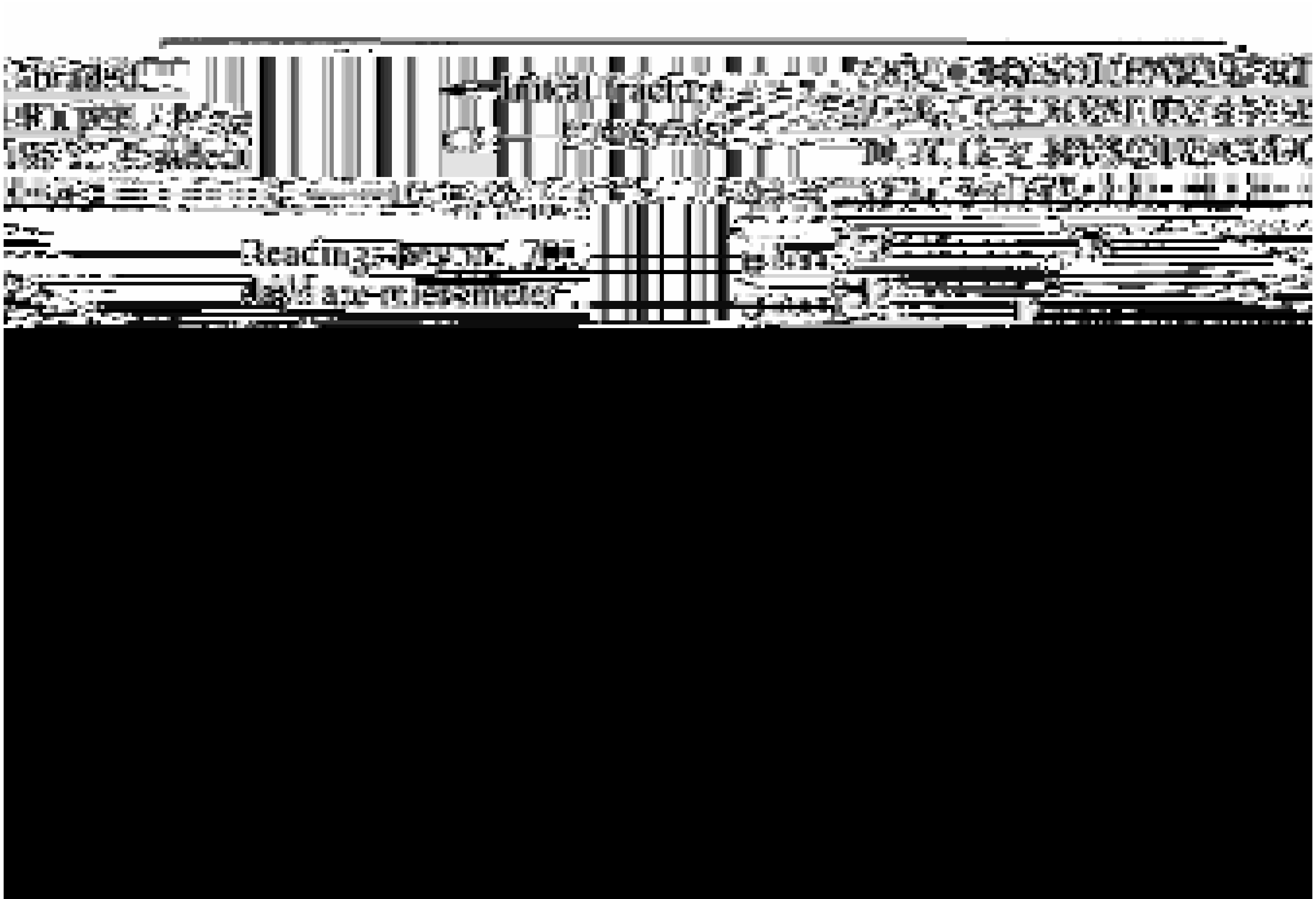
Introduction

- Motivation and Key Issues
  - Adhesive bonding has been used in the manufacture and repair as a direct competition to mechanical fastening.
  - Adherend surface preparation is a critical issue to the structural integrity and durability of bonded structures.
- Objective
  - benchmark surface preparation quality assurance methods
  - identify and validate definitive analytical chemistry methods to provide sufficient in-field quality assurance.
- Approach
  - Literature review and analysis
  - Surface chemistry analysis
  - Electrochemical sensor development
  - Experimental validation

Main Results

- Literature database, complete
- Summary of literature review
  - Surface treatment, complete
  - Surface chemistry analyses, complete
- An electrochemical sensor for surface chemistry analysis, testing in progress
- Carbon nano-tube sensor for humidity sensing, testing in progress
- AFM/SEM study of surface-contamination (peel-plies, etc),testing in progress

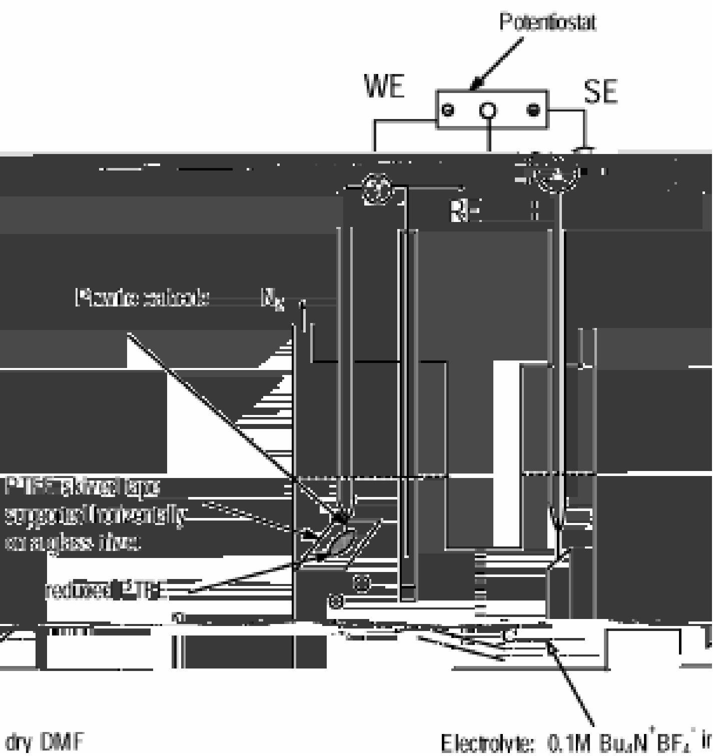
Literature Review: Effect of Various Surface Pretreatment Method

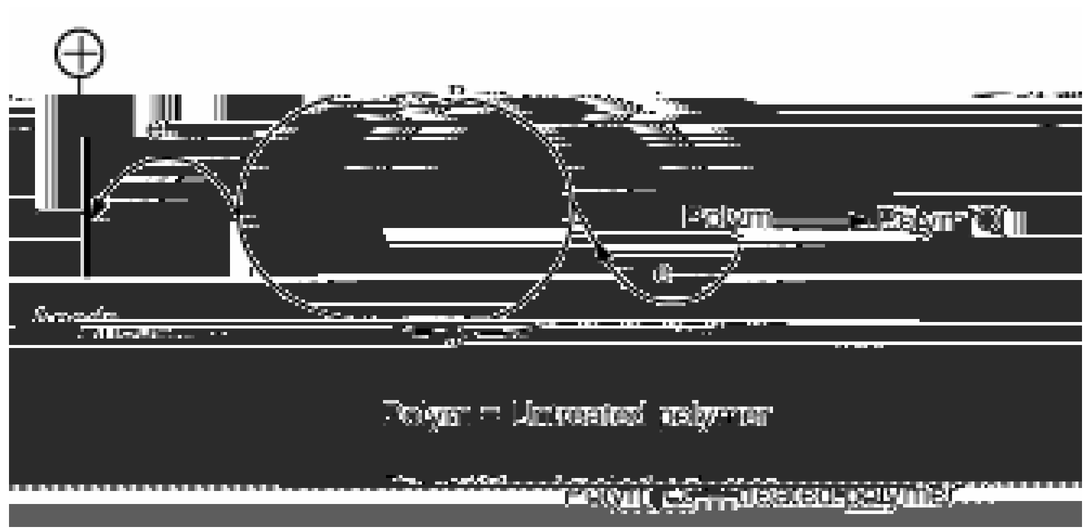


Literature Review: Concentration of Oxygen versus Strength

Polymer	Treatment	Surface composition (at%) C	Surface composition (at%) O	Failure load/N
HDPE	No treatment	100.0	0.0	400
	2.1 V, Pt edge, 50 passes	95.5	4.5	1330
	2.4 V, Pt edge, 50 passes	96.2	3.8	1320
	2.9 V, Pt disc, 5 min	92.4	7.6	1110
PP	No treatment	100.0	0.0	0
	3.25 M nitric acid, 60 s	—	—	267
	2.1 V, Pt edge, 50 passes	[illegible]	[illegible]	[illegible]
	2.4 V, Pt edge, 50 passes	93.1	6.9	2560
	2.9 V, Pt edge, 50 passes ($H_2SO_4^-$)	100	0	50
	2.9 V, Pt disc, 300 s, not touching	—	—	270
	2.9 V, Pt disc, 300 s, fat removed	[illegible]	[illegible]	[illegible]
SBS	No treatment	100.0	0.0	—
	2.5 V, Pt edge, 50 passes	83.6	[illegible]	[illegible]
PS	No treatment	100.0	0.0	550
	2.9 V, Pt disc, 300 s	94.5	5.5	670

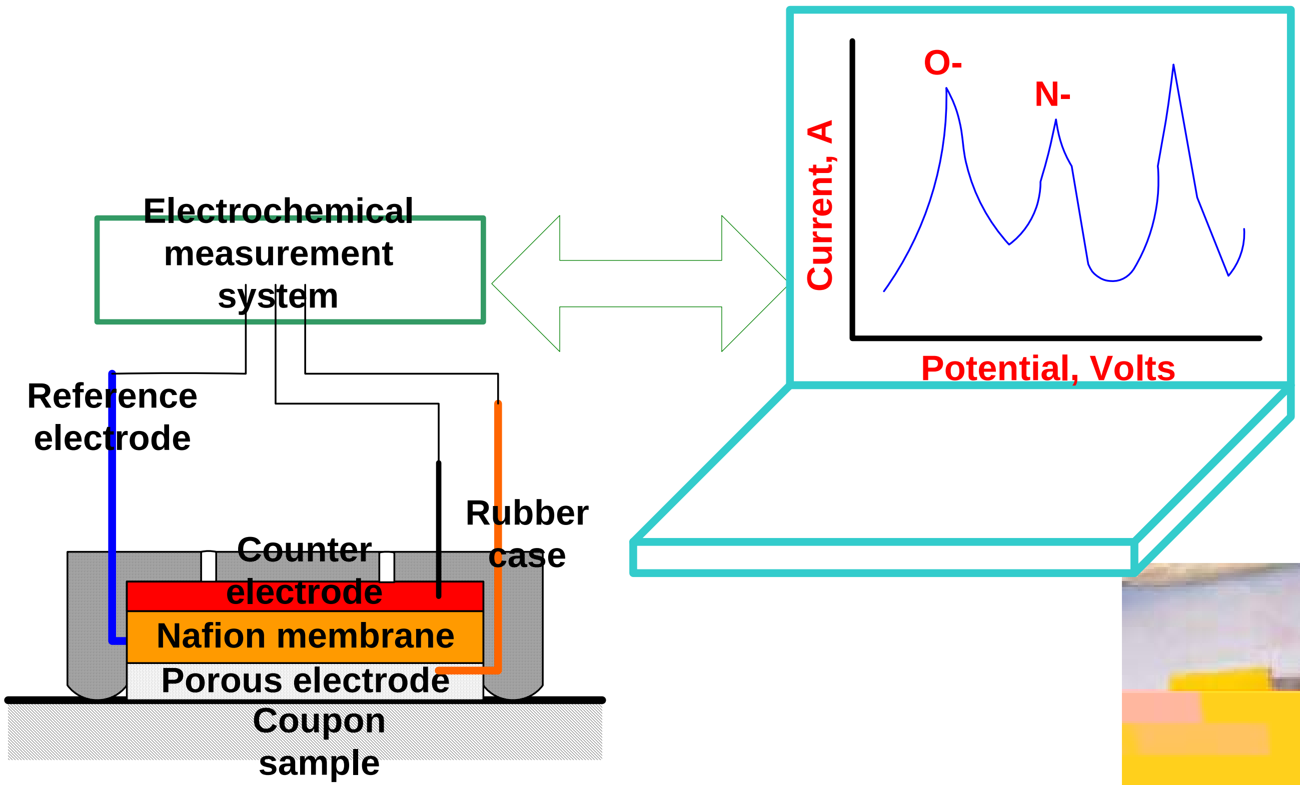
Electrochemical Sensor





D.M. Brewis, R.H. Dahm / International Journal of Adhesion & Adhesives 21 (2001) 397–409

Solid-State Electrochemical Sensor



Candidate Mediator Couples

- Ag(II)/Ag(I) E^0=1.98 V
- Ag+/Ag E^0=0.8 V

- **Ce(IV)/Ce(III) E^0=1.72 V**

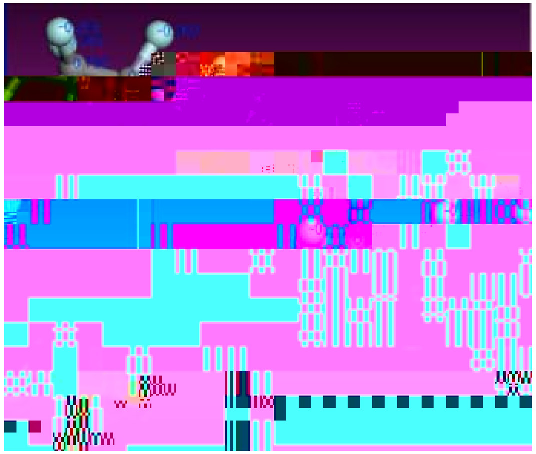
Solid-State Electrolyte

Nafion + Nafion Resin
(Sulfonated tetrafluorethylene copolymer)

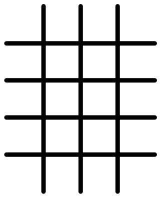
Extremely resistant to chemical attack

Proton conductor

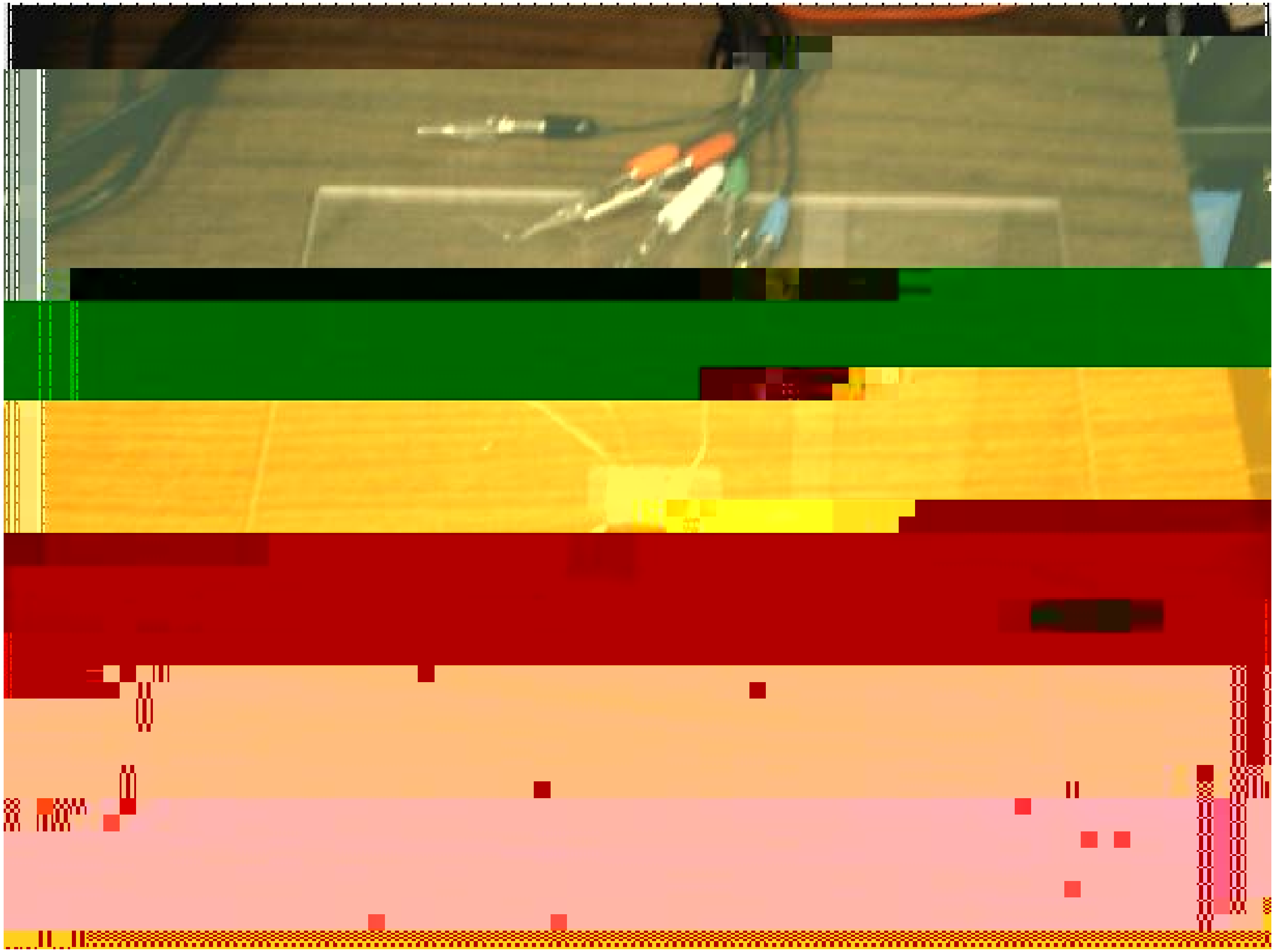
Strong proton donor and free electron acceptor-superacid catalysts
(neutral pH)



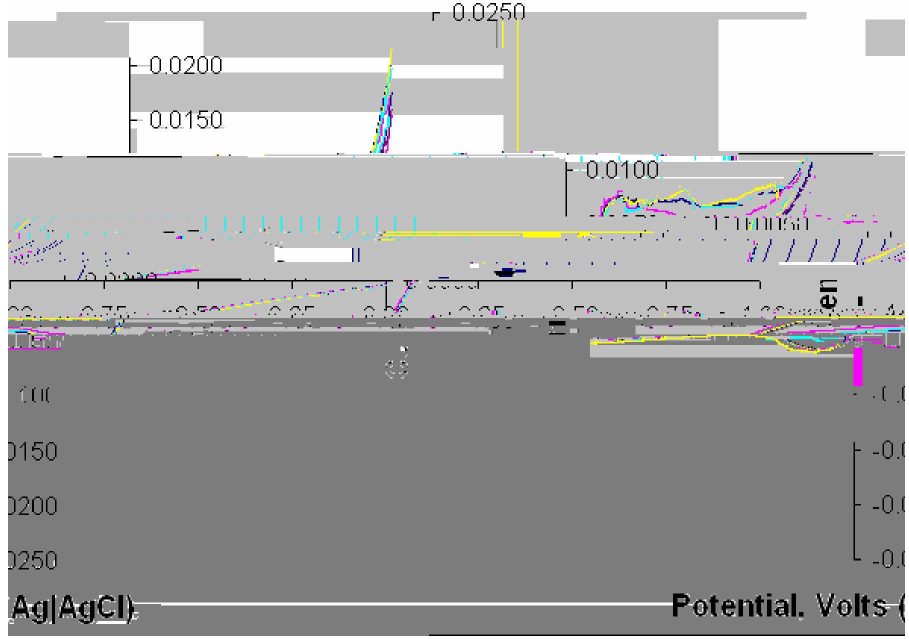
Minimum oxidation and reduction of water

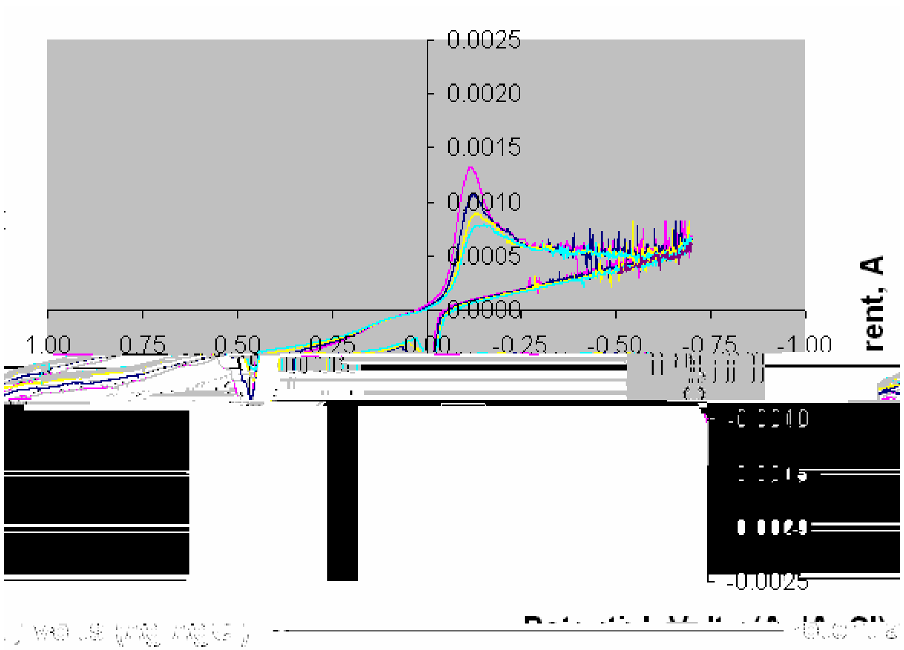


Solid-state Electrochemical Sensor

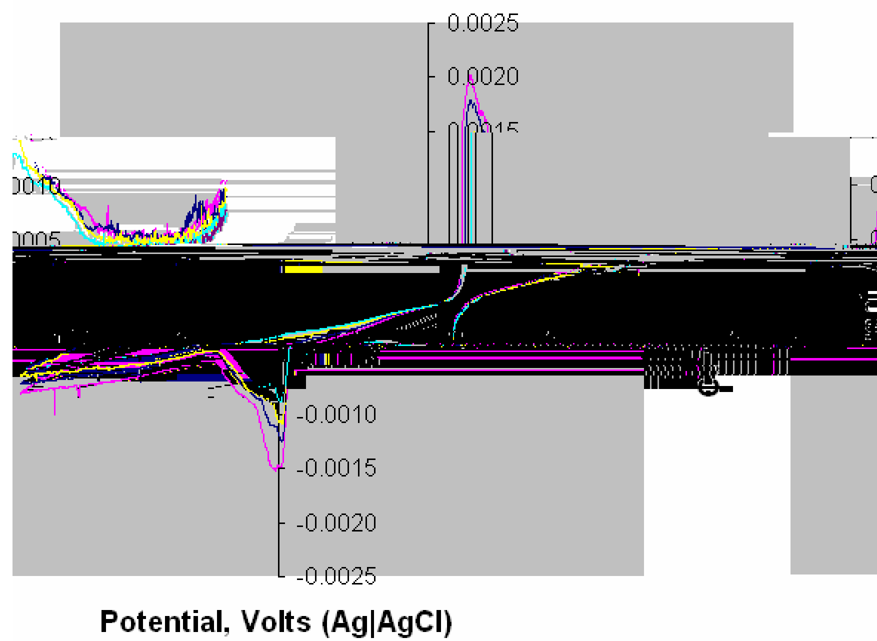


Treated with sulfuric acid



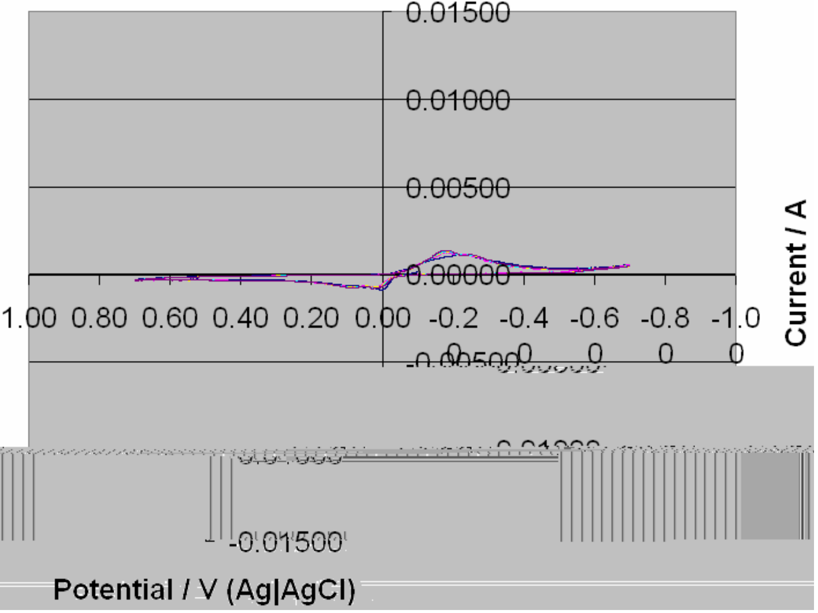


Original acrylic plastic surface

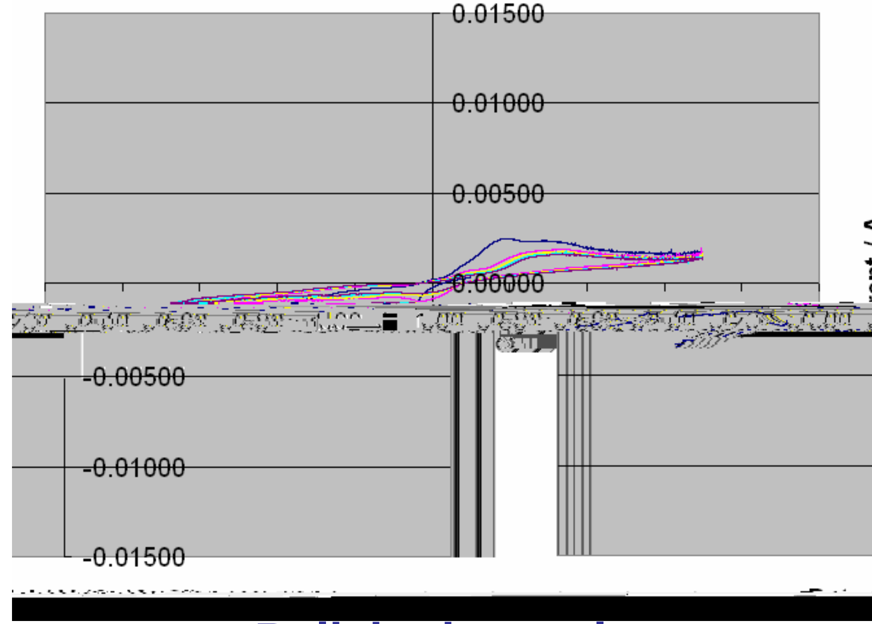


Polished acrylic plastic surface

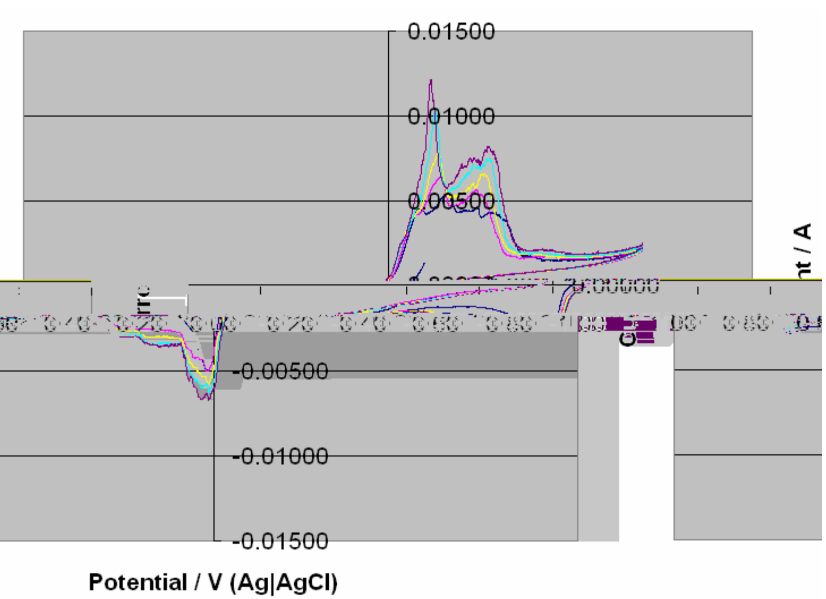
Original acrylic plastic surface



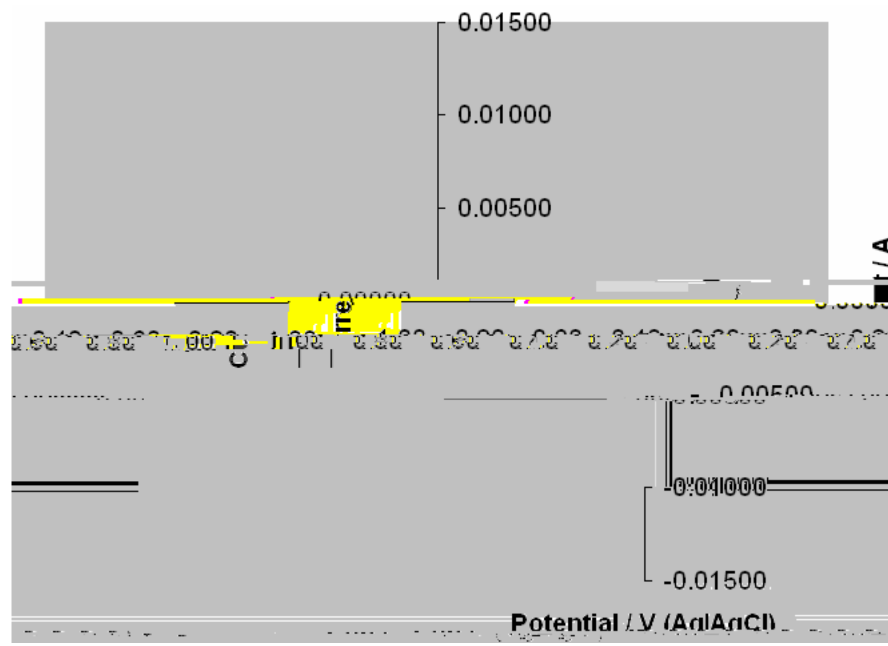
Original peel ply sample



Polished sample

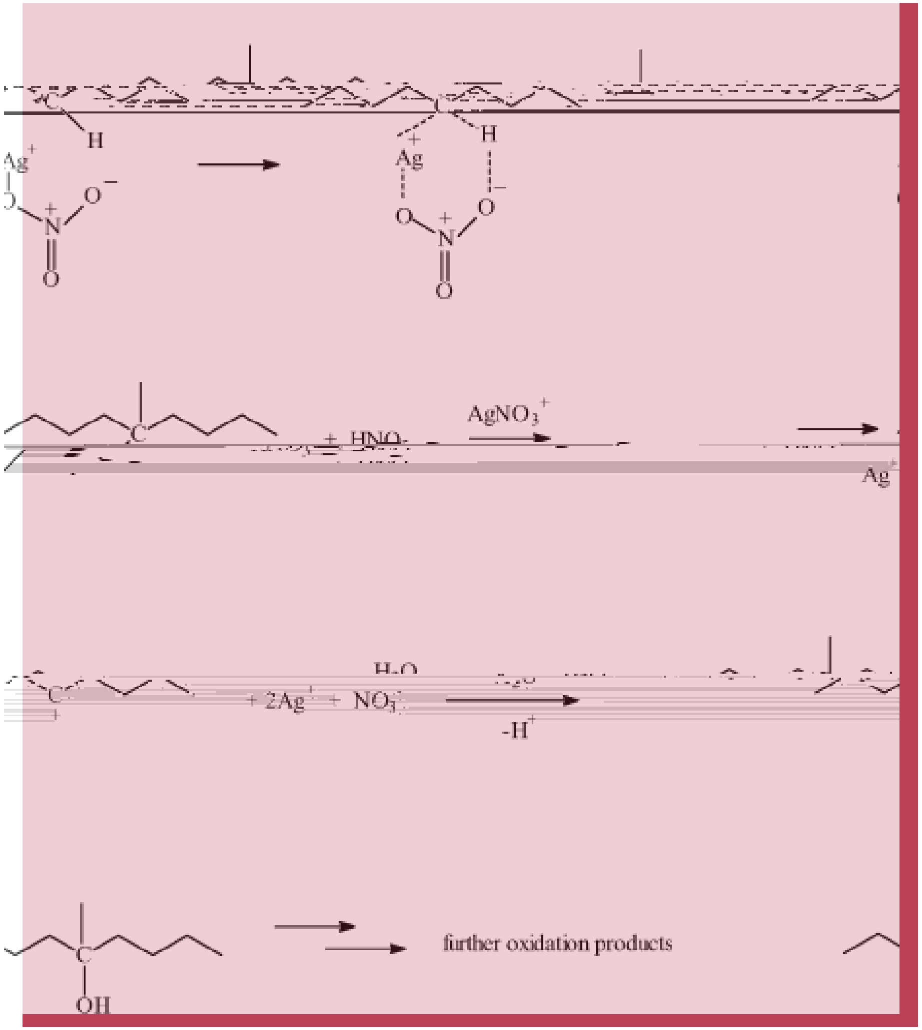


Sulfuric acid treated sample

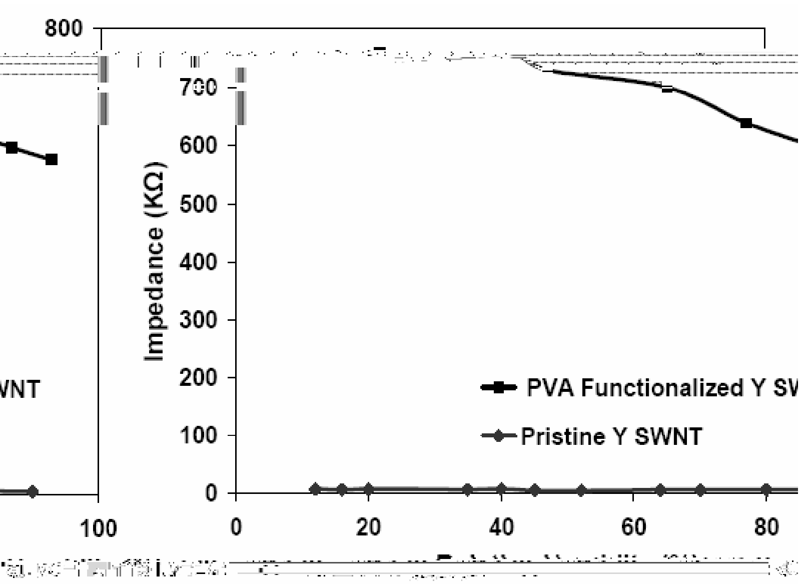


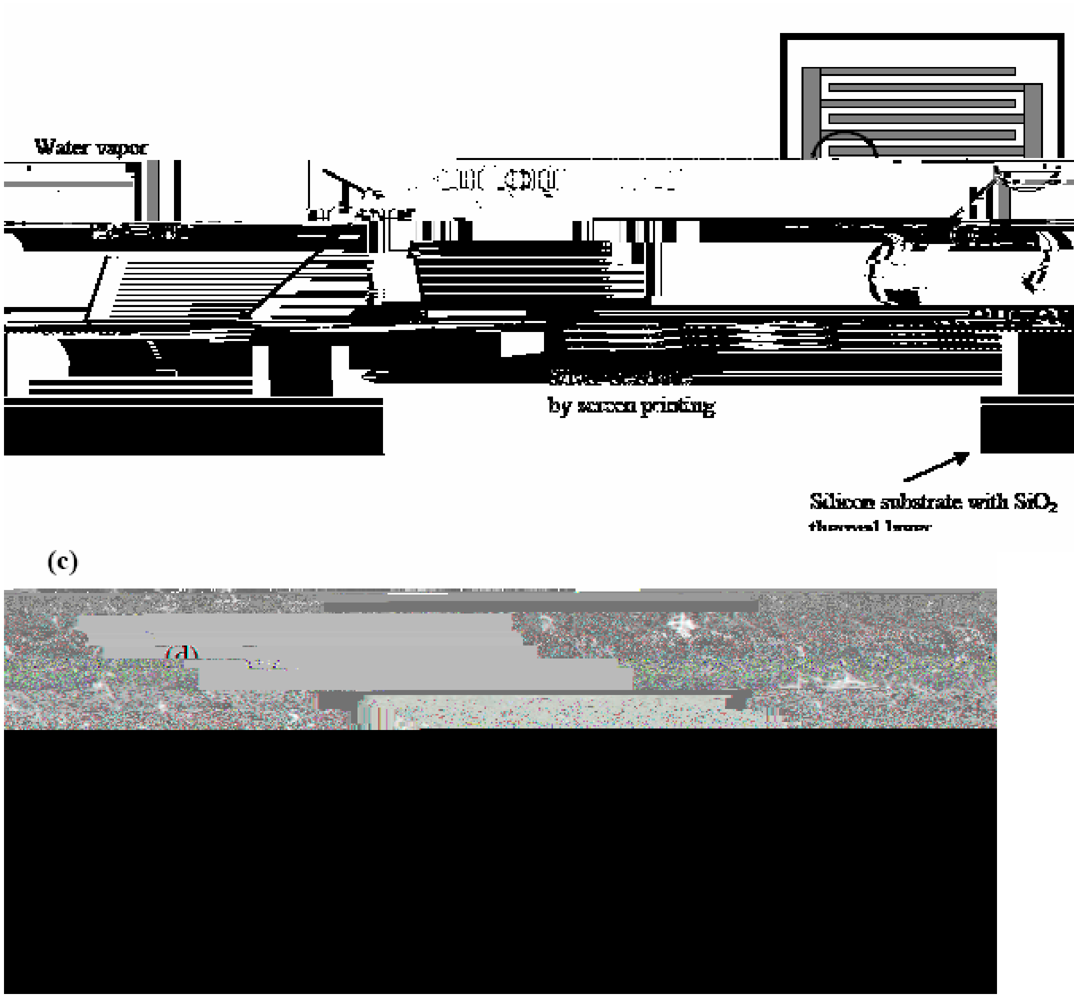
In air

Reaction Mechanisms

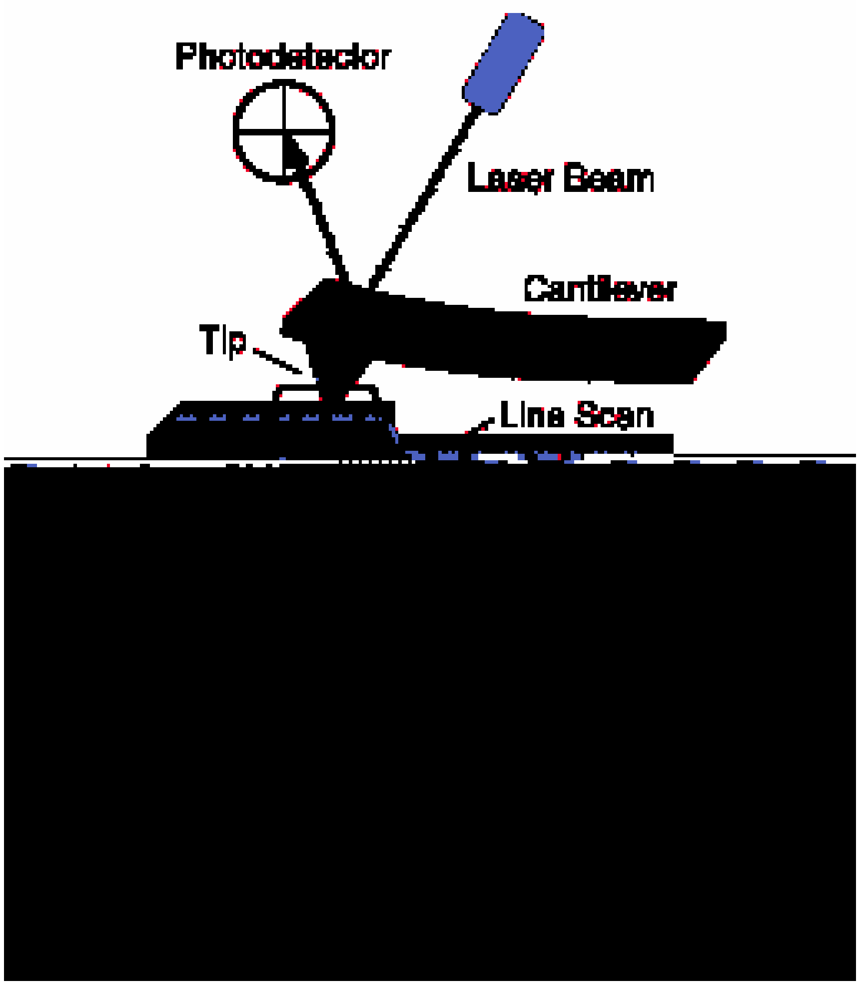


Carbon Nanotube Based Humidity Sensor

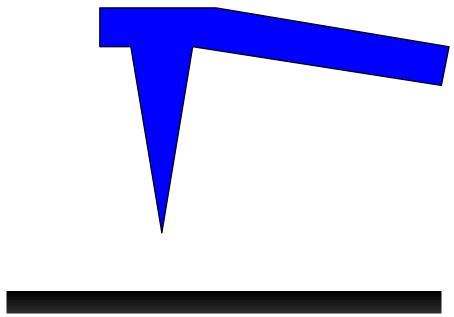




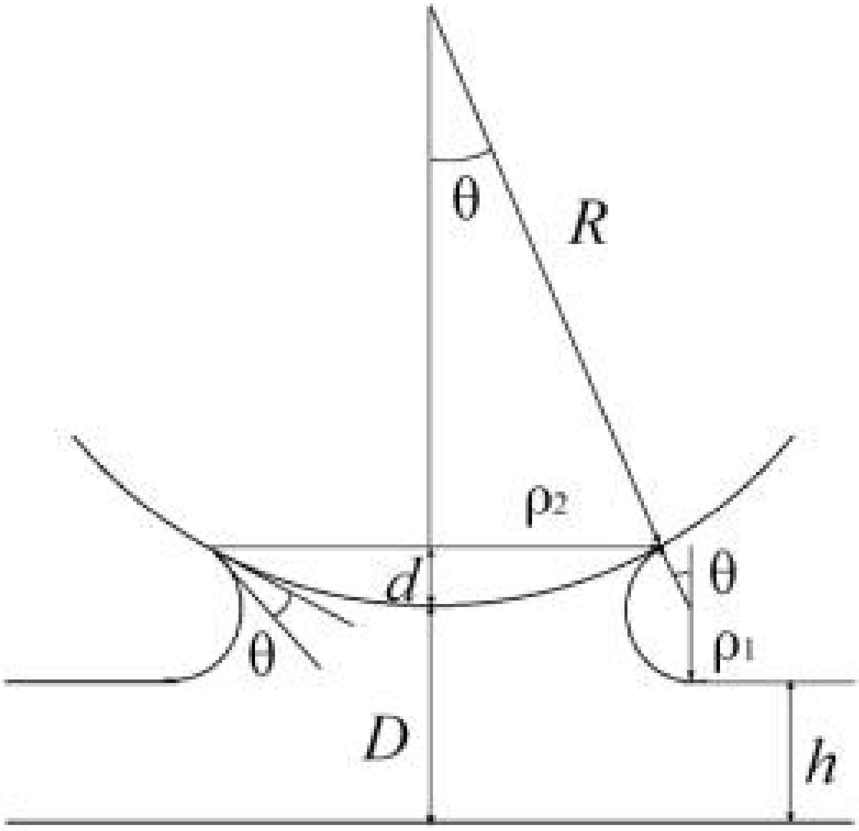
Atomic Force Microscopy (AFM) Study



AFM Force Spectroscopy



Evaluation of Surface Adhesion



$$\sigma = \frac{F_{\max.attraction}}{4\pi R\cos\theta}$$

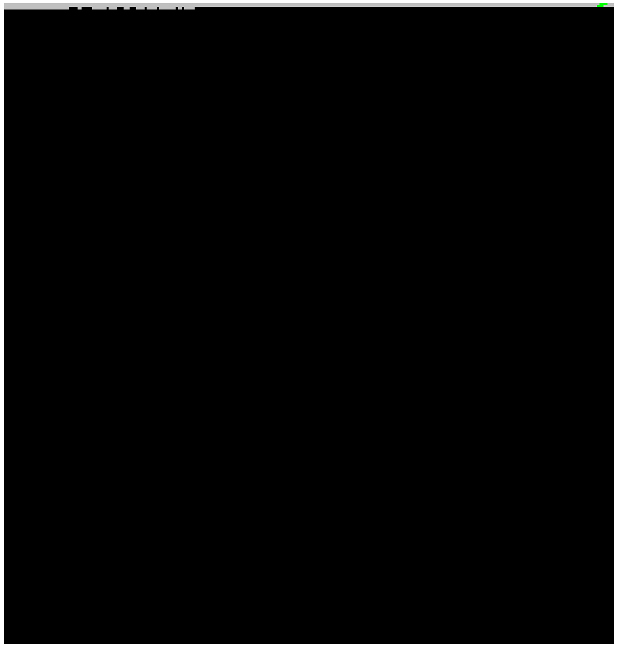
Previous SEM and XPS Results on Peel Ply Surfaces

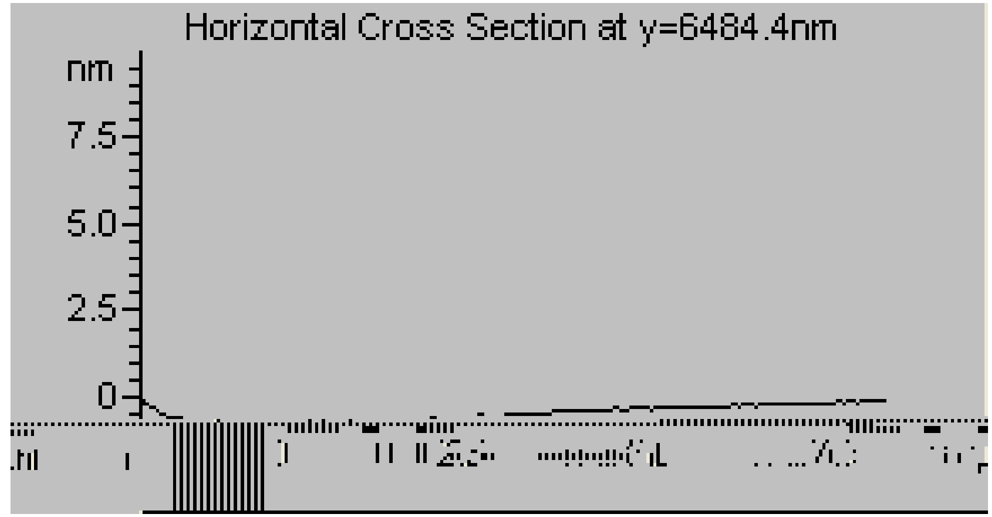
- **Polyester (PF 60001): No transfer, strong bonds**

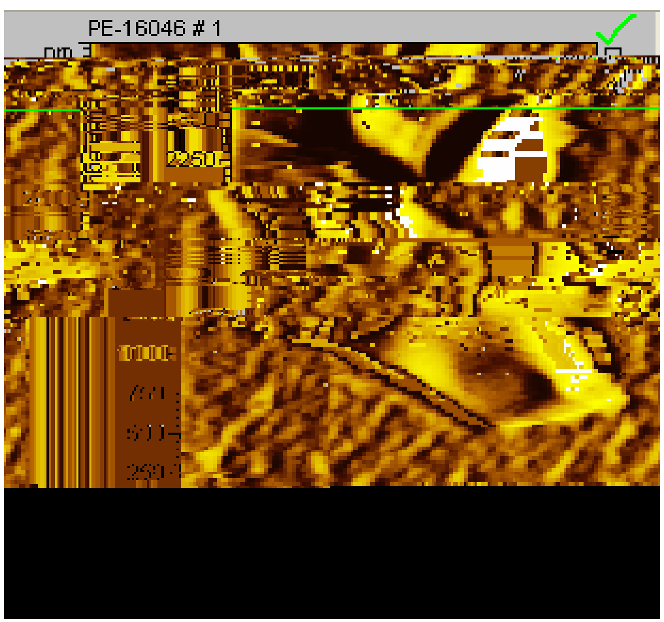
- **SRB (PF 60001): Siloxane coating transfer, weak bonds**

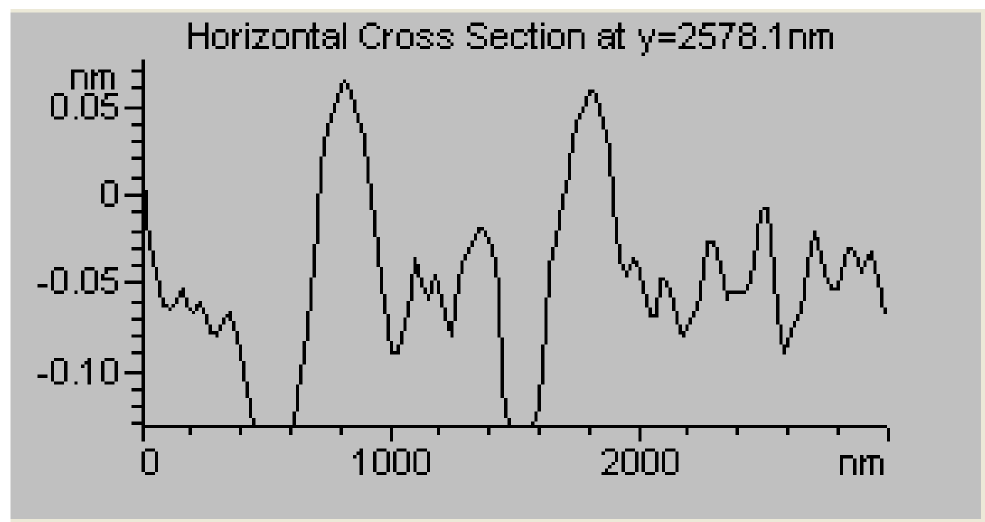
- **Nylon (PF 52006): Fiber transfer, bond strength depends on adhesive**

Polyester Peel Ply Surface

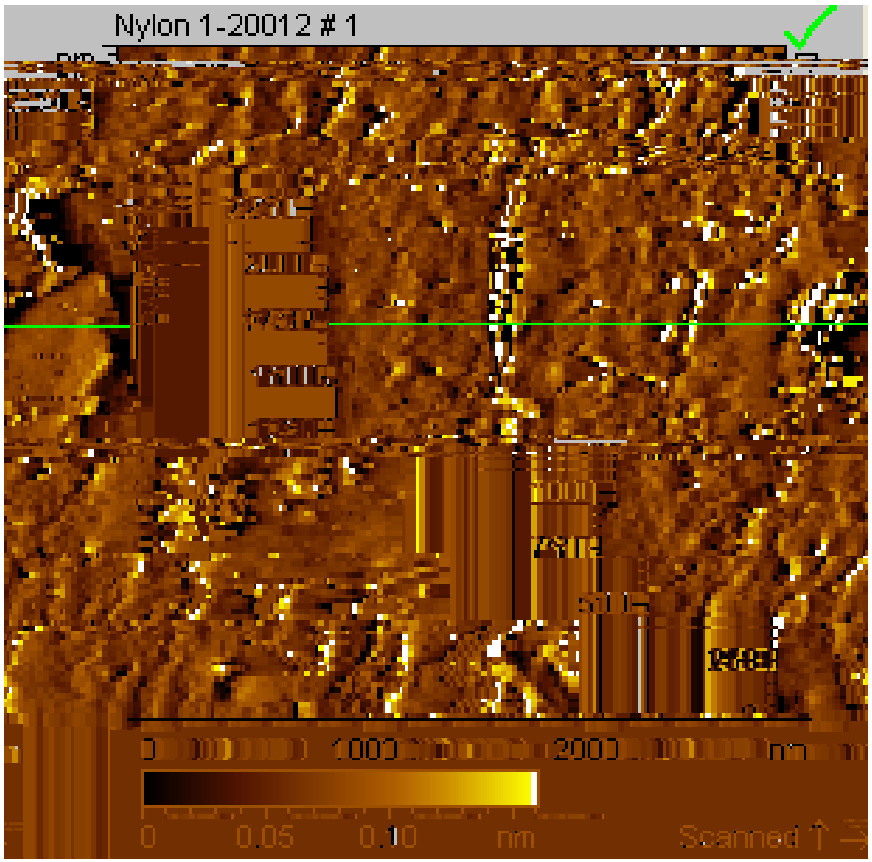




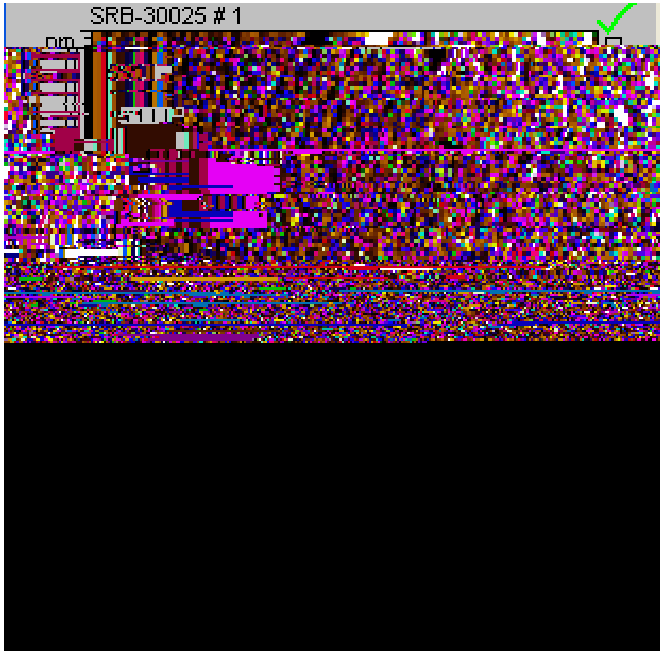


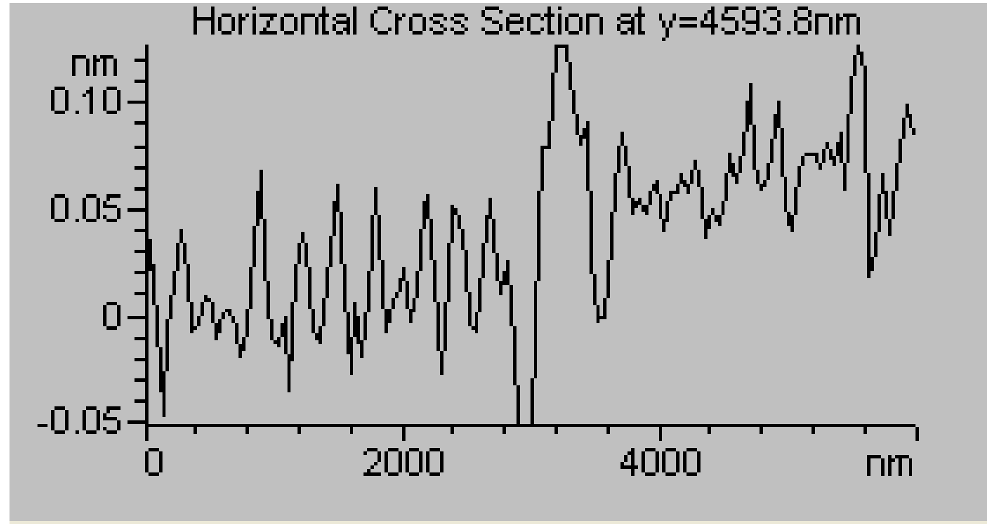


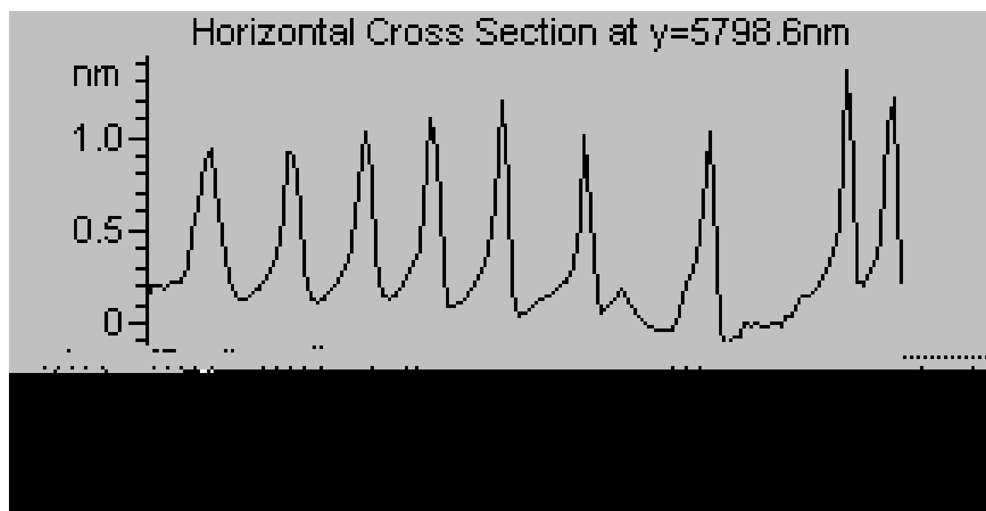
Nylon Peel Ply Surface



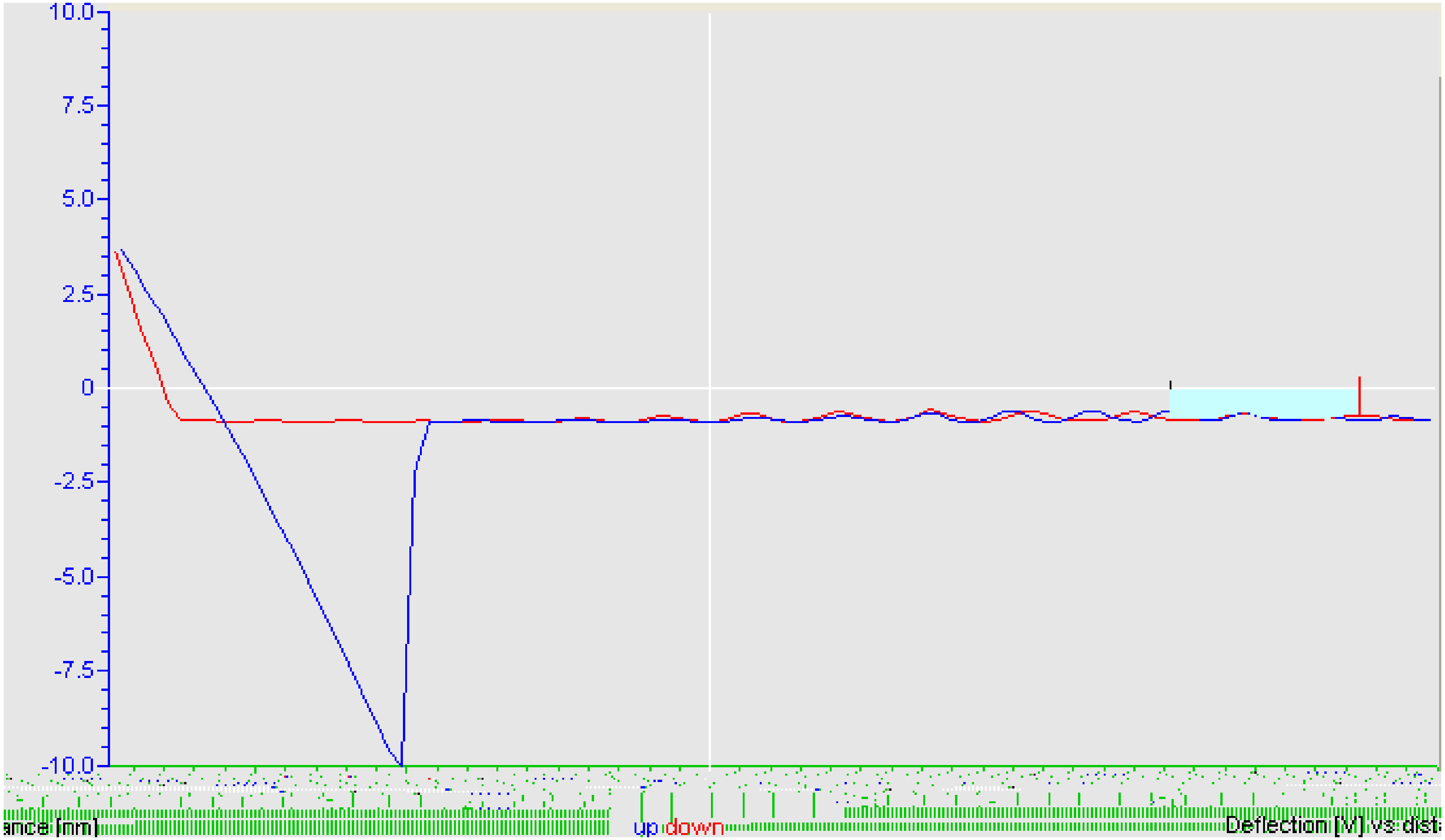
SRB Peel Ply Surface







Typical Force vs. Distance For PE Peel Ply Prepared Surface



Conclusions

- AFM can evaluate the contalusions

A Look Forward

- Benefit to Aviation
  - Better understanding of the pre-bond surface preparation methods
  - Better understanding of bond strength and durability versus surface preparation
  - Novel in-field, online certification and assurance technology for surface preparation
  - Reduced costs for surface preparation and adhesive bonding processes
- Future needs
  - In-field, online analytical detection and monitoring technologies for manufacture, chemical, environmental, and energy industries.