

## **Speaker Introduction**

- Alexander Niebroski, Research and Development Lab Manager at HZO
  - Worked at the company for 6 years, starting as an R&D Engineer
  - Patents in plasma processing

#### Education:

- Master's graduate of the Department of Materials Science Engineering at NC State.
  - Jay Narayan research group (novel semiconductors)
- Bachelor's graduate of Materials Science and Engineering department at Arizona State University.
  - Bertoni DEfECT research group (photovoltaic semiconductors)
  - Karl Sieradzki research group (corrosion/electrochemistry)

#### New father

2 months today



#### **Problem Statement**

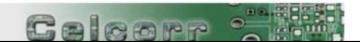
- Electronic devices are highly susceptible to failure when exposed to moisture
- Moisture takes many forms from rain, beverages, and even humid environments
- Conformal coatings provide a key line of defense against such damage
- What techniques can be performed to evaluate coating performance?



## **Uncoated sample**

2V bias potential

2μl droplet added containing 0.2 g/L NaCl



## **Agenda**

- Introduction to Conformal Coatings and Electronics
  - Parylene chemical vapor deposition (CVD) coating process
  - Comparison to traditional conformal coatings
- Test Methods for Coating Properties and Performance
  - Dielectric performance
  - Thickness via spectral reflectance
  - Chemical identification using FTIR and UV-Vis-NIR
  - Crosshatch adhesion tape testing
  - Pencil hardness and nanoindentation
  - Surface wettability or hydrophobicity using water contact angle
  - Other assorted tests
- General lessons learned





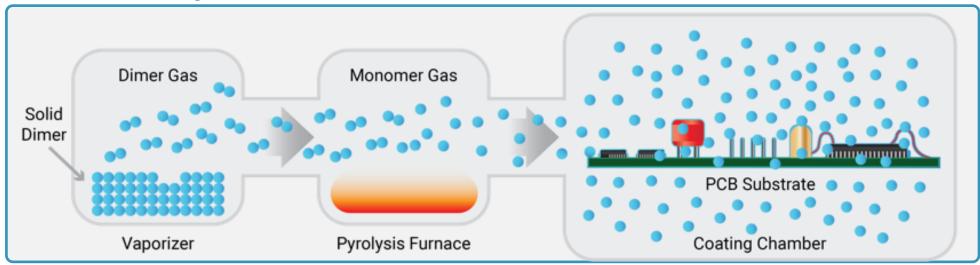






## Parylene Chemical Vapor Deposition (CVD) Coatings

## **Parylene Coating Process**



No VOCs

No **Catalysts** 

No **Disposal Issues** 

> No Cure

Time

H<sub>2</sub>C `ÇH<sub>2</sub>  $H_2$ H<sub>2</sub>Ċ

Vaporizer

Solid dimer sublimes

to a gas in the vaporizer

Pyrolysis Furnace

Dimer gas is cracked into a

di-radical in the pyrolizer

**Coating Chamber** 

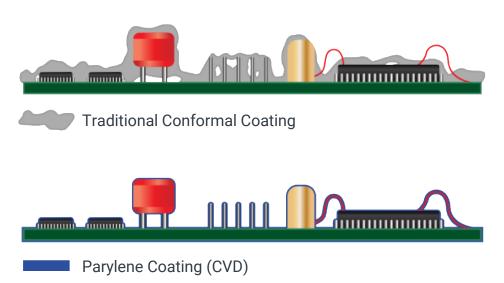
Di-radicals adsorb on

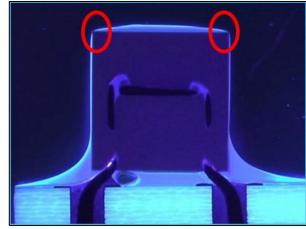
substrates and combine

to form a polymer

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# Traditional Conformal Coatings vs. Parylene

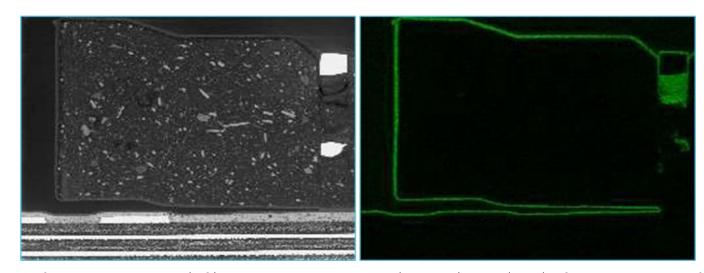




Example of traditional liquid-based conformal coating. *Image from Rockwell Collins* 

**Traditional conformal coatings** are not uniform. Hills and valleys form, especially at corners & edges.

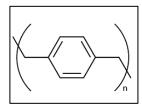
**Parylene** forms a uniform 3D coat on flat surfaces AND all components.



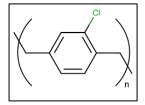
A low magnification SEM image (left) and a corresponding CI (chlorine) map (right) of a cross-section of a board-to-board connector. Note that the 10-micron thick coating covers the surfaces of the thin gap under the connector, reaching as far as the edge of the opening allowed it to.



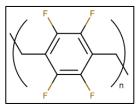
## **Parylene Coating Types**



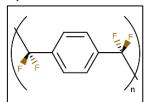
Parylene N



Parylene C



Parylene F (VT-4)



Parylene F (AF-4)

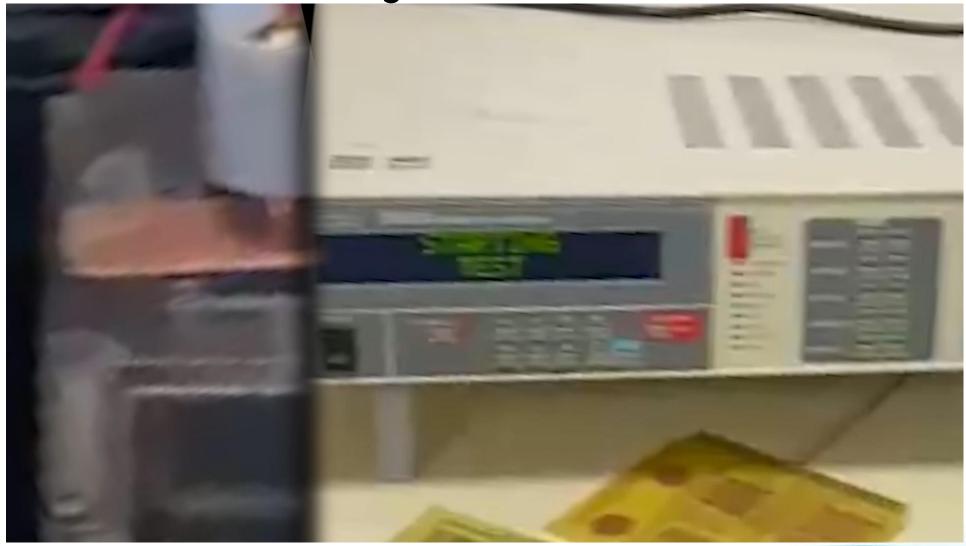
General Preference	Properties	<b><i>≡</i></b> Decreasing − Parylene-Type − Increasing <b><i>≡</i></b>			
Lower Values	Raw Material Cost	N	С	VT-4	AF-4
	Coefficient of Friction	AF-4	N	С	VT-4
	Refractive Index	AF-4	VT-4	С	N
	Deposition Rate	AF-4	N	VT-4	С
	High-Temperature Exposure Performance	N	С	VT-4	AF-4
	Ultraviolet (UV) & Direct Sunlight Exposure Performance	N	С	VT-4	AF-4
Higher	Moisture Barrier Performance	N	VT-4	AF-4	С
Values	Gas Permeability Performance	N	VT-4	AF-4	С
	Crevice, Gap, and Stand-Off Penetration	С	VT-4	N	AF-4
	Dielectric Properties (Dielectric Constant)	С	N	VT-4	AF-4
	Mechanical Properties (Tensile Strength)	N	AF-4	VT-4	С
	Crystallinity Level after Thermal Treatment	N	С	AF-4	VT-4

## **Test Methods for Coating Properties and Performance**

- Electrical
- Various spectroscopy (light based)
- Mechanical
- Hydrophobicity and surface energy
- Barrier Performance/Chemical resistance

## **Electrical techniques**

Dielectric Breakdown Voltage



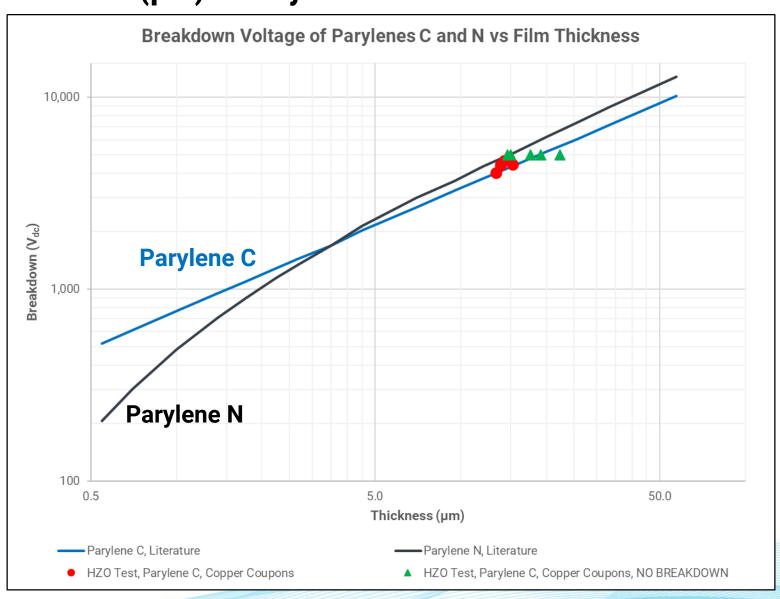


## Breakdown Voltage vs. Film Thickness (µm) - Parylenes C and N

Parylene C Film Thickness (µm)	Breakdown* (V <sub>dc</sub> )	Breakdown Status
13.3	4,038	Yes
13.8	4,443	Yes
14.2	4,680	Yes
14.6	5,000	No
15.0	5,000	No
15.2	4,462	Yes
17.6	5,000	No
19.1	5,000	No
22.3	5,000	No

<sup>\*</sup> The HZO Metrology Lab's breakdown voltage system has an upper limit of 5,000 V, so films above that limit won't experience a dielectric breakdown event.





## **Dielectric properties of Parylene for RF signals**

- After the last PCB West meeting, we received an email stating "Our boards carry RF signals that are very sensitive to the
  dielectric constant of materials near the traces. Is there any chance you have Dk or Df values in the 10-40GHz range for
  your Parylene?"
- A literature review revealed the following:

Parameter	Frequency	Parylene N	Reference Title	Reference Link	
	1 MHz	2.65	A 6 to 24 GHz Continuously Tunable, Microfabricated, High-Q Cavity Resonator With Electrostatic MEMS Actuation	https://doi.org/10.1109/MWSYM.2012.6259684	
	< 60 GHz	2.35 – 2.4	Characterization of Parylene-N as Flexible Substrate and Passivation Layer for Microwave and Millimeter-Wave Integrated Circuits	https://doi.org/10.1109/TADVP.2008.2006760	
Dielectric Constant [Real Permittivity (ε')]	< 60 GHz	2.4	Low-Loss Coplanar Waveguide Transmission Lines and Vertical Interconnects on Multi-Layer Parylene-N	https://doi.org/10.1109/SMIC.2009.4770533	
	0.2 THz (200 GHz)	2.63		https://doi.org/10.1007/s10762-019-00584-2	
	1 THz (1,000 GHz)	2.61	Dielectric Properties of Low-Loss Polymers for mmW and THz Applications		
	2 THz (2,000 GHz)	2.56			
	1 MHz	0.0006	A 6 to 24 GHz Continuously Tunable, Microfabricated, High-Q Cavity Resonator With Electrostatic MEMS Actuation	https://doi.org/10.1109/MWSYM.2012.6259684	
B	< 60 GHz	0.0006	Characterization of Parylene-N as Flexible Substrate and Passivation Layer for Microwave and Millimeter-Wave Integrated Circuits	https://doi.org/10.1109/TADVP.2008.2006760	
Dissipation Factor [Loss Tangent (tan δ)]	< 60 GHz	0.0006	Low-Loss Coplanar Waveguide Transmission Lines and Vertical Interconnects on Multi-Layer Parylene-N	https://doi.org/10.1109/SMIC.2009.4770533	
	0.2 THz (200 GHz)	0.0056			
	1 THz (1,000 GHz)	0.0280	Dielectric Properties of Low-Loss Polymers for mmW and THz Applications	https://doi.org/10.1007/s10762-019-00584-2	
	2 THz (2,000 GHz)	0.0580			

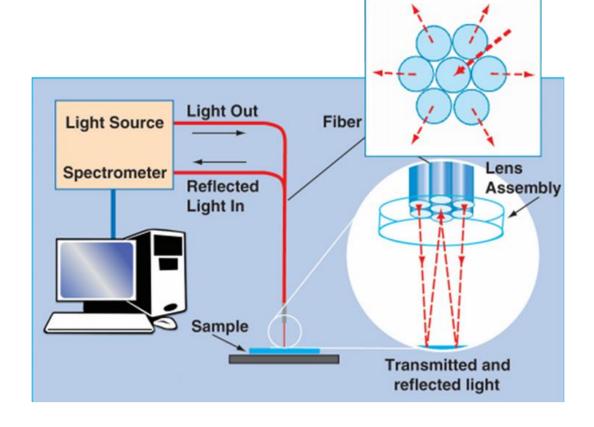


## Reflectometry and various spectroscopy

## **Reflectometry – Determining thickness & transmittance**

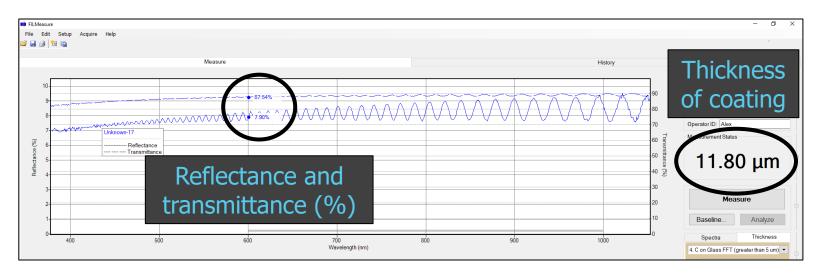


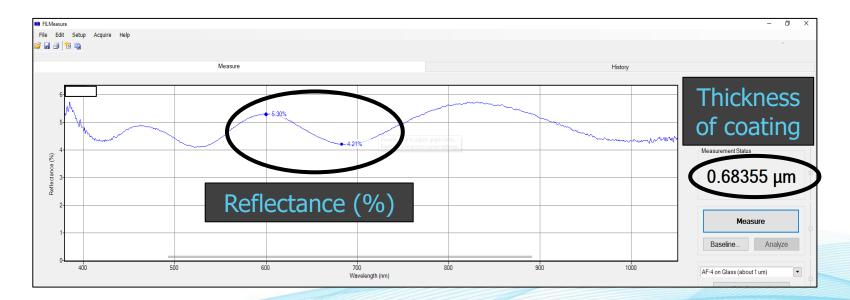
Images adapted from Filmetrics marketing materials



## **Reflectometry – Determining thickness & transmittance**





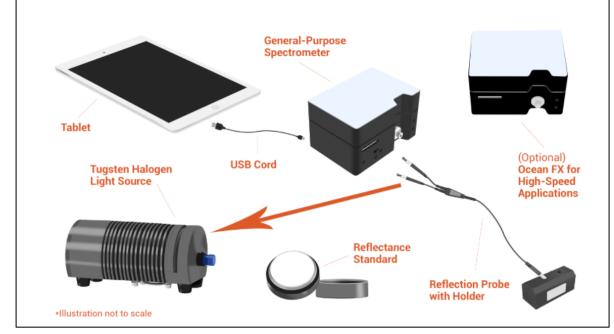




## **UV-Vis-NIR Spectroscopy – Optical Absorption**

In this tech tip, we review the components you'll need - spectrometer, light source, sampling optics — to make reflectance and color measurements. Configure components to optimize your setup for myriad applications.

Here's a sample Ocean Optics reflectance system that uses a reflection probe as the sampling device. The tablet shown here is a stand-in for laptop PCs and other computing devices. This is just one example of dozens of different reflectance and reflected color measurement configurations possible with Ocean Optics spectrometers and accessories.



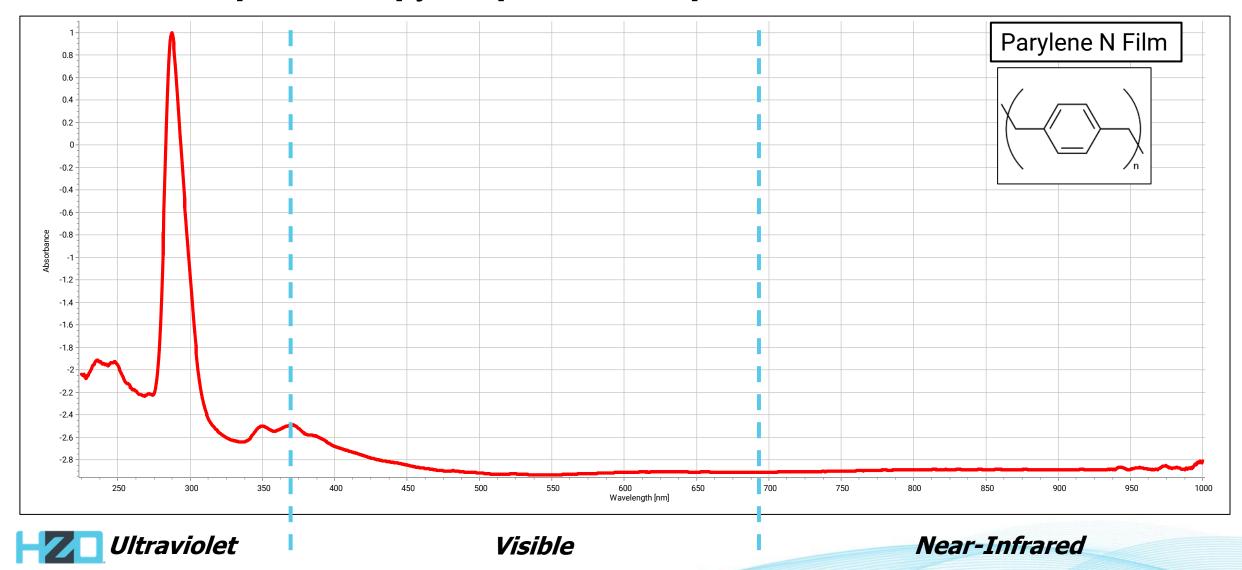






From "Example Reflectance Measurement Setup" <u>https://www.oceanoptics.com/blog/example-reflectance-measurement/</u>

## **UV-Vis-NIR Spectroscopy – Optical Absorption**

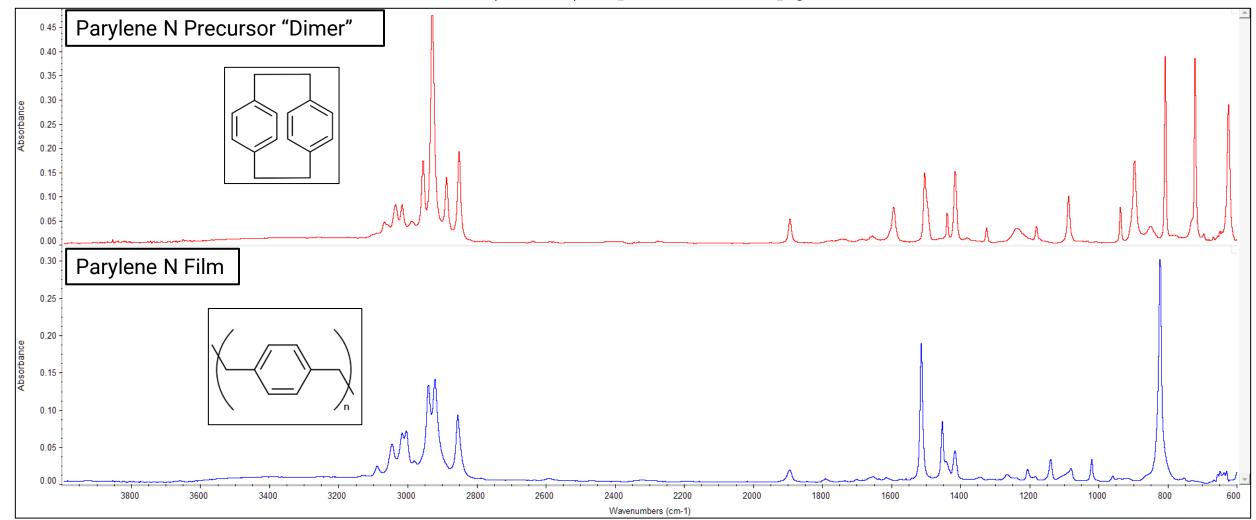


## Fourier-Transform Infrared (FTIR) Spectroscopy



- Nicolet is50-FTIR set up in ATR mode
- Uses system of mirrors and light to obtain chemical information on a bulk sample.
- Test sample can be a powder, liquid, or thin film.

## Fourier-Transform Infrared (FTIR) Spectroscopy





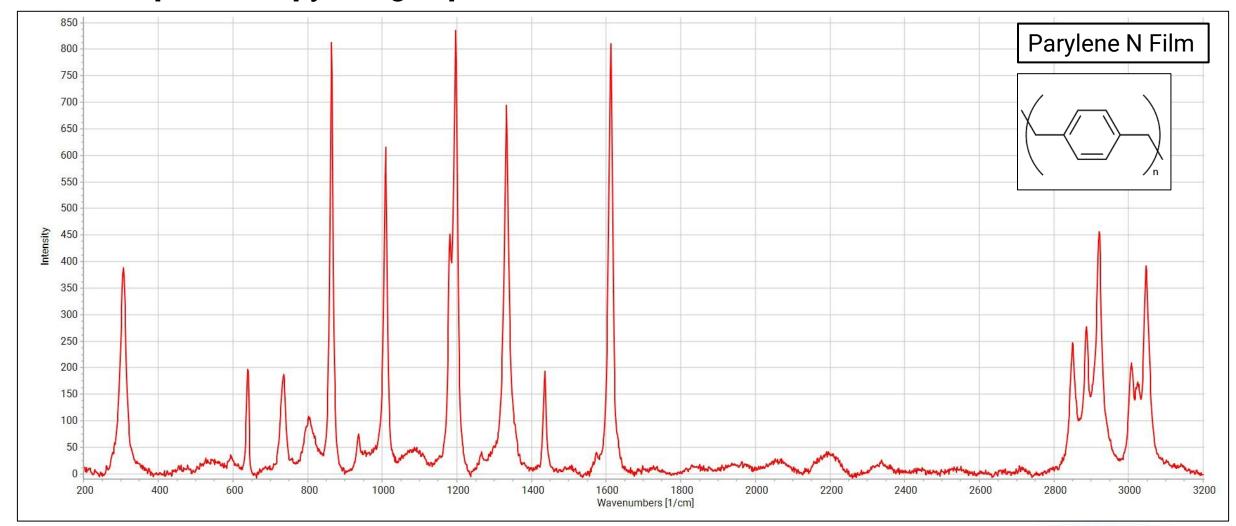
### Raman Spectroscopy - High spatial resolution chemical identification



- Horiba XploRA PLUS Confocal Raman Microscope.
- Uses laser to excite sample and obtain chemical information
- Can focus laser below the surface
- Using the microscope, can focus the laser onto a very small feature (0.5 µm spatial resolution)



## Raman Spectroscopy - High spatial resolution chemical identification

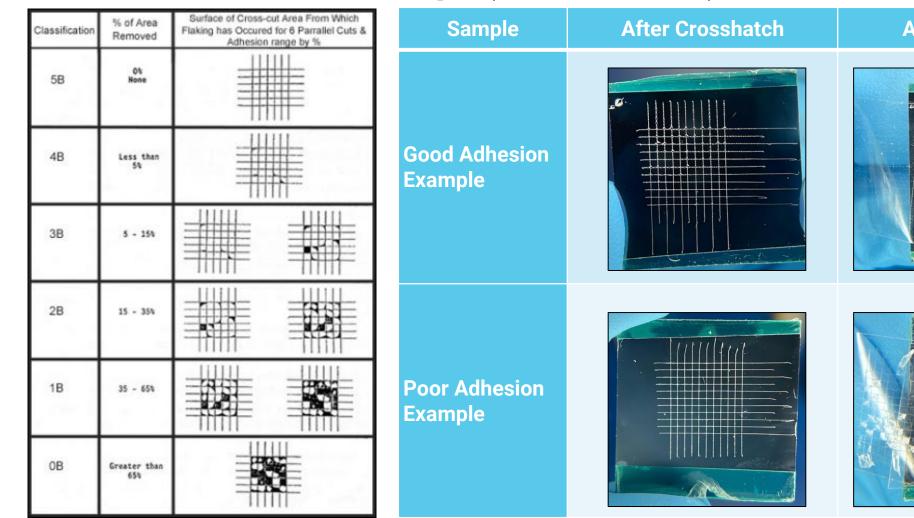


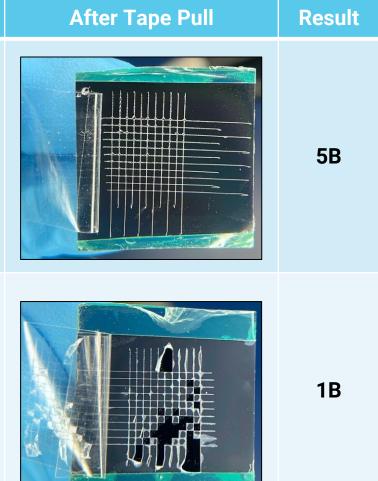


Measured at AIF

## Mechanical techniques

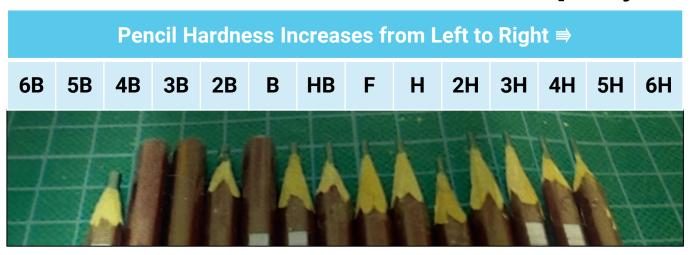
## **Cross Hatch Adhesion Tape (ASTM D3359) – Adhesion**







## **Pencil Hardness – Mechanical Property**

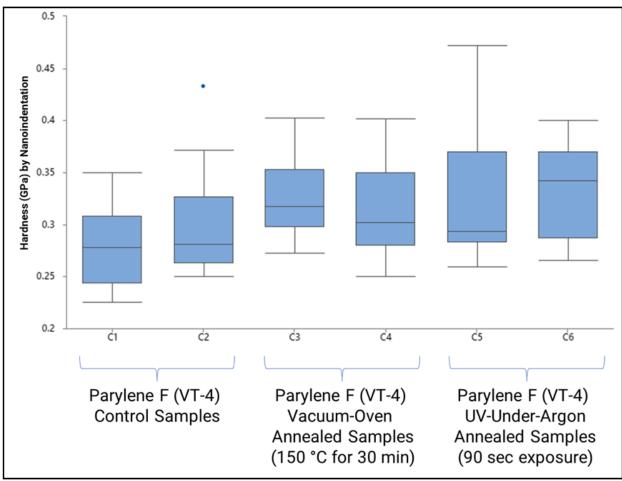




Coating	Pencil Hardness
Parylene C	2H
Parylene N	F
Parylene F (VT-4)	F
Parylene F (AF-4)	Н

## Nanoindentation (at AIF) – Mechanical Properties





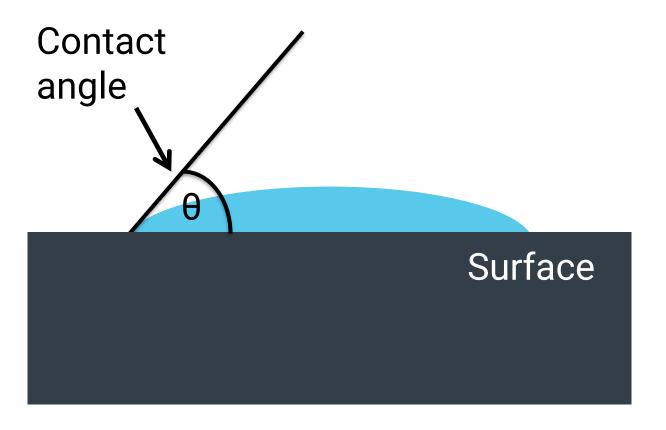


Measured at AIF

## Hydrophobicity and surface energy

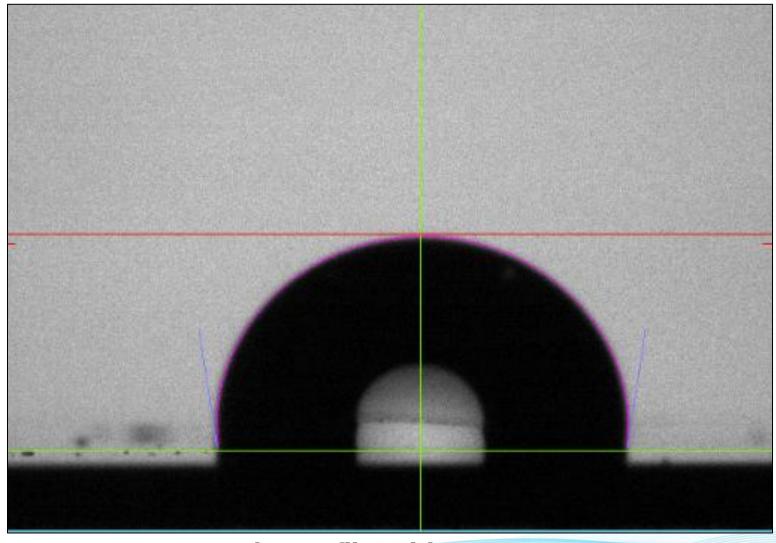
## **Contact angle goniometry (Water or Oil Contact Angle)**





## **Contact angle goniometry (Water or Oil Contact Angle)**





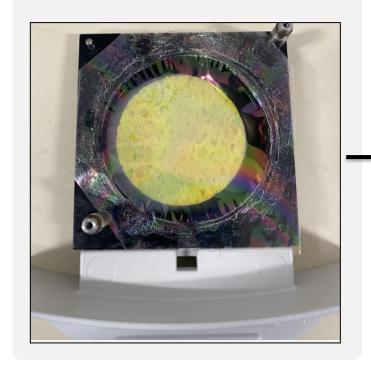


Parylene C film with a 99° WCA

## Barrier Performance/Chemical resistance techniques

## **Water Vapor Transmission Rate (WVTR)**

#### PET coated with thin film



A wet sponge acts as an "infinite" source of 100% relative humidity

#### Loaded in chuck



The top provides mechanical support and alignment

## Loaded in system with sensors



Loaded into tool with temperature and humidity control



## Water Vapor Transmission Rate (WVTR)

- WVTR measures how much water vapor transfers through a barrier.
- A piece of PET is cut to fit in the system,
   then coated with a thin film.
- A sponge is placed on one side of the coating
- A sensor on the other side measures relative humidity over time.
- This process is repeated for many cycles in order to remove hysteresis effects

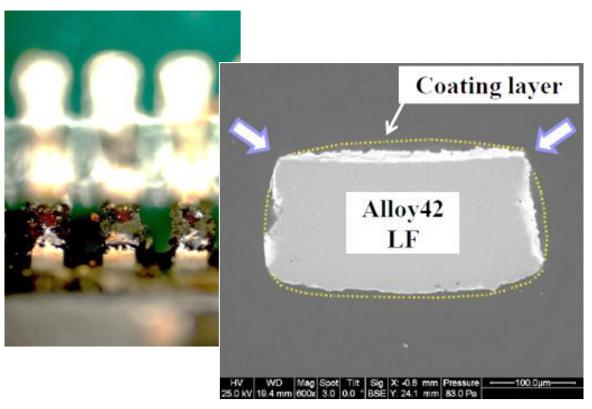
Dohumou	Gas Permeability at 25 °C, (cc·mm)/(m²·day·atm)							WVTR,(g·m
Polymer	N <sub>2</sub>	O <sub>2</sub>	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> S	SO <sub>2</sub>	CI <sub>2</sub>	m)/(m²·day )
Parylene C	0.4	2.8	3.0	43.3	5.1	4.3	0.1	0.08
Parylene N	3.0	15.4	84.3	212.6	313	745	29.2	0.59
Parylene F (VT-4)	-	16.7	-	-	-	-	-	0.28
Epoxy (ER)	1.6	4	3.1	43.3	_	_	-	0.94
Polyurethane (UR)	31. 5	78.7	1,181	-	-	-	-	0.93
Silicone (SR)	-	19,68 5	118,110	17,710	_	-	-	_

Licari, James J. Coating Materials for Electronic Applications – Polymers, Processes, Reliability, Testing

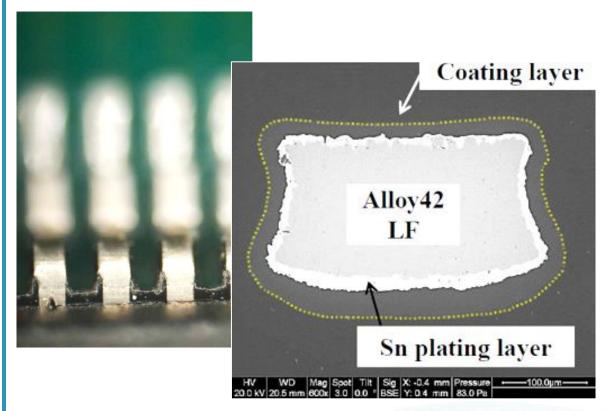


### Mixed Flowing Gas Testing – Sprayed vs CVD Coatings

Silicone Spray-Coated QFP Exposed to MFG Testing, Resulting in Creep Corrosion



Parylene CVD-Coated QFP Exposed to MFG Testing, Resulting in No Corrosion





## **HZO Passed 100% of US Army S/K Challenge**

#### **Background:**

- Coated computers exposed to biological and chemical attack simulants, as well as toxic industrial chemicals.
- Chemical attack simulants presented corrosive vapors from organic acids and common corrosive gases.
- After exposure, the coated samples were decontaminated.
- All the HZO Parylene coated computers passed exposure testing and decontamination procedures.



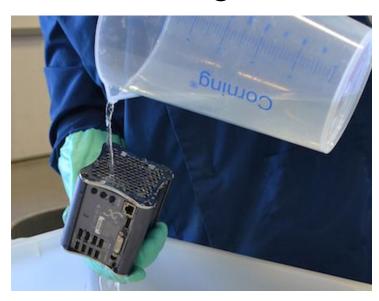
## S/K Challenge – 47 Trials with Biological and Chemical Attack Simulants and Toxic Industrial Chemicals

# Exposed to the following simulants and chemicals: Bacillus atrophaeus (BG) Erwinia herbicola (EH) Male-specific bacteriophage type 2 (MS2) Ovalbumin (OV) Decontaminated with the following and a pure water rinse: Water/bleach mixture (10:1) Bleach solution (5%) High-test hypochlorite (HTH, 3%) Vaporized hydrogen peroxide (VHP)

- Acetic acid (AA)
- Methyl salicylate (MeS)
- Triethyl phosphate (TEP)
- Chlorine gas (Cl<sub>2</sub>)
- Ammonia gas (NH<sub>3</sub>)
- Sulfur dioxide gas (SO<sub>2</sub>)



## S/K Challenge – HZO Coated Computers Passed All the Tests



- Decontamination agent was followed with a pure water rinse
- HZO coated computers passed biological and chemical attacks and all decontamination tests
- Dunk Test was an IPX7 immersion in tap water at 1 meter depth for 30 minutes



HZO Coated / Uncoated	Decontamination Agent	Pass/Fail
Uncoated	10:1 water/bleach mixture	Fail
HZO Coated	10:1 water/bleach mixture	Pass
HZO Coated	10:1 water/bleach mixture	Pass
HZO Coated	10:1 water/bleach mixture	Pass
HZO Coated	10:1 water/bleach mixture	Pass
HZO Coated	10:1 water/bleach mixture	Pass
HZO Coated	10:1 water/bleach mixture	Pass
HZO Coated	10:1 water/bleach mixture	Pass
HZO Coated	Vaporized hydrogen peroxide	Pass
HZO Coated	Vaporized hydrogen peroxide	Pass
HZO Coated	HTH	Pass
HZO Coated	HTH	Pass
HZO Coated	5% high-test hypochlorite	Pass
HZO Coated	5% high-test hypochlorite	Pass
HZO Coated	5% high-test hypochlorite	Pass
HZO Coated	5% high-test hypochlorite	Pass
HZO Coated	Dunk Test	Pass

## Immersion Surface **Insulation Resistance** (SIR) in Artificial **Sweat Test Method**

#### **Surface Mount Technology (SMT) Test Features:**

- OFP = Ouad Flat Pack
  - Simulates active (integrated circuit) component lead layout
- 0603 = Passive Component
  - Simulates passive (capacitor/resistor) component lead layout

#### **Measurement Instrument:**

Gen3 Systems AutoSIR2

#### **Test Coupon:**

- STC14 V1.0 Board with 16 test features:
  - 2 x TQFP32-5-0.5mm (Pad size 1.5 mm x 0.25 mm, Pad gap 0.25 mm) subdivided into 4 test features each.
  - 8 x 0603 pad sets (Pad size 1.1 mm x 1.0 mm, Pad gap 0.6 mm)

#### **Parameters:**

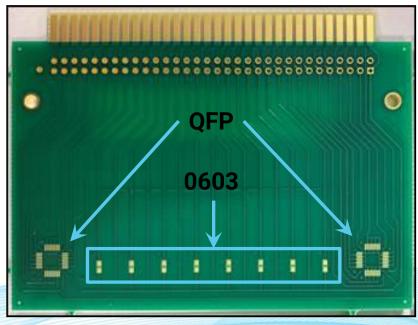
- Samples Number: 1440 minutes (Data point captured on each channel every minute for the full 24 h)
- Measurement Bias: 5 V
- Conditioning Bias: 5 V
- Stabilization Time (sec): 60

#### **Artificial Sweat Solution Recipe:**

- 1 I DI water
- 10 g sodium chloride
- g disodium hydrogen phosphate
- 0.25 g histidine



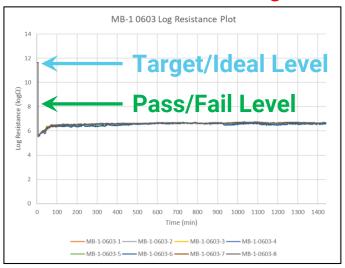
**Measurement Instrument** 

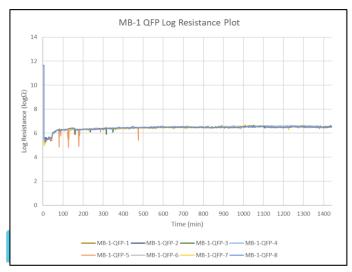


1 q lactic acid

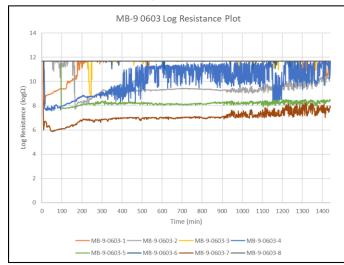
## Immersion SIR - Parylene N Performance vs. Thickness

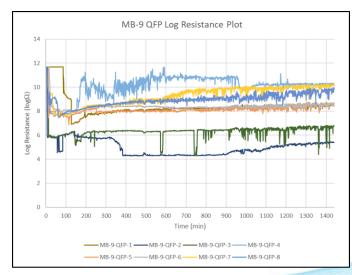
#### **Control – No Coating**



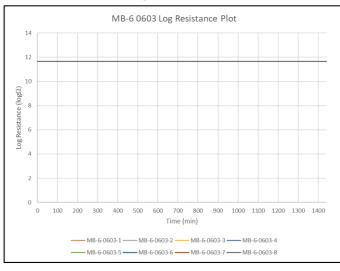


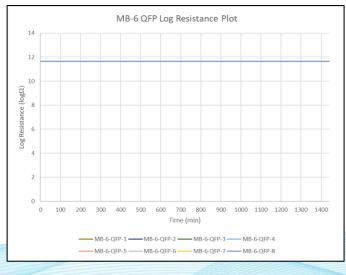
#### 1.1 µm Parylene N Coating





#### 22 µm Parylene N Coating





## **Salt Fog Testing**

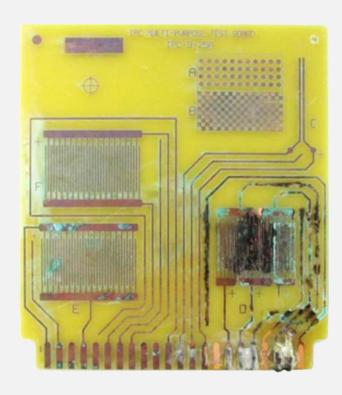


- IPC-B-25A test boards were coated with Parylene and placed in a salt fog chamber.
- Environmental conditions of 35 °C, 5 wt. %
   salt water, 168 hours of salt fog
- After test period, no signs of corrosion present on the Parylene coated board

Performed by Trace Labs / NTS for Third-Party certification

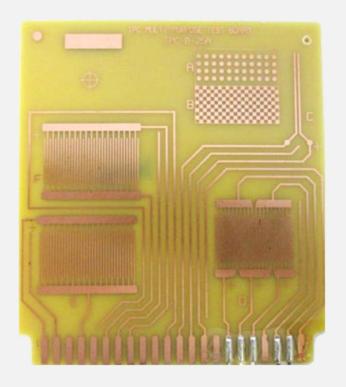


#### **No Protection**



Positive control board showing massive corrosion

#### **HZO Protected**



No corrosion

## **Overview of techniques discussed today**

Technique name	Information gained	How quick are results?
Dielectric Breakdown Voltage	Dielectric Breakdown Voltage	Minutes
Reflectometry	Thickness and optical transmittance	Minutes
UV-Vis Spectroscopy	Optical reflection	Minutes
Raman Spectroscopy	Chemical identification	Minutes
FTIR	Chemical identification	Minutes
Cross-hatch	Adhesion	Minutes
Pencil Hardness	Hardness	Minutes
Nanoindentation	Hardness, additional mechanical properties	Hours
Contact angle goniometry	Contact angle and surface energy	Minutes
Water Vapor Transmission Rate (WVTR)	Barrier, especially for gases	Days
Mixed flowing gas	Barrier/chemical resistance	Days
Surface Insulation Resistance (SIR)	Contamination	Days
Salt Fog	Barrier/chemical resistance	Days



## **Summary**

- Parylene coatings can improve reliability by protecting electronics from corrosion
- Quick screening techniques provide efficient early evaluation for R&D and quality control
  - Focus majority of resources on equipment/training for screening techniques
- Leverage advanced tools for detailed analysis when feasible
- Collaborations with local universities can offset equipment costs and expand testing capabilities – especially with expertise
- Engage certified third-party labs for validation and compliance testing

## Thank you! Questions?

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