BTG LABS

# Predicting Bond Performance of Aerospace Materials Through Non-Destructive Testing

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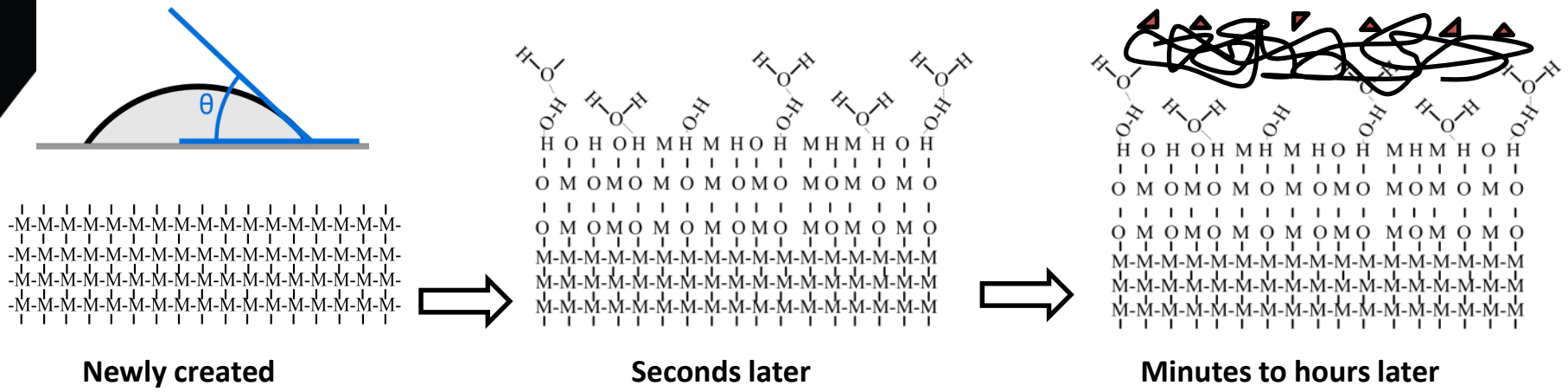
NDT – A4A Forum 2018

# Agenda

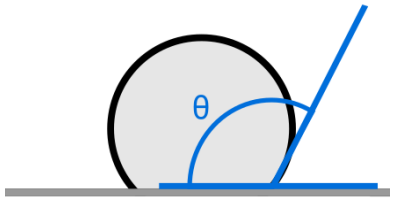
- What is a surface?
- Measuring Surface Energy – why does this matter?
- Contact Angles as a convenient way to evaluate Surface Energy
- Controlling your Surface – Contamination
- The Surface Analyst™
- Applications in Aerospace
- Correlating surface energy to system performance

# What is a Surface & How Does it Decay?




**Surfaces tend towards a lower energy state**

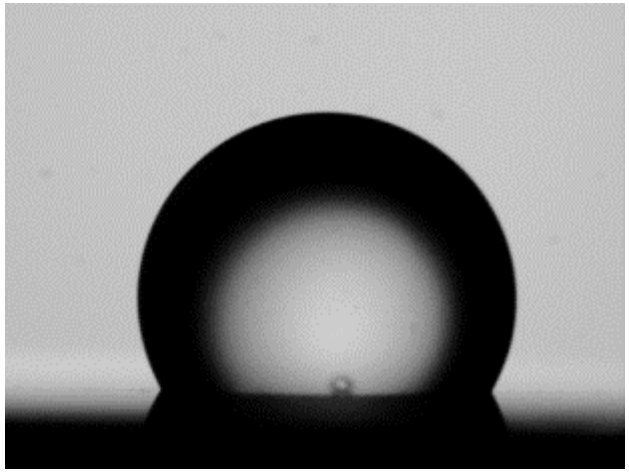
- Newly created surfaces are very reactive
- This reactivity is what we call surface energy
- These layers are influenced by contaminants
  - Oxygen, water vapor, oil vapor from machinery, contact contaminants, mold release

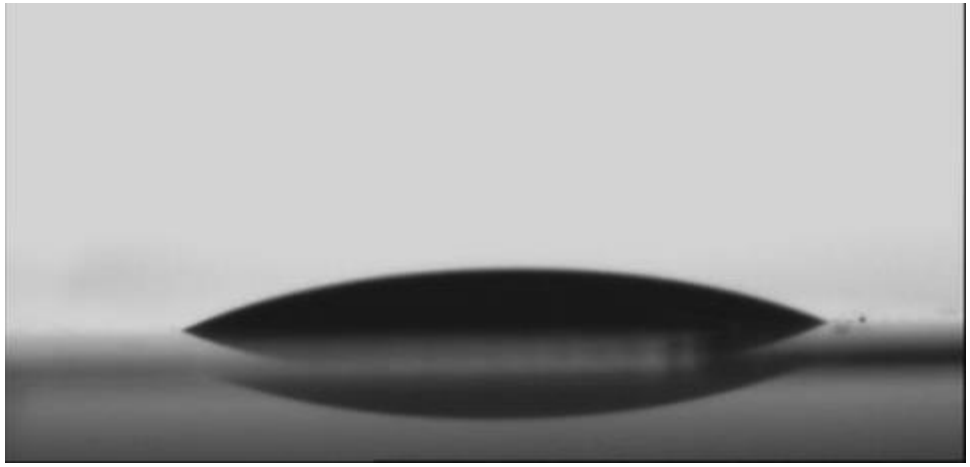


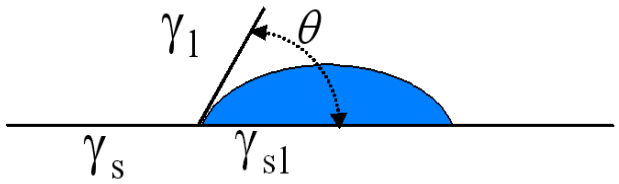

***Quality of bonding is dependent upon the top few molecular layers of the surface***

# Contact Angles and Surface Energy

- An isolated liquid drop in the absence of external forces assumes a spherical shape
- Upon contact with a surface, drop shape is determined by the balance of liquid-liquid vs liquid-solid forces
- Contact angle cosine is directly proportional to surface energy of solid
- **Surface energy is strongly affected by presence of contamination**



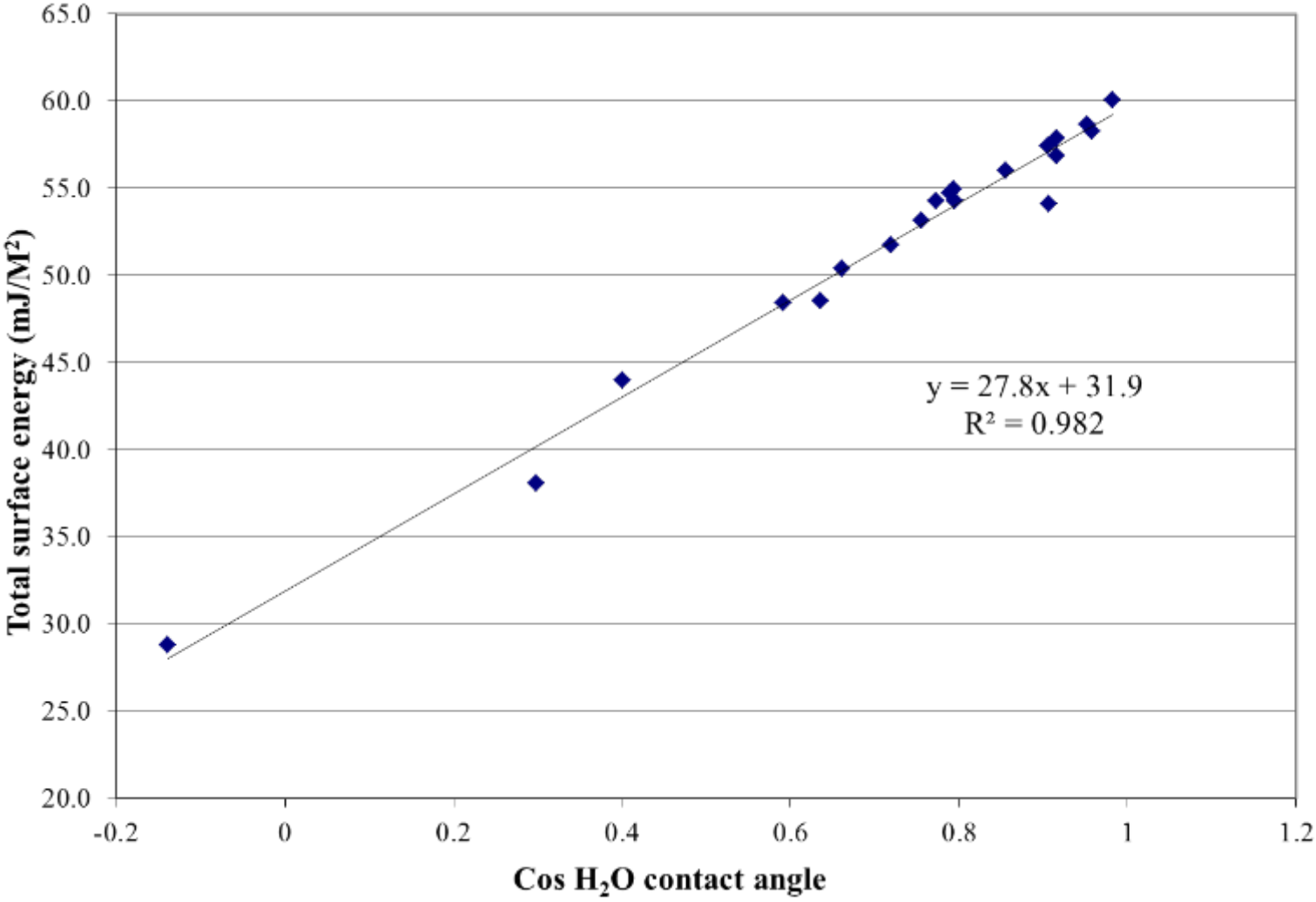





Young equation:

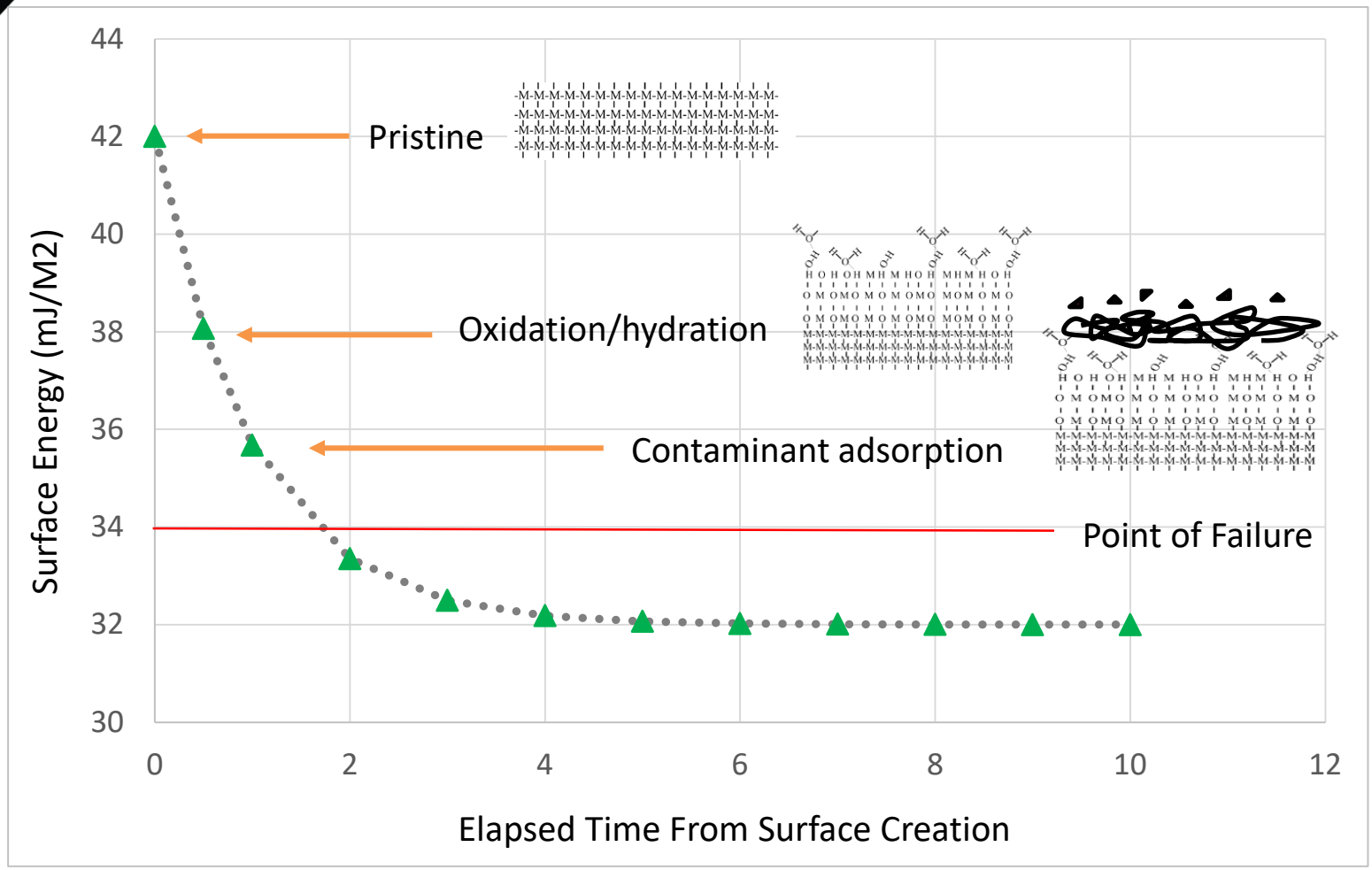
$$\gamma_s = \gamma_{sl} + \gamma_l \cos\theta$$

# Contact Angle of Water is Proportional to Surface Energy



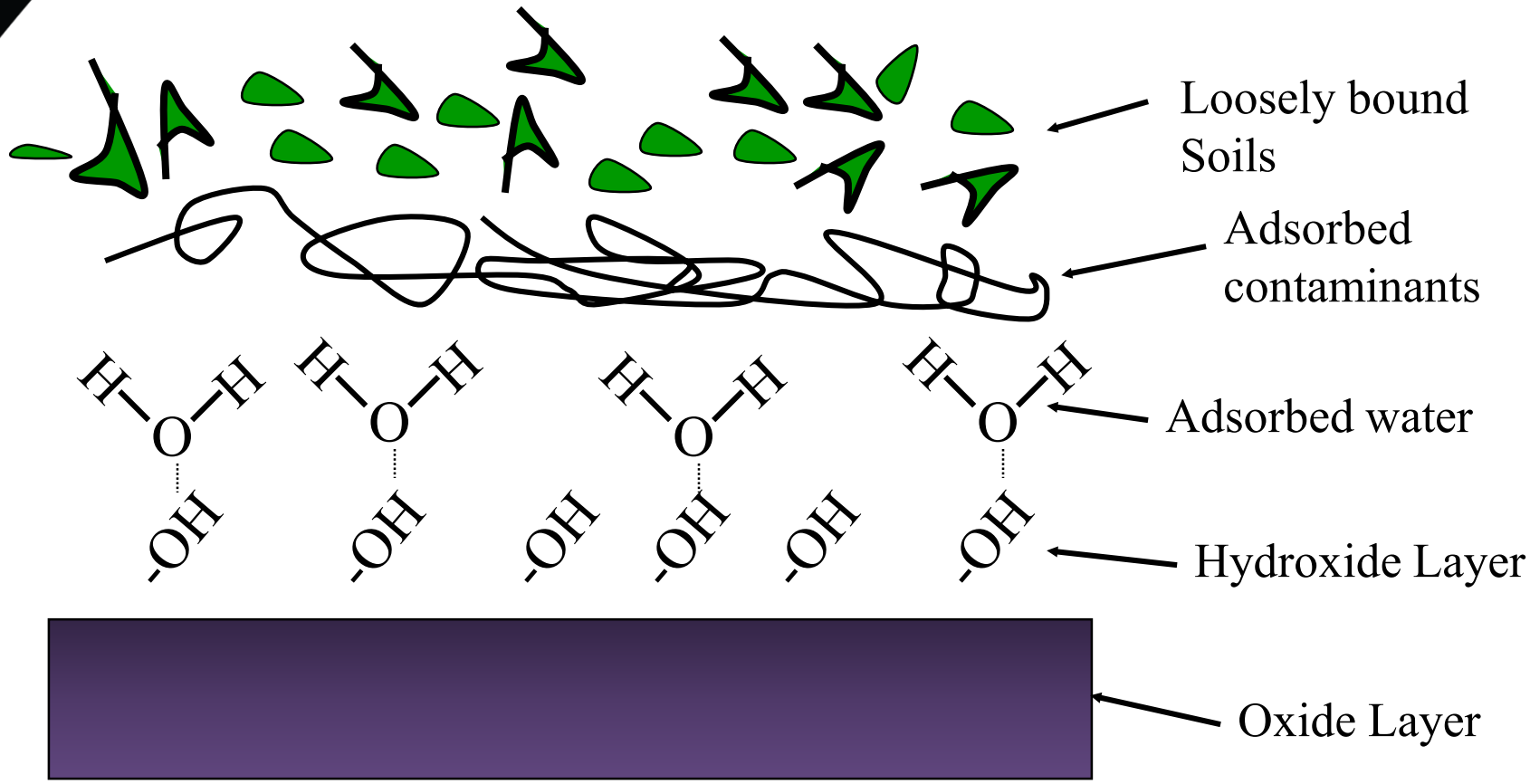

Dillingham, Oakley et al. J Adhesion Science Technology 29 (2015) 890-895.

# Manifestation of Surface Energy

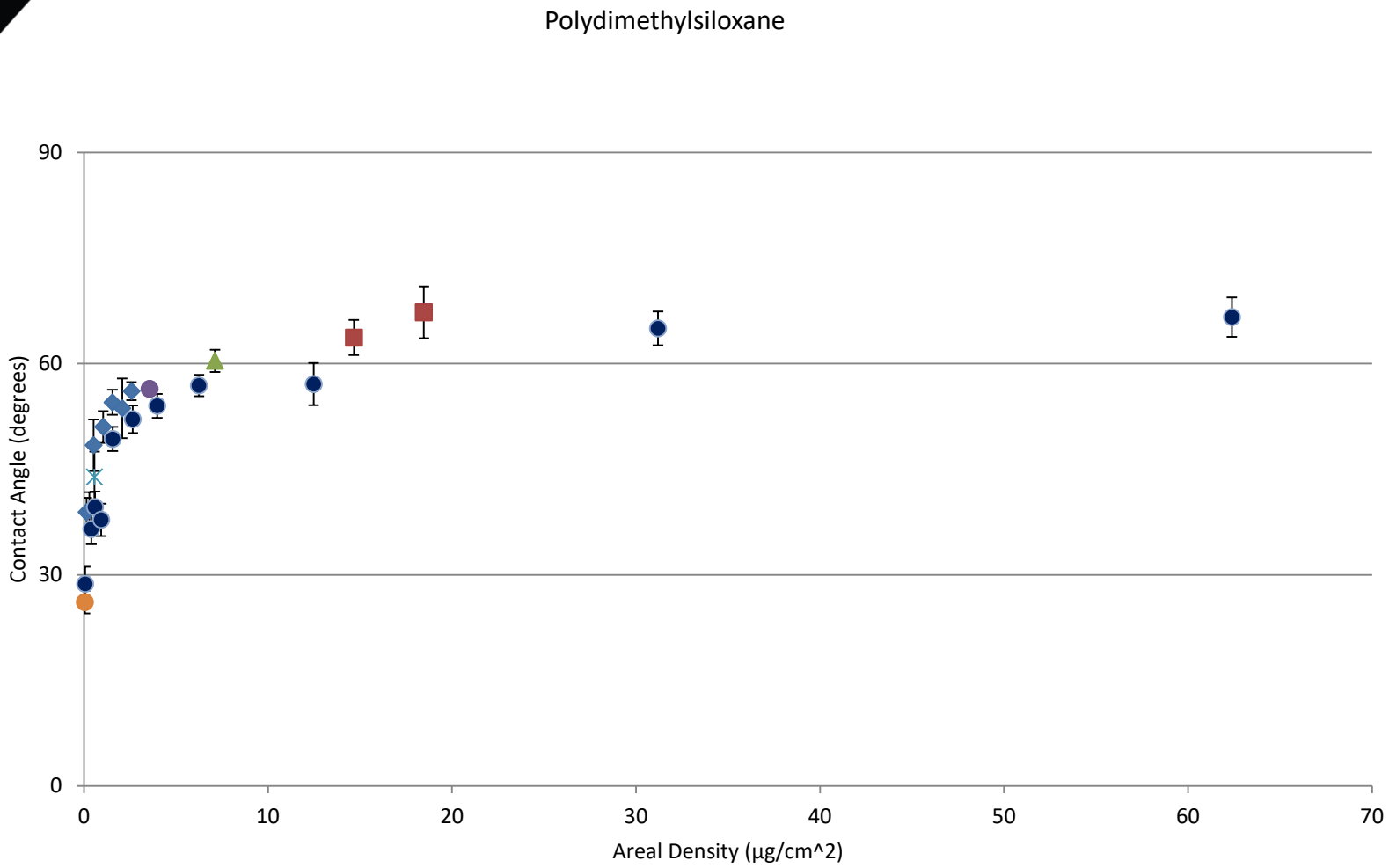



- Rate of surface energy decay depends on initial surface, surface energy and environment

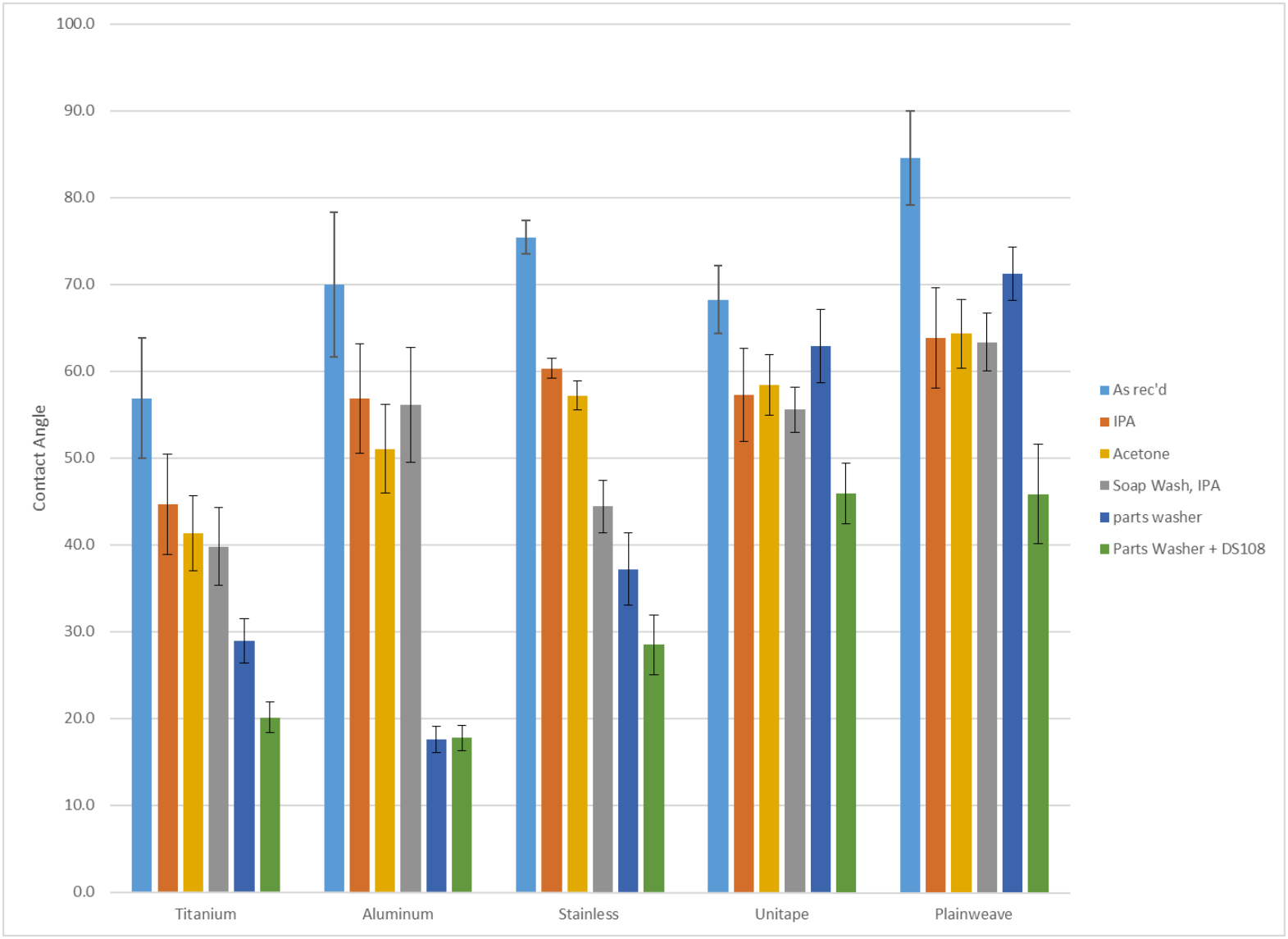
# Adhesives are Competing with Contaminants and Soils for Active Surface Sites



# Contact Angle Behavior

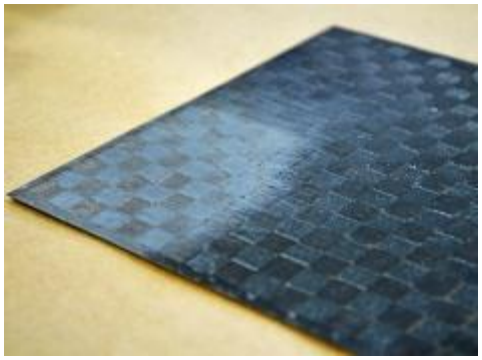


# Contact Angle for Evaluation of Cleaning Processes**



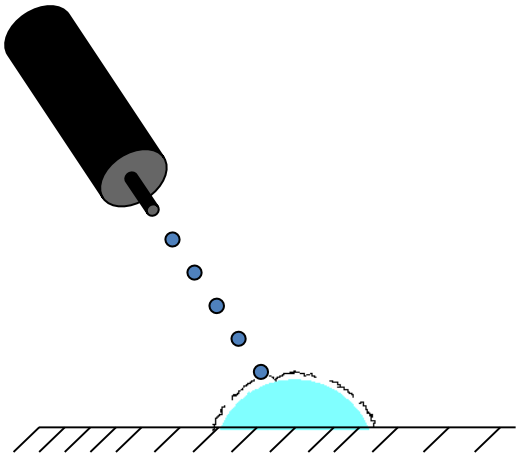
# The Surface Analyst™

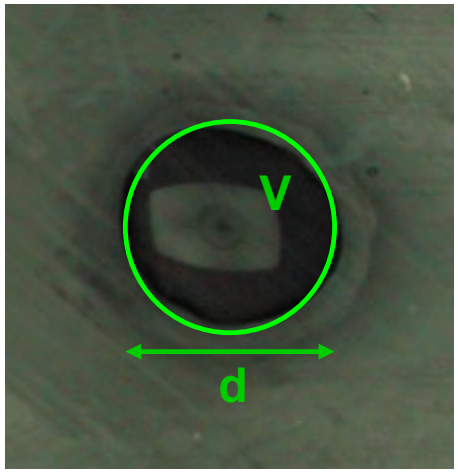
- **Takes sensitive surface chemistry measurements out of the laboratory and puts them in the hands of manufacturing and quality personnel**
- **Fast** - <2 second inspection
- **Easy** - anyone can use it
- **Accurate** – clean to a number
- **Non-destructive** – will not harm part being inspected
- **Flexible** – multi-directional inspections, can be used in many industries and applications
- **Repeatable** – passes Gage R&R

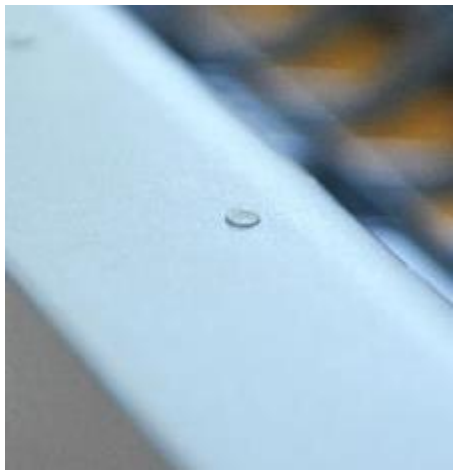



# What does the Surface Analyst™ do?

- Small droplet (<2 µl) of probe fluid is created on the surface from a pulsed stream of micro drops
- Contact angle is calculated from drop diameter
- <2 sec measurement cycle
  - Single button operation
  - No operator input
- Immediate, easy-to-understand feedback about cleanliness and reactivity of the surface: is it ready for bonding, painting, coating, sealing?
- Useful in all manufacturing industries





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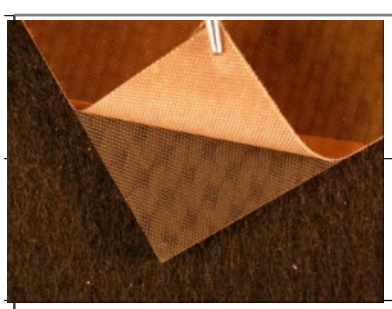
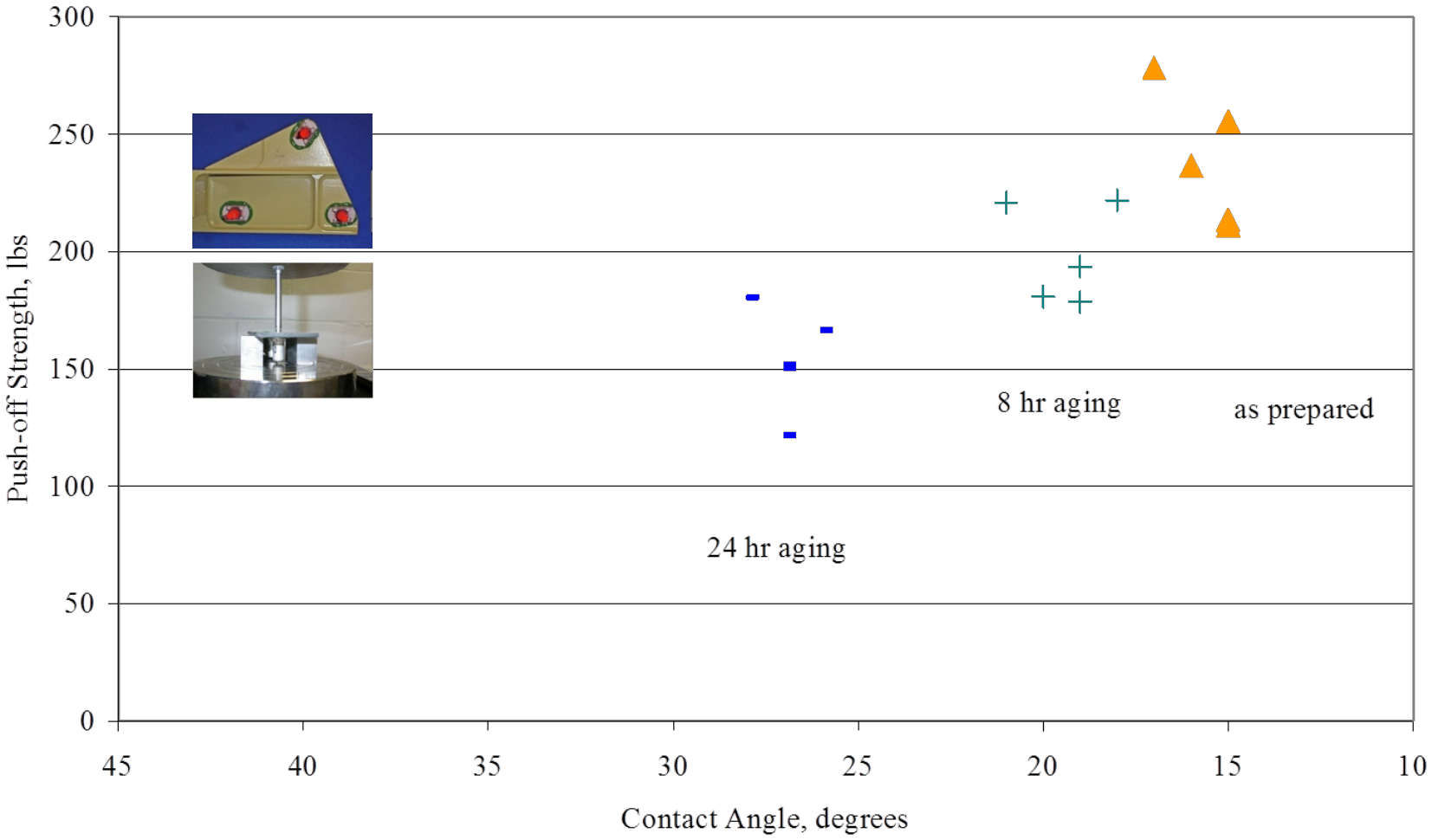
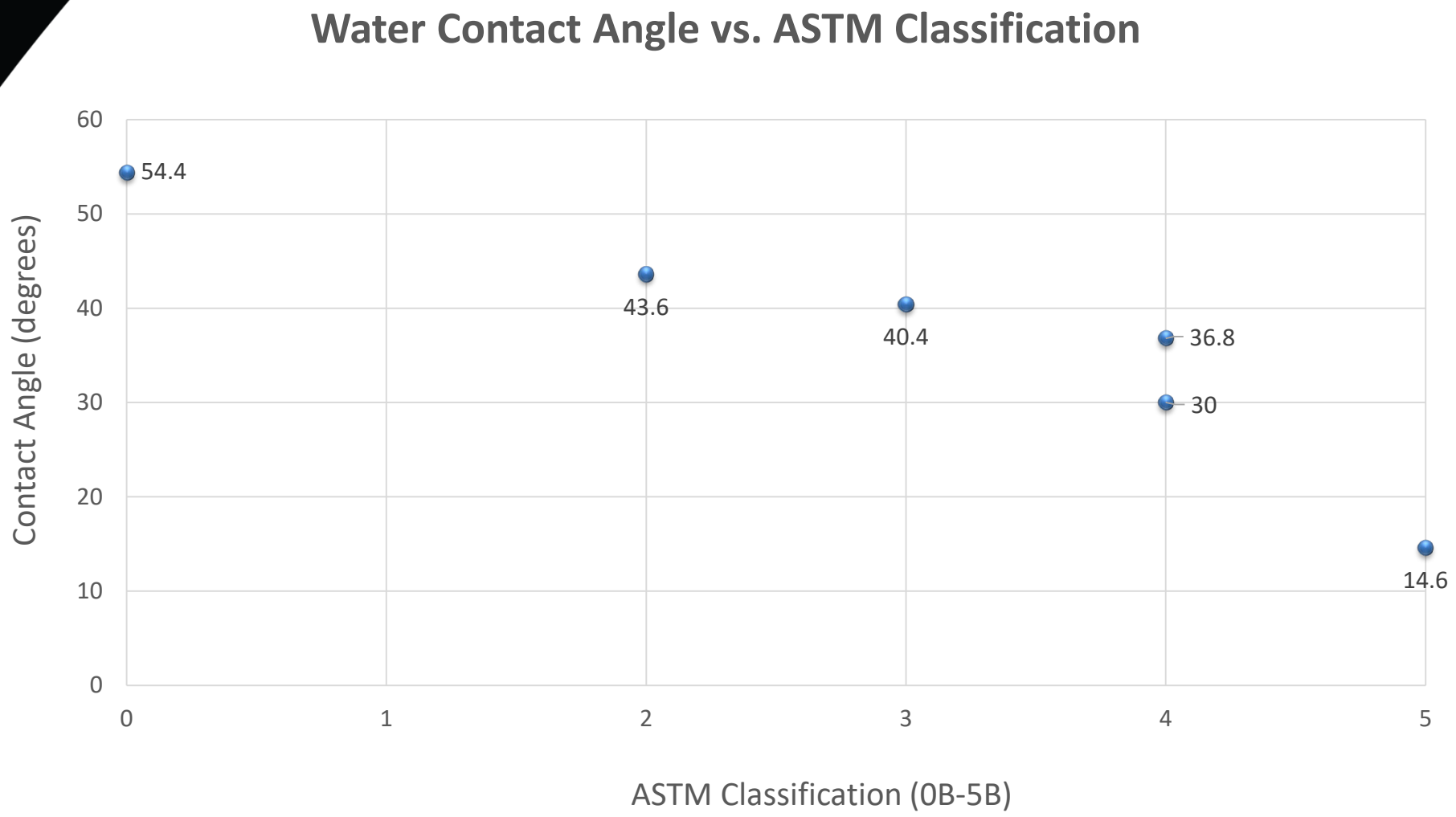
# Characterizing Out Time of a Peeled Laminate Surface



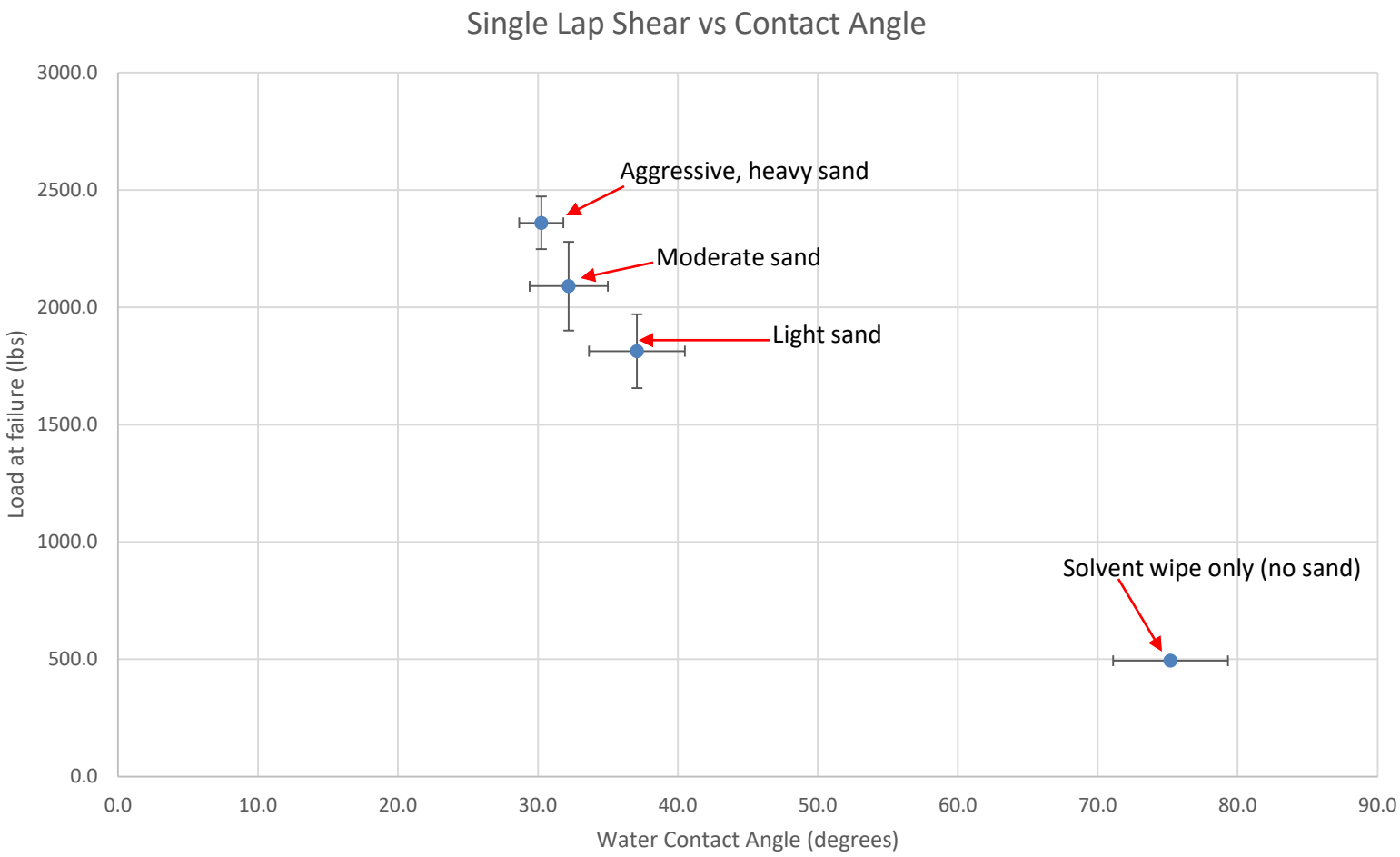
# $\theta_{H2O}$ as Quantitative Prediction of Adhesion – Aluminum



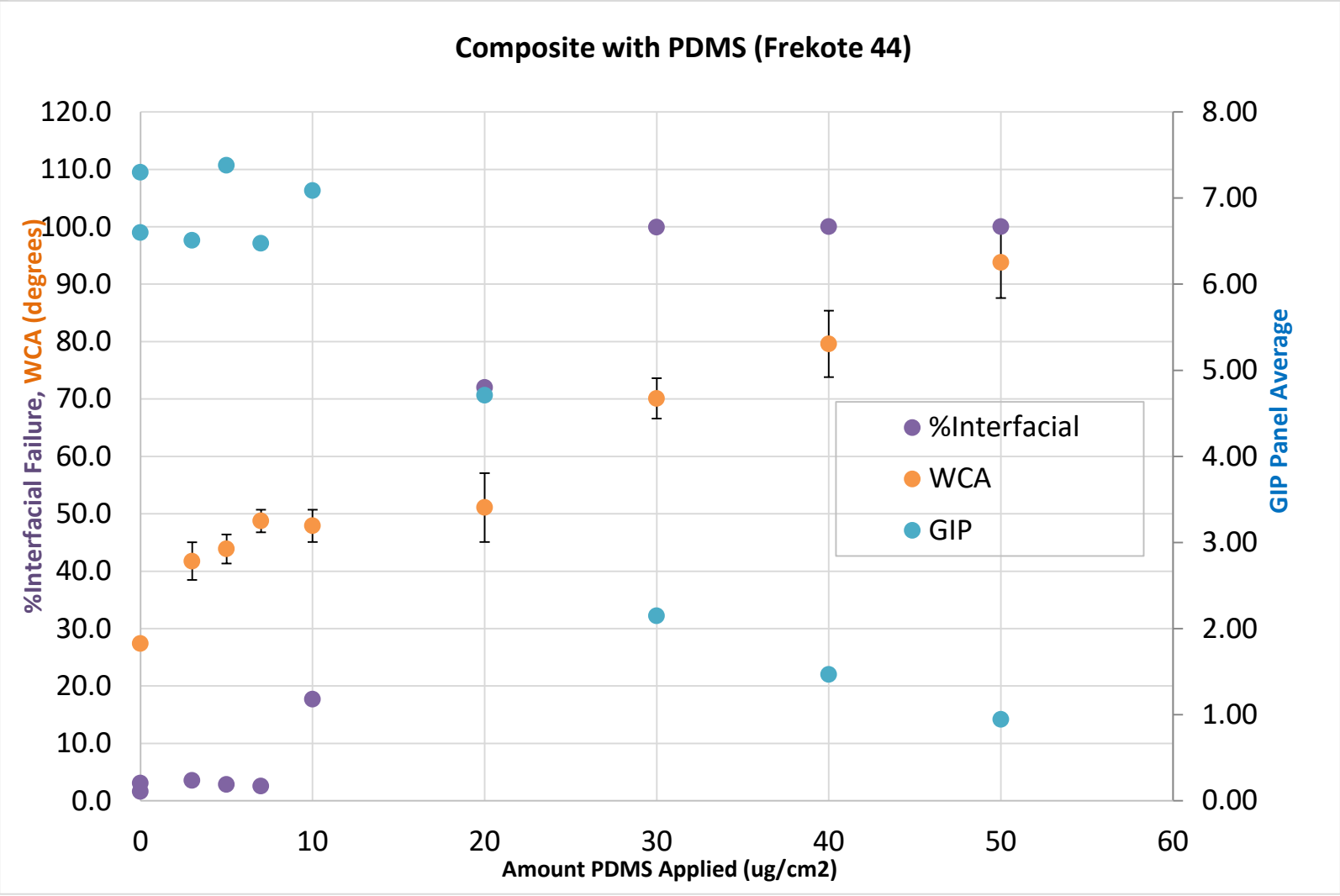
# $\theta_{H2O}$ as Quantitative Prediction of Adhesion - Polypropylene



# Contact Angle for Predicting Bond Performance



# Utilizing Water Contact Angle to Predict Bond Strength



# Additional Applications

- Detection of plasma treatment on wiring inside aircraft
- Predicting penetrate wettability for landing gear
- Bonding interior paneling

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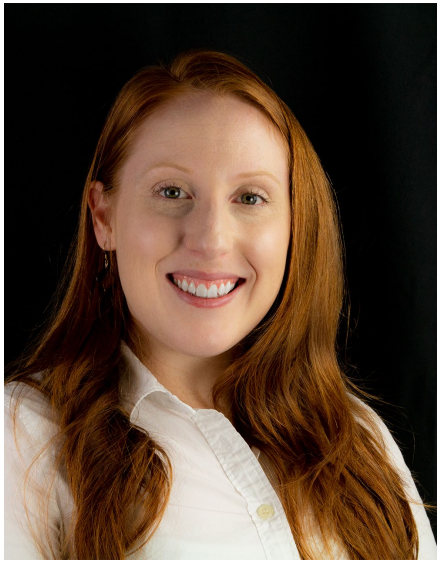
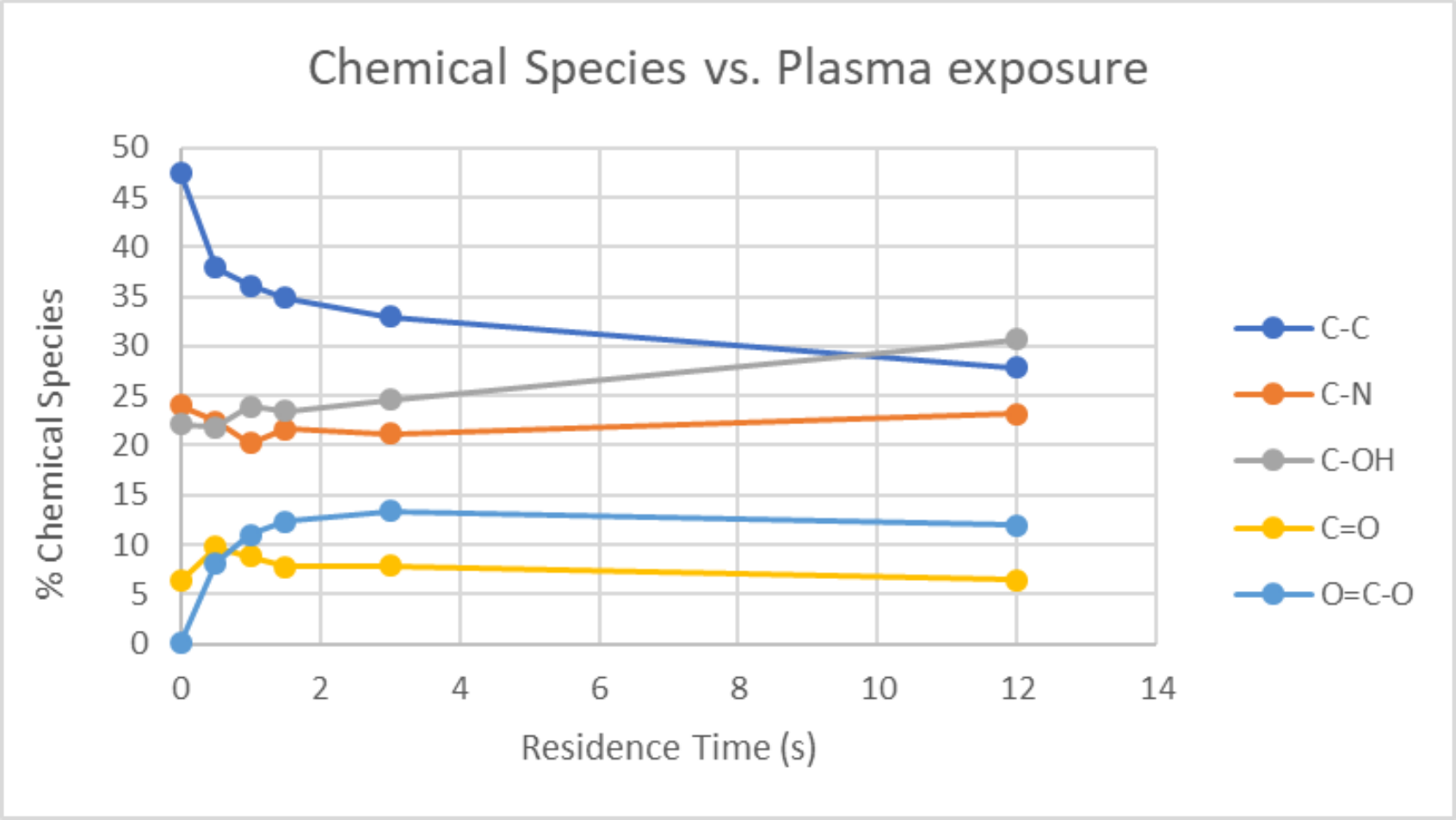
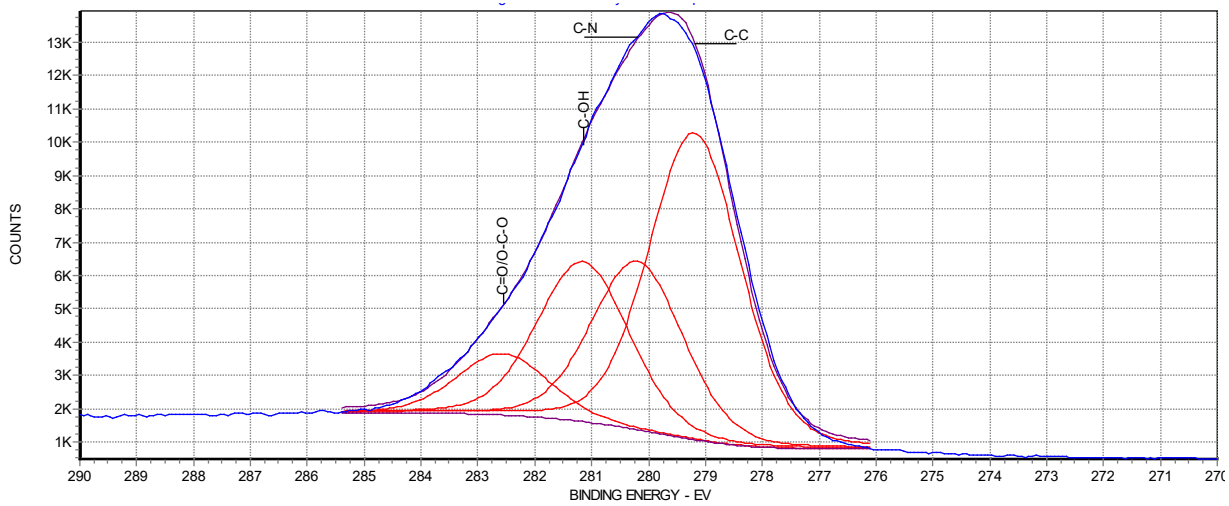


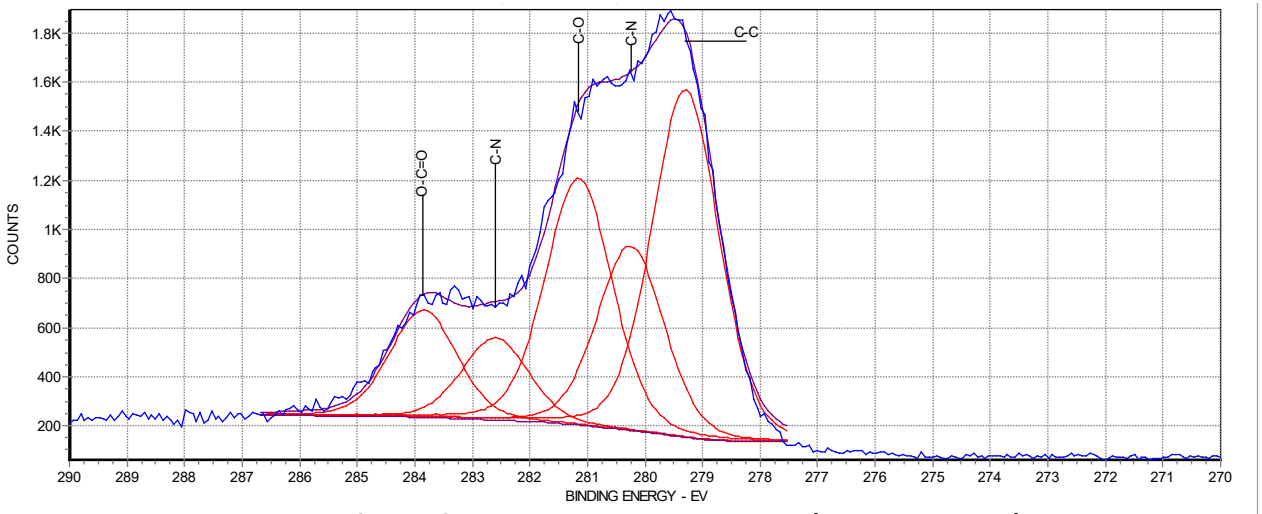
# Plasma Treatment Effect on Surface Chemistry




- Carboxyl group incorporation plateaus after ~1 second
- Hydroxyl group concentration continues to increase

# Plasma Treatment Effect on Surface Chemistry




- Control and 1s exposure time (100 watts)
- Increasing –OH; creating O-C=O (carboxylic acid)

# Repeatable

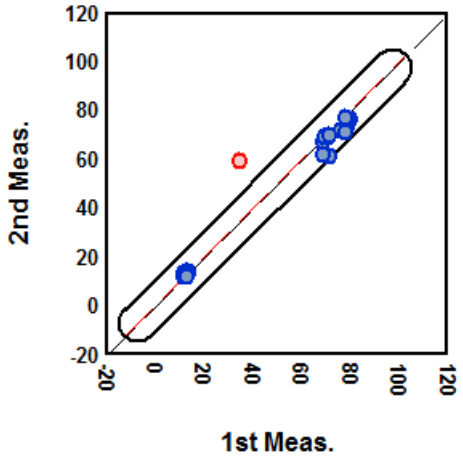
- Passes Gage R&R
- Quantitative and objective measurement

Operator to operator correlation on prepared and unprepared:

**Aluminum**

**Isoplot**

Surface Analyst Gage – Aluminum
A1007

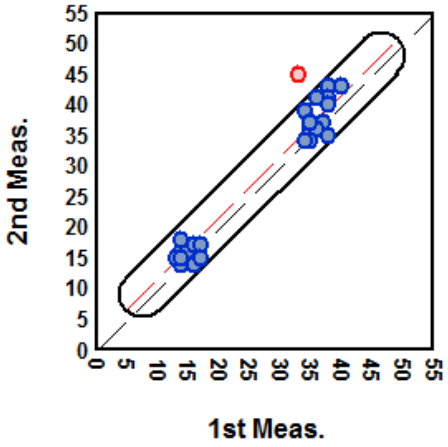



Delta P = 115
Delta M = 16
Discrimination Ratio = 7.4

**Carbon Fiber Composite**

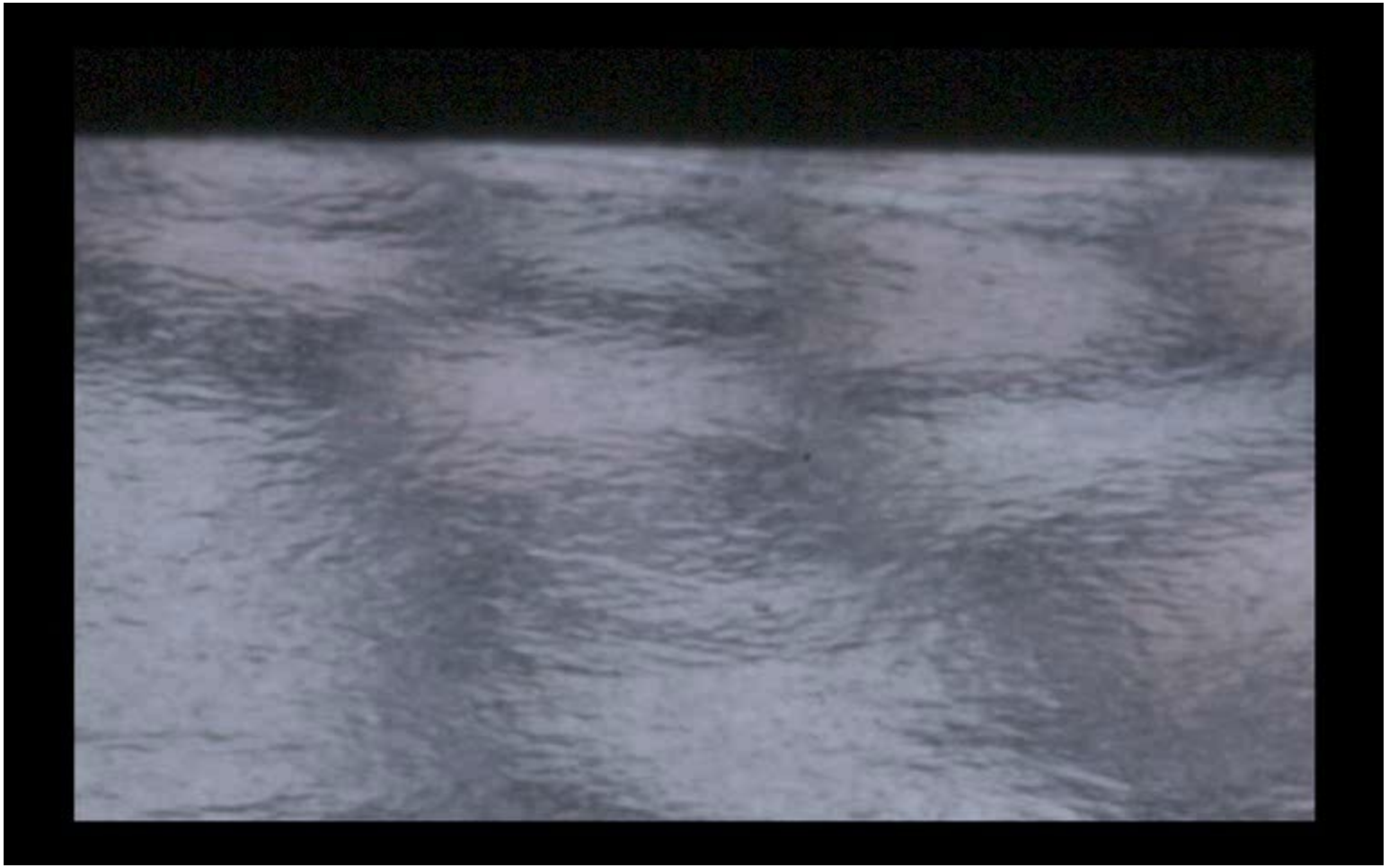
**Isoplot**

Surface Analyst Gage – Composite
A1007



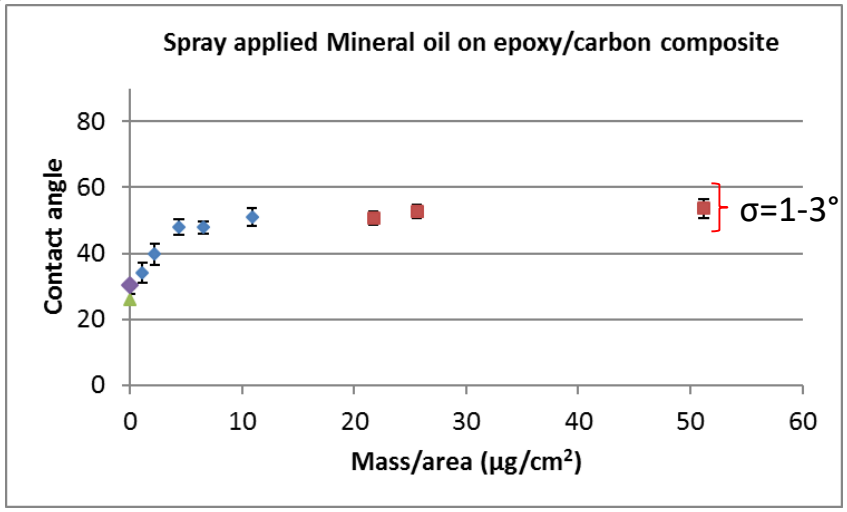

Delta P = 44
Delta M = 8
Discrimination Ratio = 5.7

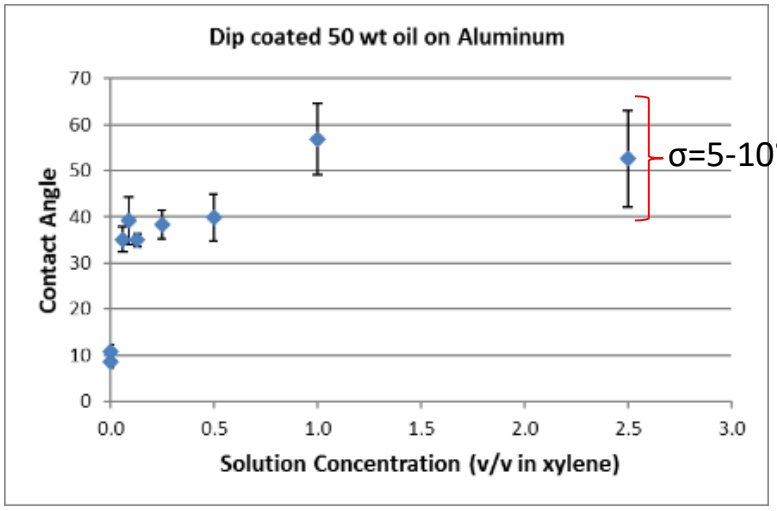
# Ballistic Deposition Video

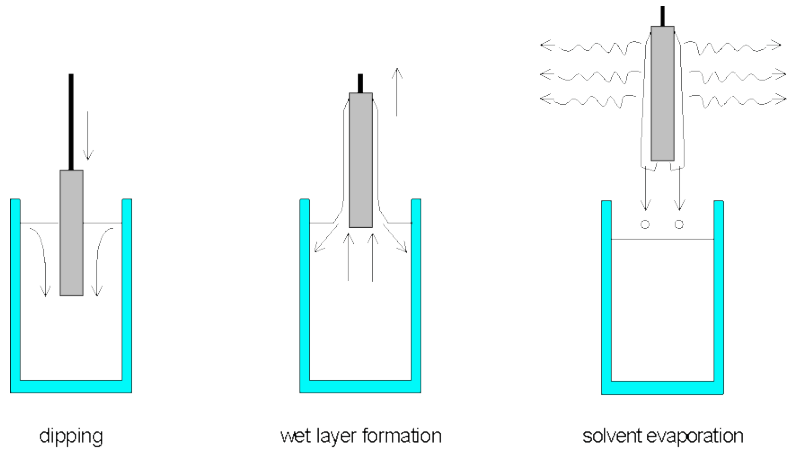


# Ballistic Deposition Video

# Controlled Contamination

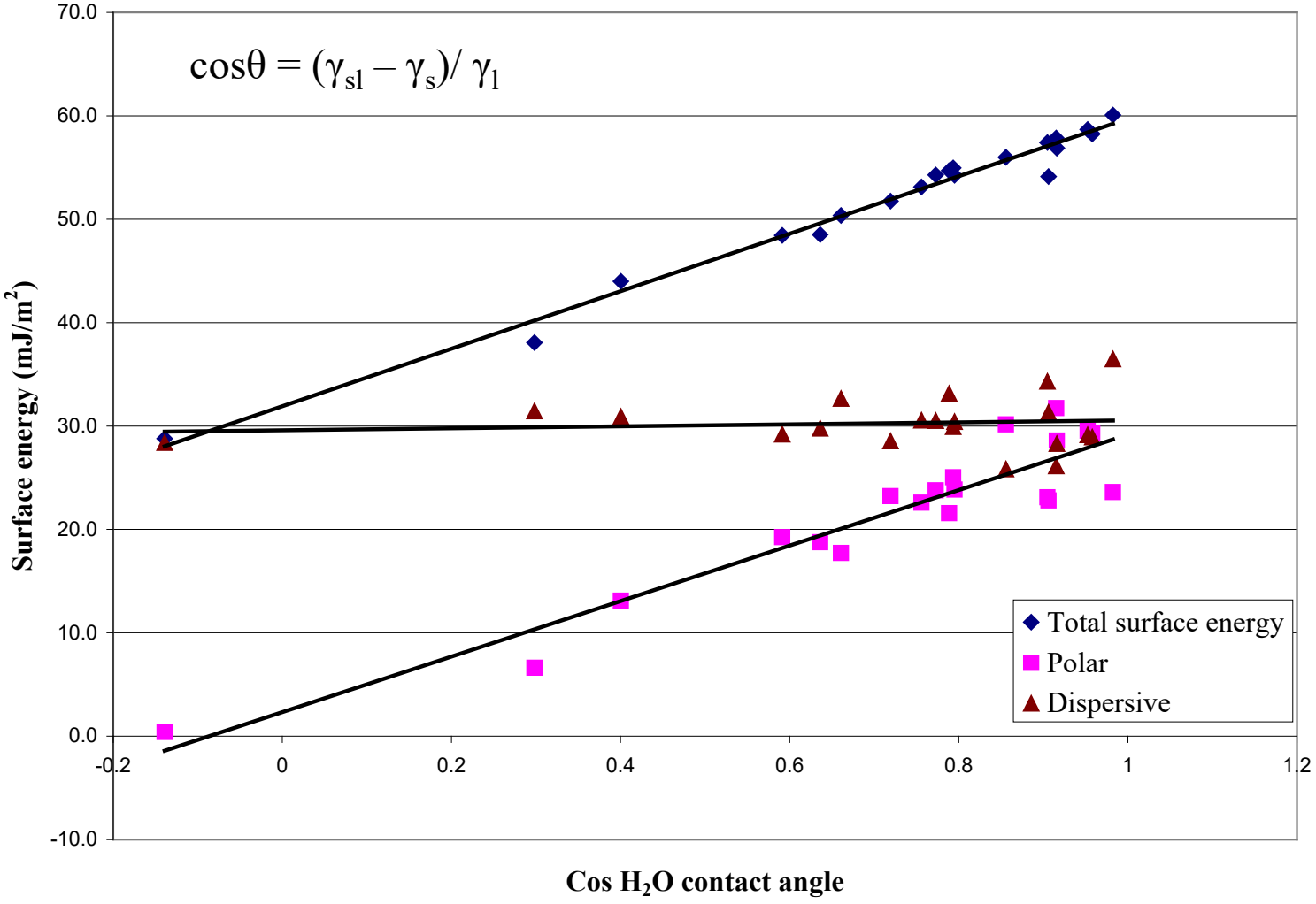


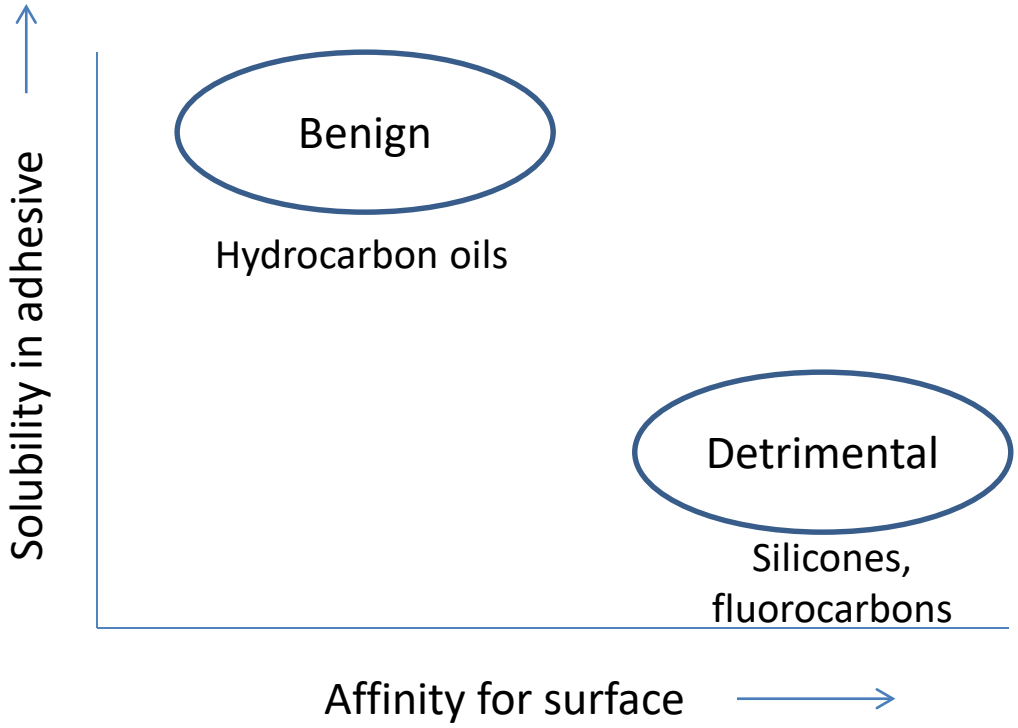

σ is determined by consistency of contaminant:
Uniform surfaces have σ=1-3°

# What Matters When Measuring Surface Energy?




Dillingham, Oakley et al. J. Adhesion Science and Technology V29 9 (2015).

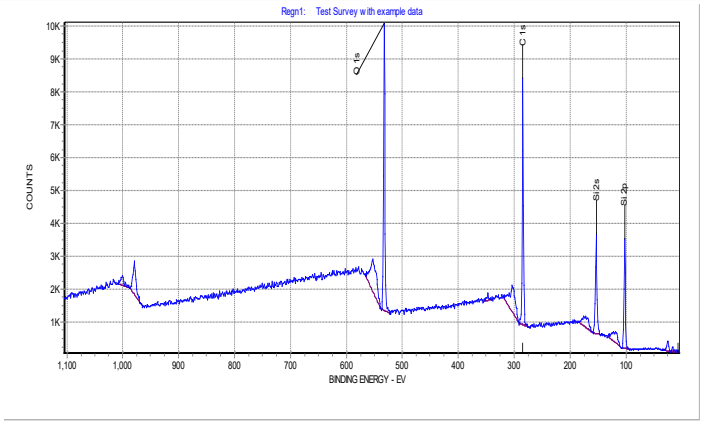
# Contaminant Classification CUT DIS



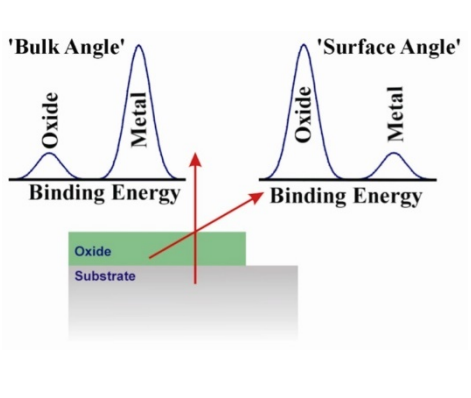

- Surfaces that are prepared for adhesive bonding have high surface energy: they are chemically active and unstable
  - Decay of these surfaces is rapid
  - Rate of decay depends in environment
- Strategies:
  1. Control environment to protect surface from contamination
  2. Apply adhesive before contamination occurs
  3. Use an adhesive system engineered to displace or absorb contaminants

# XPS Analysis Techniques

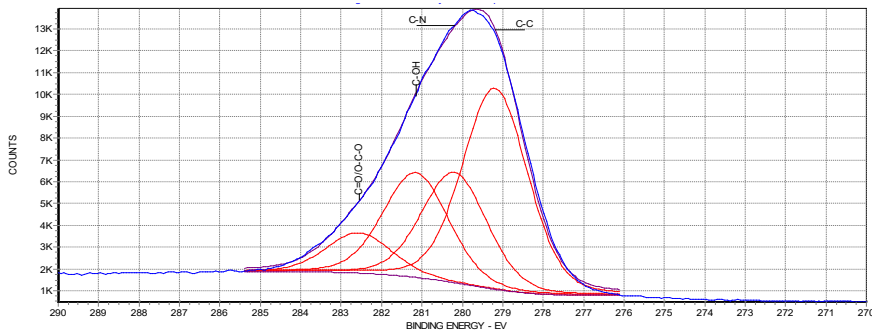
1. Survey Spectral Analysis
   - Scans a broad range of binding energies
   - Quantify elements present on surface




1. Depth Profiling (2 methods)
   - Angle Resolved X-Ray Photoelectron Spectroscopy (ARXPS)
   - Ion Beam Etching

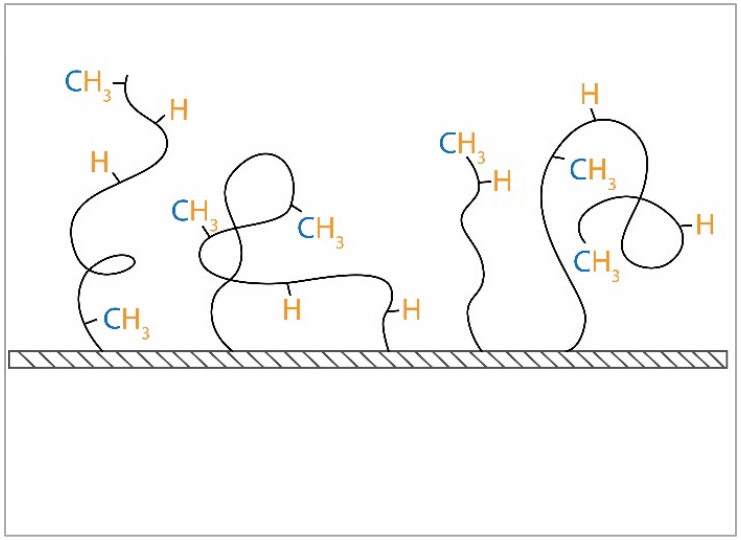



2. High Resolution Spectral Analysis
   - High res scan of small energy range
   - Identify the small shifts in binding energies that accompany formation of chemical bonds.

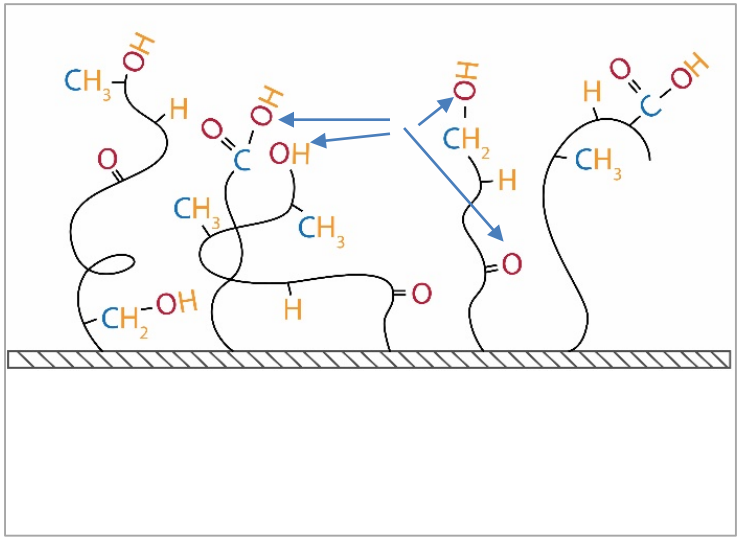


# Polymer Surface: Treated & Aged

**Untreated Polymer Surface**

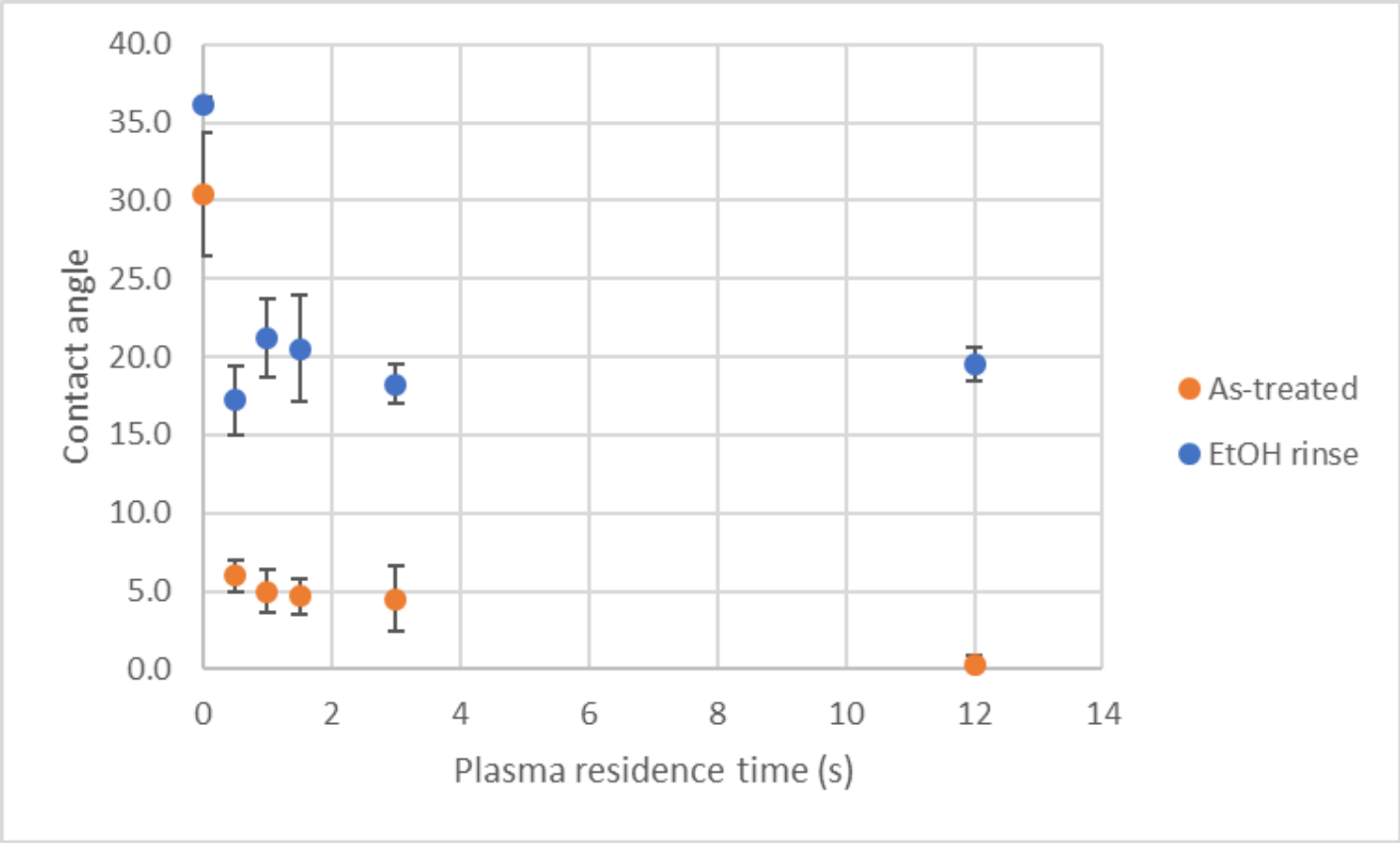


**Treated Polymer Surface**



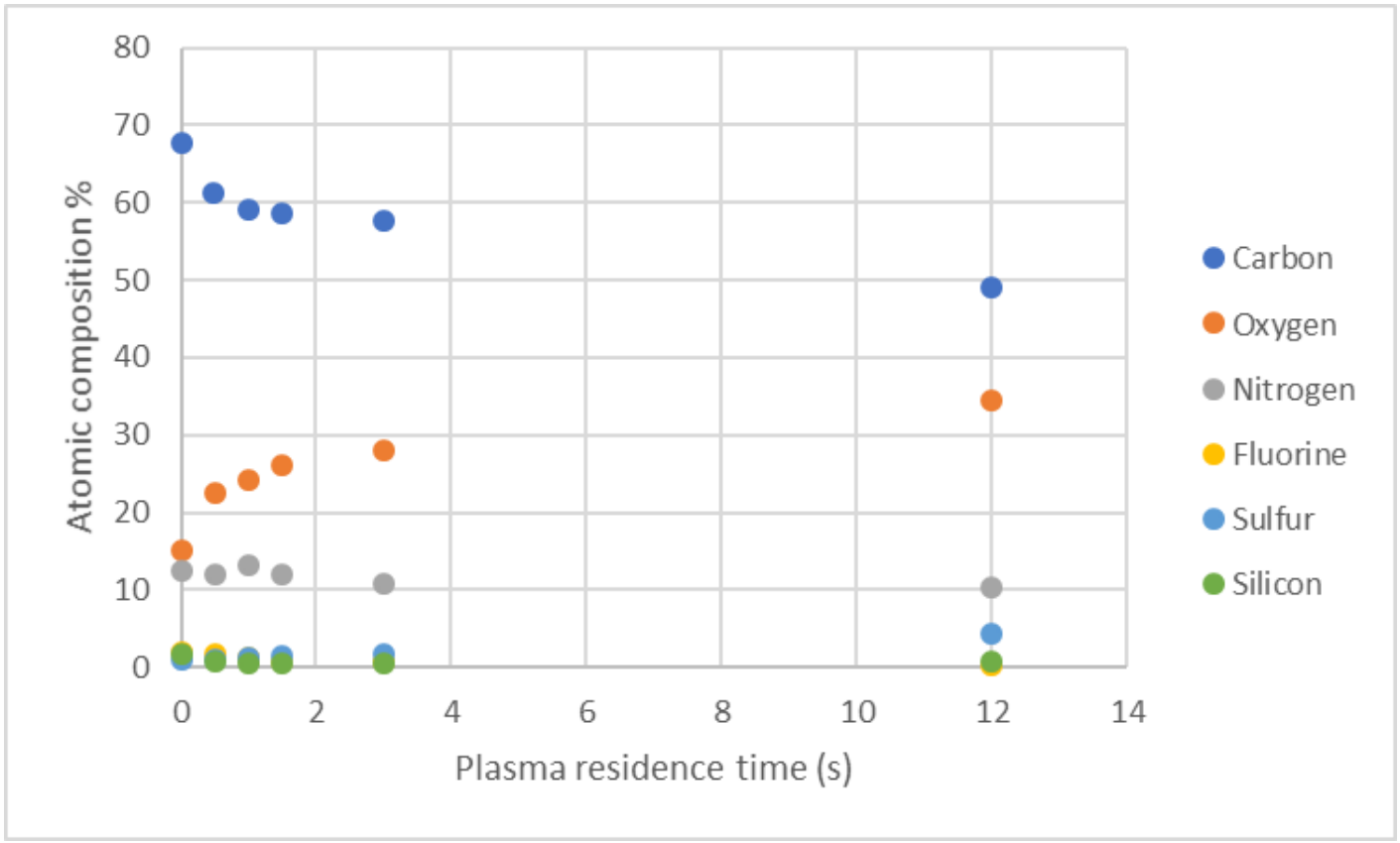
- Polymers have non-reactive, low energy surfaces
- Treatment frequently involves oxidation (corona, flame, plasma) to increase polarity and reactivity
- These reactive groups provide attachment sites for adhesives, inks, coatings
- Surface energy is a measure of how reactive the surface is (type and density of polar and reactive groups)

# Characterizing Plasma Treatment




- Contact angle continues to decrease with treatment time up to ~12s
- Treatment creates low molecular weight material that is soluble in ethanol
  - Equilibrium levels of bound, oxidized material that can enhance adhesion reaches a maximum after ~0.5-1 second exposure
  - Depending on adhesive,

# Determine Optimum Residence Time




Oxygen incorporation is approximately linear with log time

-Diminishing returns for exposures > 2s

How long will it take to achieve optimum surface chemistry?

# XPS: Intro to Theory

Special form of photoemission:

Ejection of a core-level electron by an X-ray photon

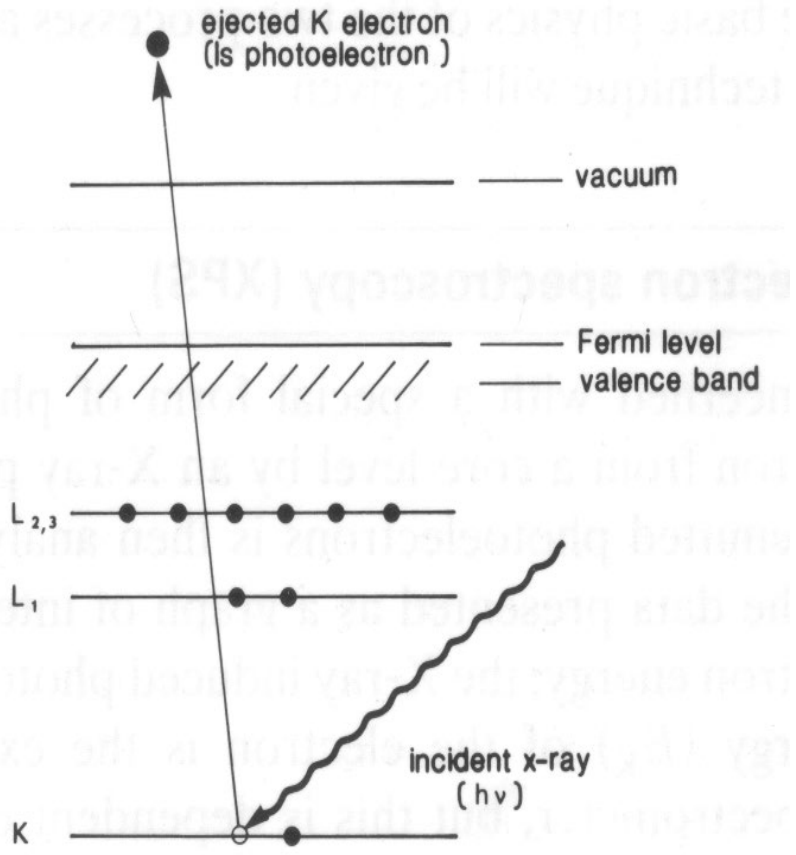
$E_K$ = electron kinetic energy
$h\nu$ = photon energy
$\Phi$ = spectrometer work function

$$E_B = \hbar\nu - E_K - \phi$$

$E_B$:

1. Identify elements
2. Quantify elements
3. Chemical state

All within 5-10 nM of surface



# Detection of Siloxane on Bond Surface via Surface Analyst™