

# Surface Engineering Techniques

Jeff Chinn, Ph.D.  
Chief Technical Officer

# Outline

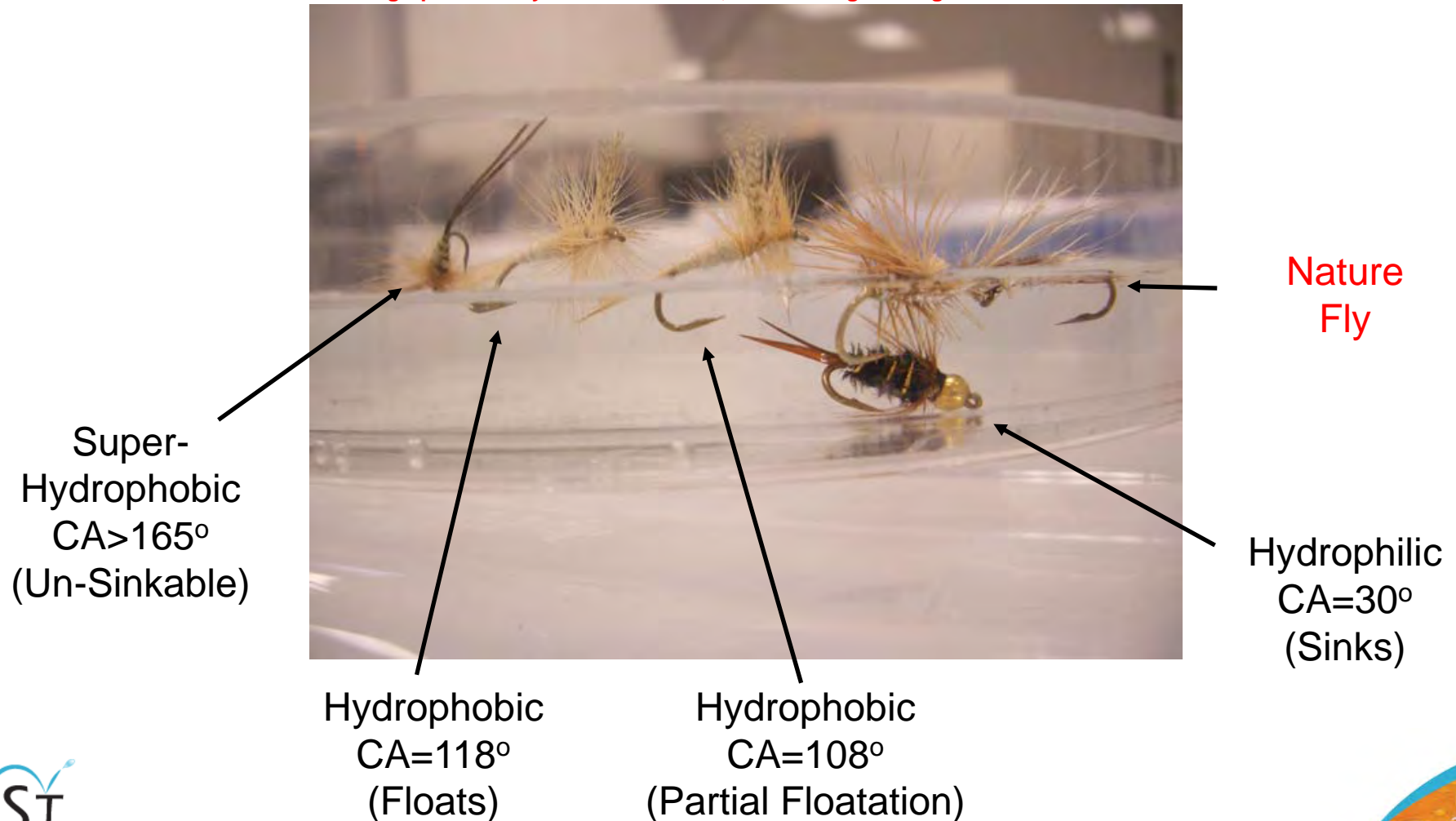
- Brief Definitions and Uses of Surface Engineering
- Who We Are
- Surface Modification Science
- Surface Reactions → Nano-Composites
- IST's as a Supplier
- Summary



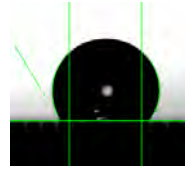
# Surface Engineering

- Changing surface characteristics without altering “Bulk” material traits.
- Ability to create custom materials with a wide range of functionalities.

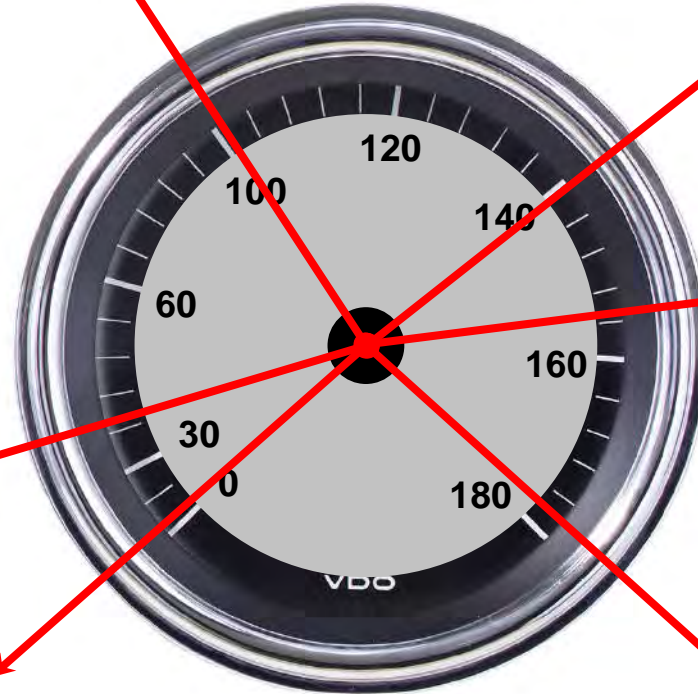
Photograph courtesy of Tom Peterson, Caldera Engineering



# Goniometer (Water Contact Angle)



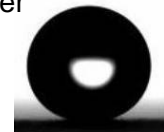
**Hydrophobic**  
Water "balls" but wets



**Oleophobic**  
Oil "balls"

**Super-Hydrophobic**  
Water bounces off  
"Air Boundary Layer"

**Hydrophilic**  
Water "Sheets"

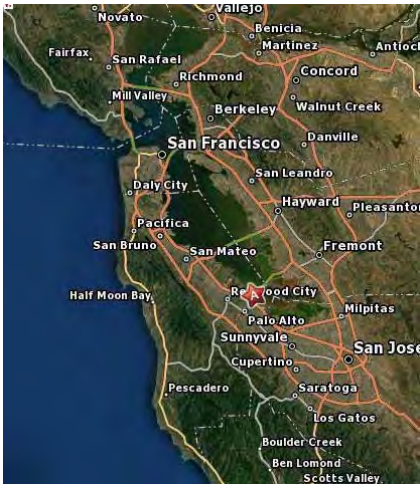


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# More on IST



**Menlo Park, CA**

**Located in the heart of former "Silicon Valley"**



**Demo Lab**



**Chemical Service**



**Metrology**

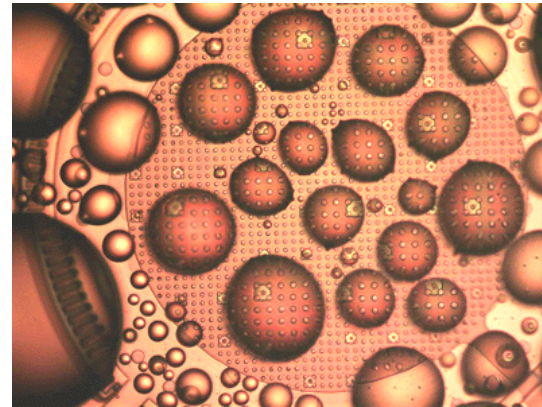
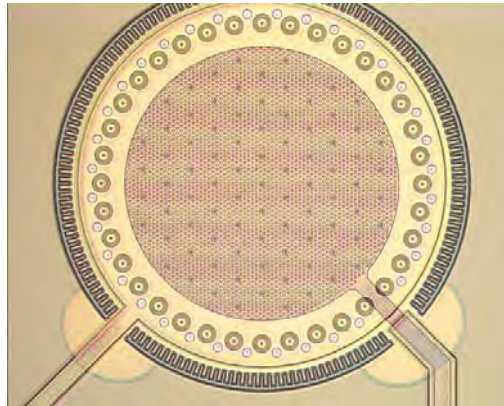


**Manufacturing**



# 3 - Core Capabilities

- **Capability-1: State-of-the-art Advanced Surface Modification** with the lowest **Cost of Ownership** in the industry.

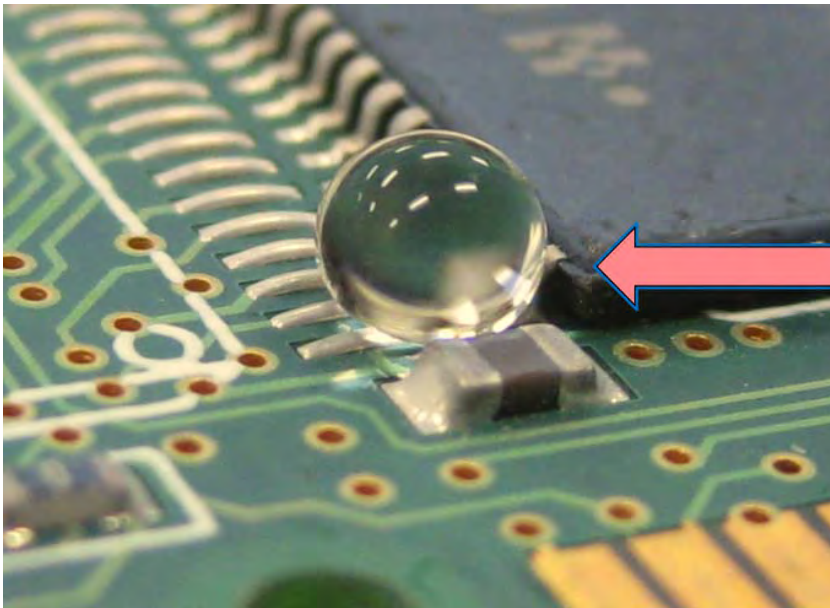


**MEMS based  
Microphones  
Hydrophobic Coating**



# 3 - Core Capabilities

- **Capability-1:** State-of-the-art Advanced **Surface Modification** with the lowest **Cost of Ownership** in the industry.
- **Capability-2:** A nano-composite coating that repels water but is penetrated by the connectors for perfect electrical contact.



**Water cannot  
wet the treated  
surface !**



# 3 - Core Capabilities

- **Capability-1:** State-of-the-art Advanced **Surface Modification** with the lowest **Cost of Ownership** in the industry.
- **Capability-2:** A nano-composite coating that repels water but is penetrated by the connectors for perfect electrical contact.
- **Capability-3:** Supplier for coating equipment & chemicals, specializing in vapor processing.



Plasma

Equipment  
ALD/CVD



Chemicals



Consumables



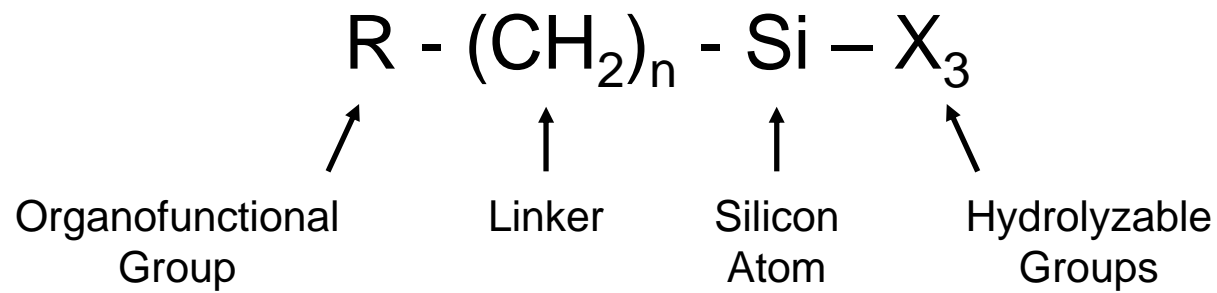
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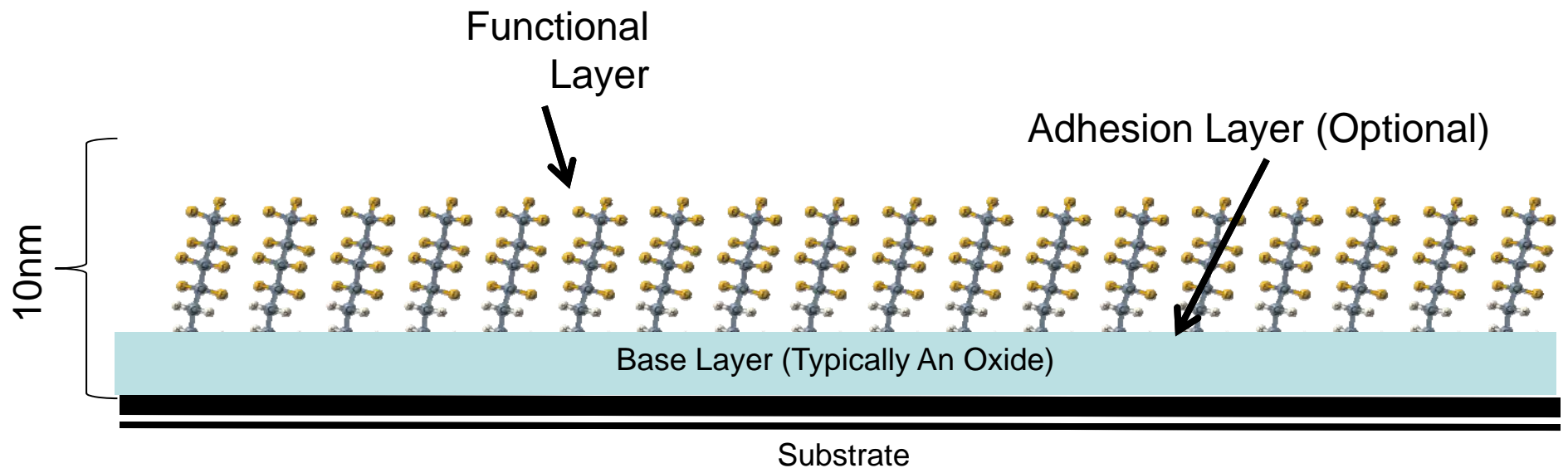
# Basics of BiFunctional Organic Precursors

- Coupling agents used to achieve control over the surface functionality and “coverage”.

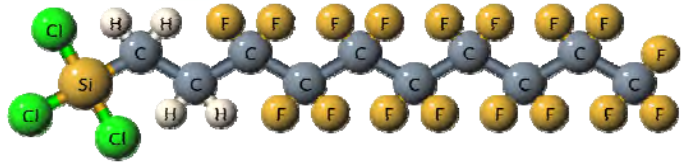


# Advanced Surface Modification

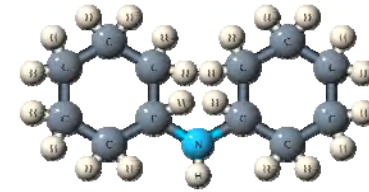
- Vapor deposited Organic
- Global Coverage
- Choices of functional chemistry
- Inert: Hydrophobic (Perfluorinated (Teflon))
- Inert: Hydrophilic (Poly Ethylene Glycol)
- Reactive: Amine or Epoxy



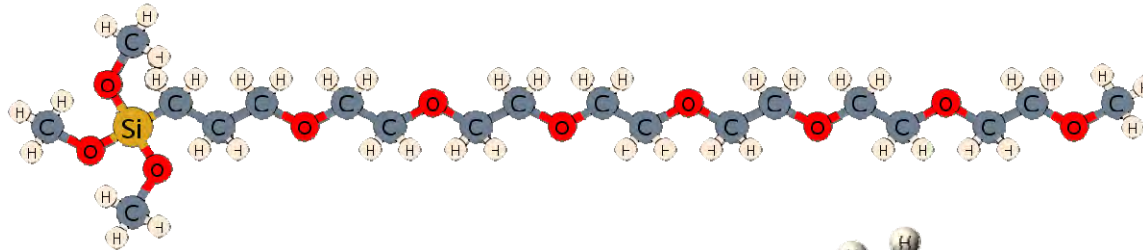
# Most Common Silanes for Surface Modification



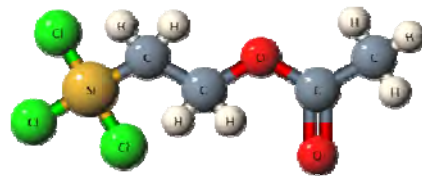
FDTM  
Hydrophobic (110°)



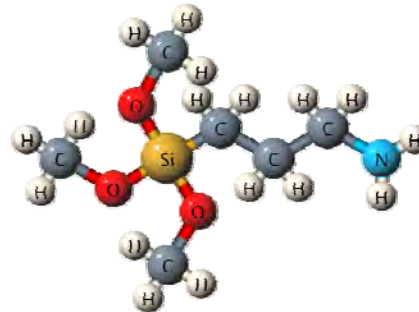
DCHA  
Neutral-  
Hydrophobic (80°)



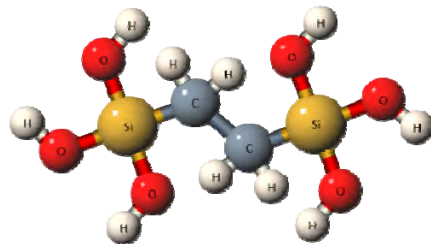
PEG  
Hydrophilic (45°)



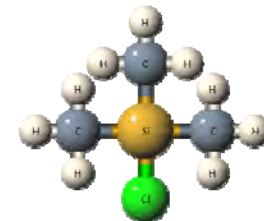
AETCS  
Hydrophilic (65°)



APTMS  
Reactive (50-60°)



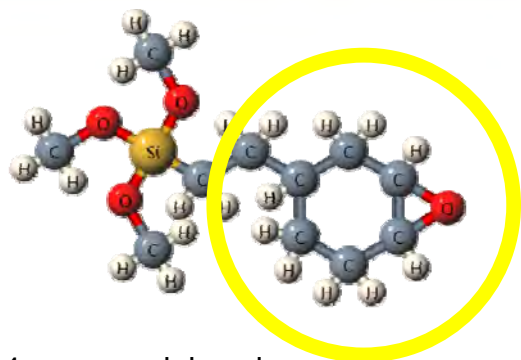
BTCSE  
Hydrophilic (30°)



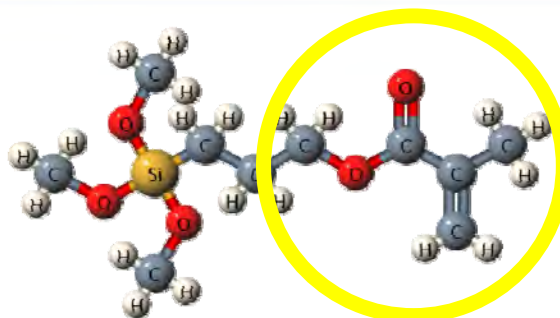
TMS  
Hydrophobic (85°)



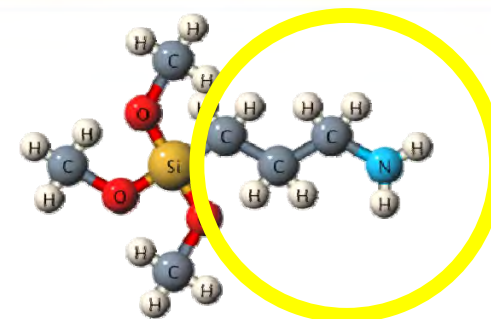
# Most Common Linking Precursors



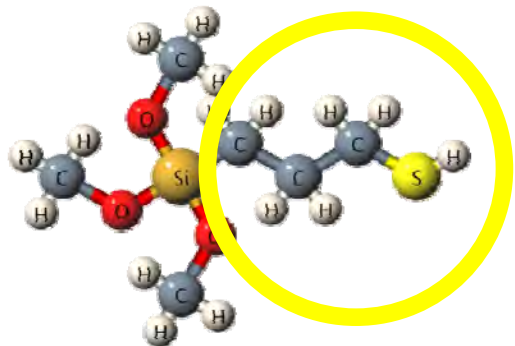
3-4-epoxycyclohexyl-  
ethyltrimethoxysilane



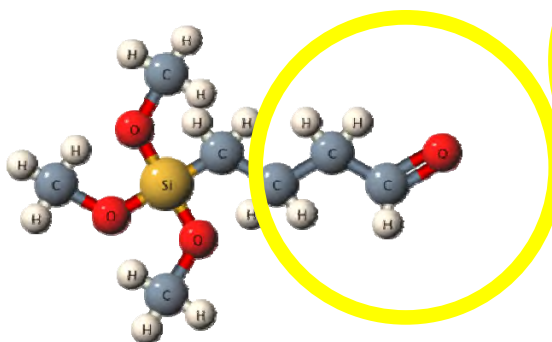
3-methacryloxy  
propyltrimethoxysilane



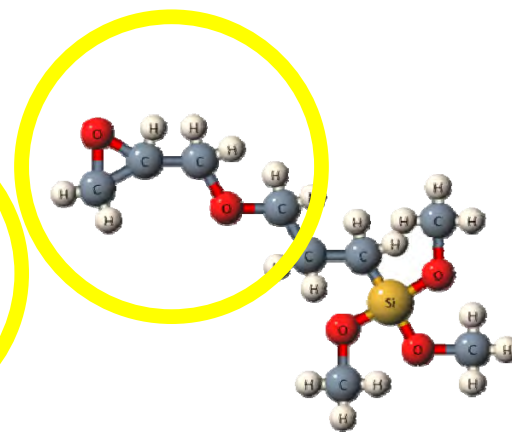
aminopropyltrimethoxysilane



mercaptopropyltrimethoxysilane



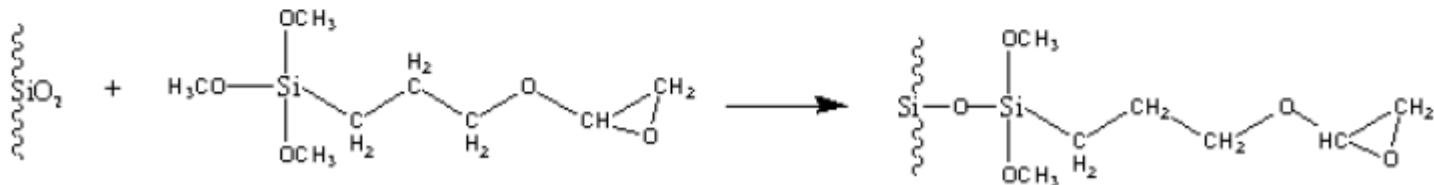
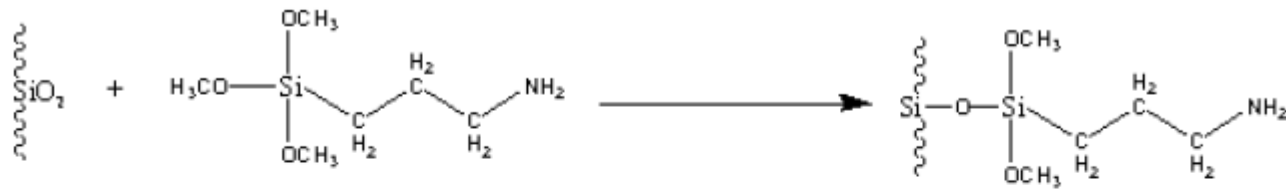
Butyl-Aldehyde-  
Trimethoxysilane



Glycidyl-  
Trimethoxysilane



# Biological Binding

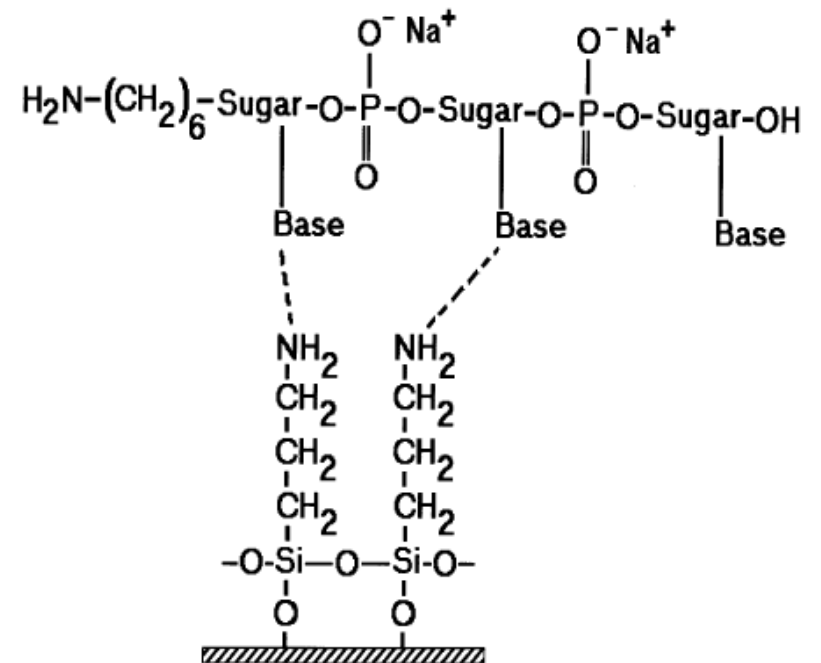
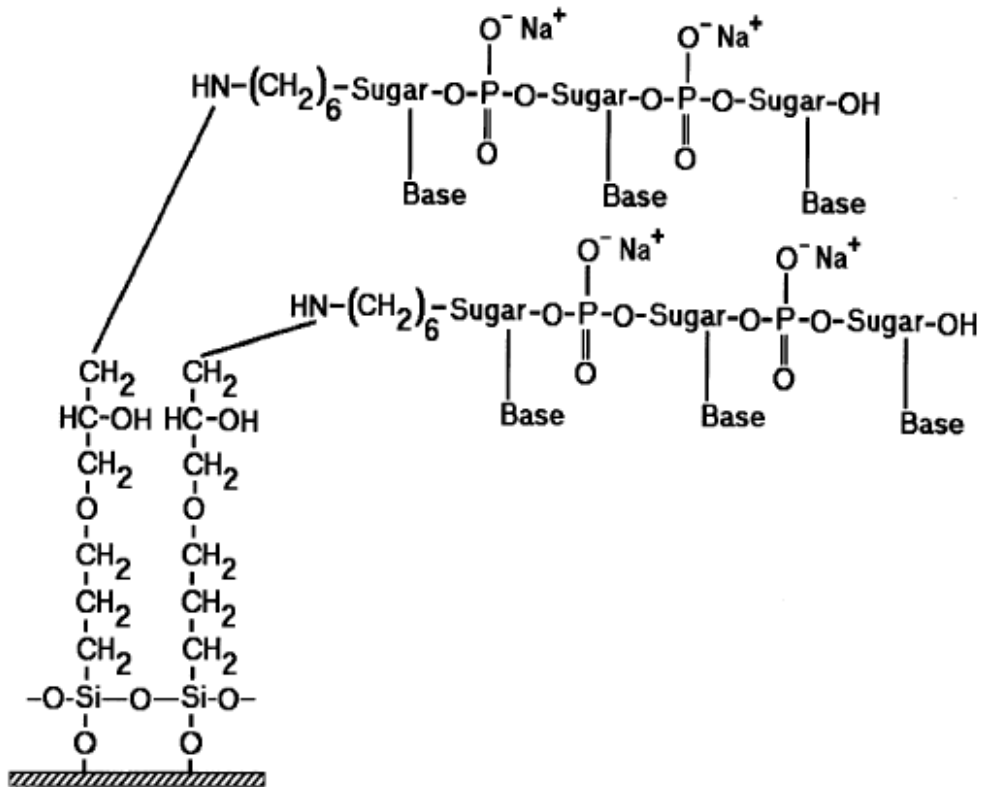


## Covalent Coupling Reactions

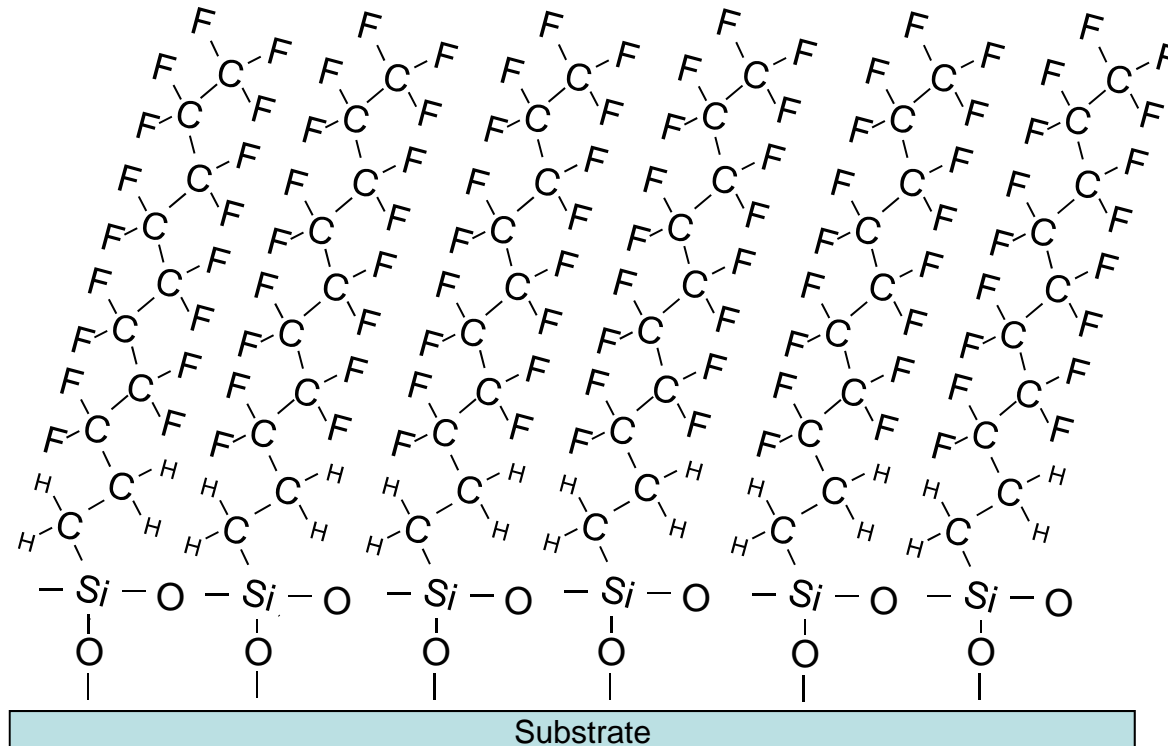
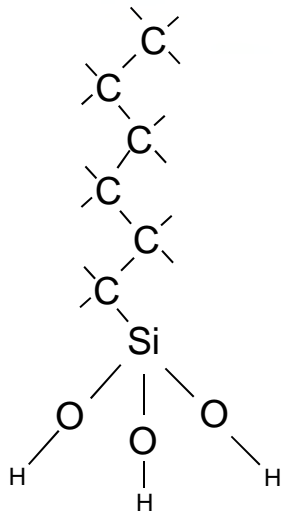
|                      |                         |
|----------------------|-------------------------|
| -COOH                | Carboxylic Acid         |
| -RNH <sub>2</sub>    | Primary Aliphatic Amine |
| -ArNH <sub>2</sub>   | Aromatic Amine          |
| -CONH <sub>2</sub>   | Amide                   |
| -CONHNH <sub>2</sub> | Hydrazide               |
| -CHO                 | Aldehyde                |
| -OH                  | Hydroxyl                |
| -SH                  | Thiol                   |
| -COC-                | Epoxy                   |



# DNA Surface Immobilization



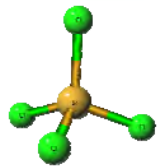
# Organic Surface Modification



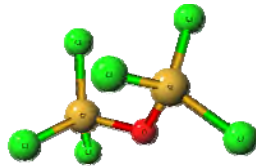
Hydroxyl surface linkages are formed.  
Molecule attaches to the substrate via covalent bonding.



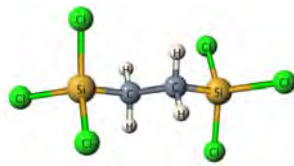
# Surface Chemistry Effects



$\sim 5^\circ$



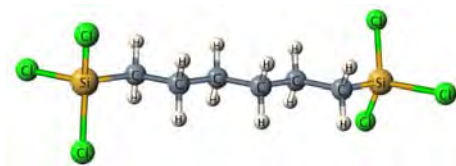
$\sim 20^\circ$



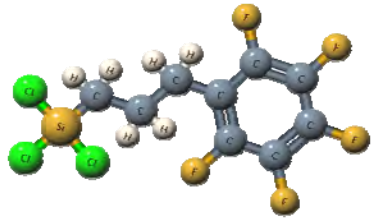
$\sim 30^\circ$



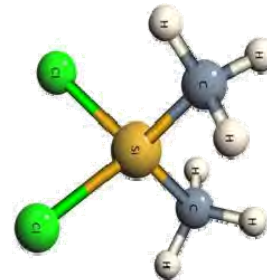
$\sim 50^\circ$



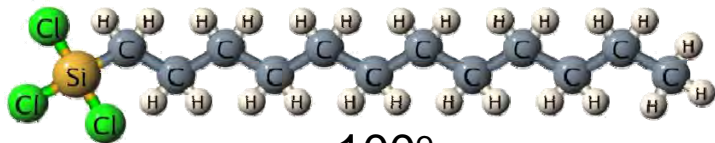
$\sim 70^\circ$



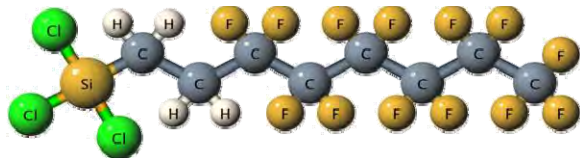
$\sim 90^\circ$



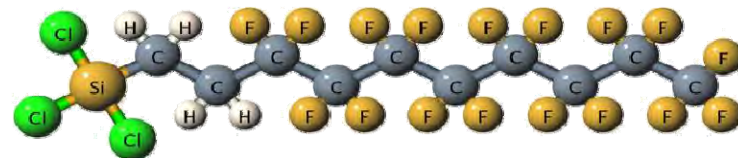
$\sim 103^\circ$



$\sim 100^\circ$



$\sim 108^\circ$



$\sim 110^\circ$

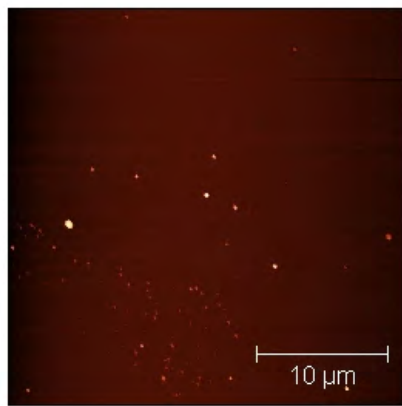


# Hydrophobicity Function of Surface Fluorine

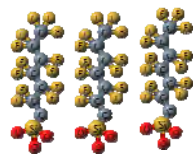
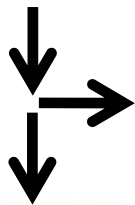
| Precursor Name   | Water Contact Angle (degs) |
|--|----------------------------|
| Henicosyl-1,1,2,2-tetrahydrododecyldimethyltris(dimethylaminosilane)       | 118.0                      |
| Heptadecafluoro-1,1,2,2-tetrahydrodecyltrichlorosilane - (FDTS)            | 110.0                      |
| Nonafluoro-1,1,2,2-tetrahydrohexyltris(dimethylamino)silane                | 110.0                      |
| 3,3,3,4,4,5,5,6,6-Nonafluorohexyltrichlorosilane                           | 108.0                      |
| Tridecafluoro-1,1,2,2-tetrahydrooctyltrichlorosilane - (FOTS)              | 108.0                      |
| BIS(Tridecafluoro-1,1,2,2-tetrahydrooctyl)dimethylsiloxymethylchlorosilane | 107.0                      |
| Dodecyltrichlorosilane – (DDTS)  | 105.0                      |
| Dimethyldichlorosilane - (DDMS)  | 103.0                      |
| 10-Undecenyltrichlorosilane - (V11)  | 100.0                      |
| Pentafluorophenylpropyltrichlorosilane                                     | 90.0                       |



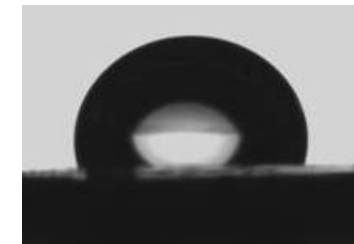
# Surface "Smoothness" Effects



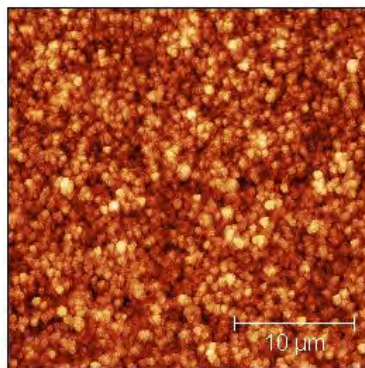
RMS = 3.5 nm



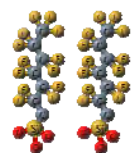
$\theta = 110^\circ$   
Smooth  
Organic Monolayer



Hydrophobic



RMS = 107 nm



$\theta = 165^\circ$   
Rough Surface  
Organic Monolayer



Superhydrophobic



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# An Advanced Coating Technology

- A room-temperature, vapor deposited, textured ceramic coating with superior anti-wetting and anti-corrosion protection for Printed Circuit Boards.

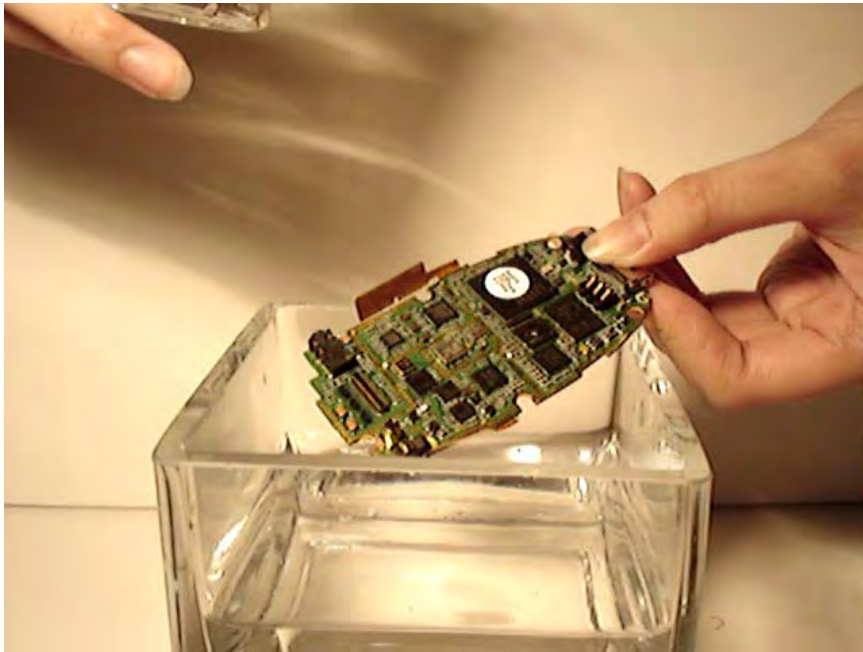


IST's Repellix™ coating creates a barrier film that protects the circuit board from water damage.



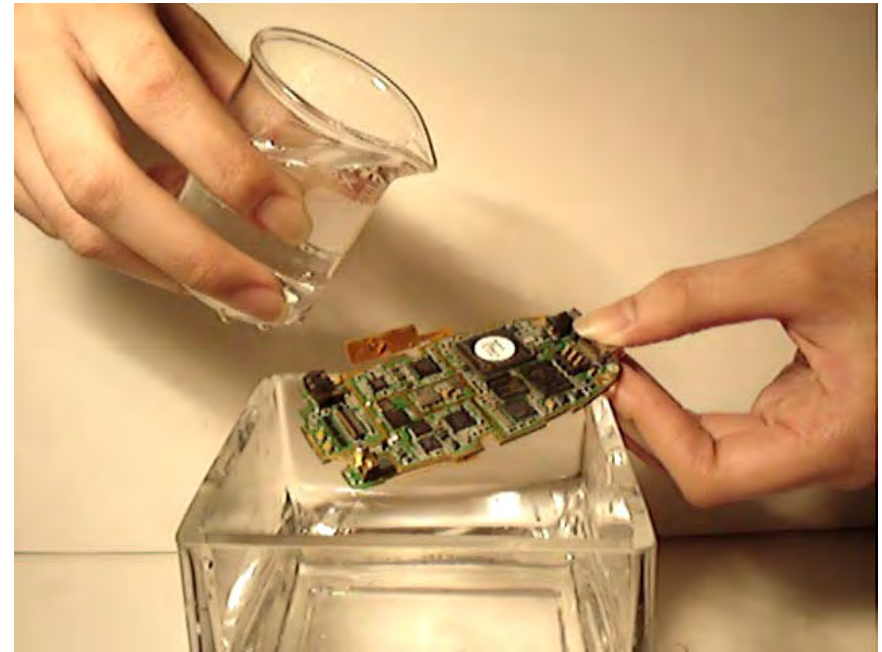
# Repellix Wetting Demo

Water on an **Uncoated**  
Cell Phone Board



The uncoated board spreads the water  
across the board

Water on a **Repellix**  
**Coated** Cell Phone Board



The Repellix coated board sheds the water,  
and creates a narrow stream



# Active Electronics

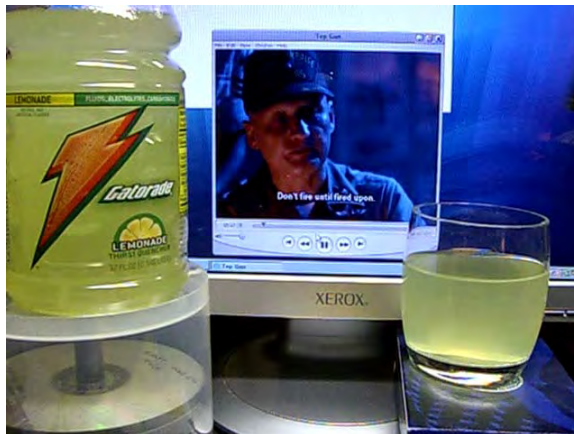
**Gatorade = Potassium Phosphate + Citric Acid (Electrolyte (Salt) Solution)**



**Standard Memory Board**



**Coated Board**



**Memory Failed ~2 sec**



# Game Changing Technologies



No Coating

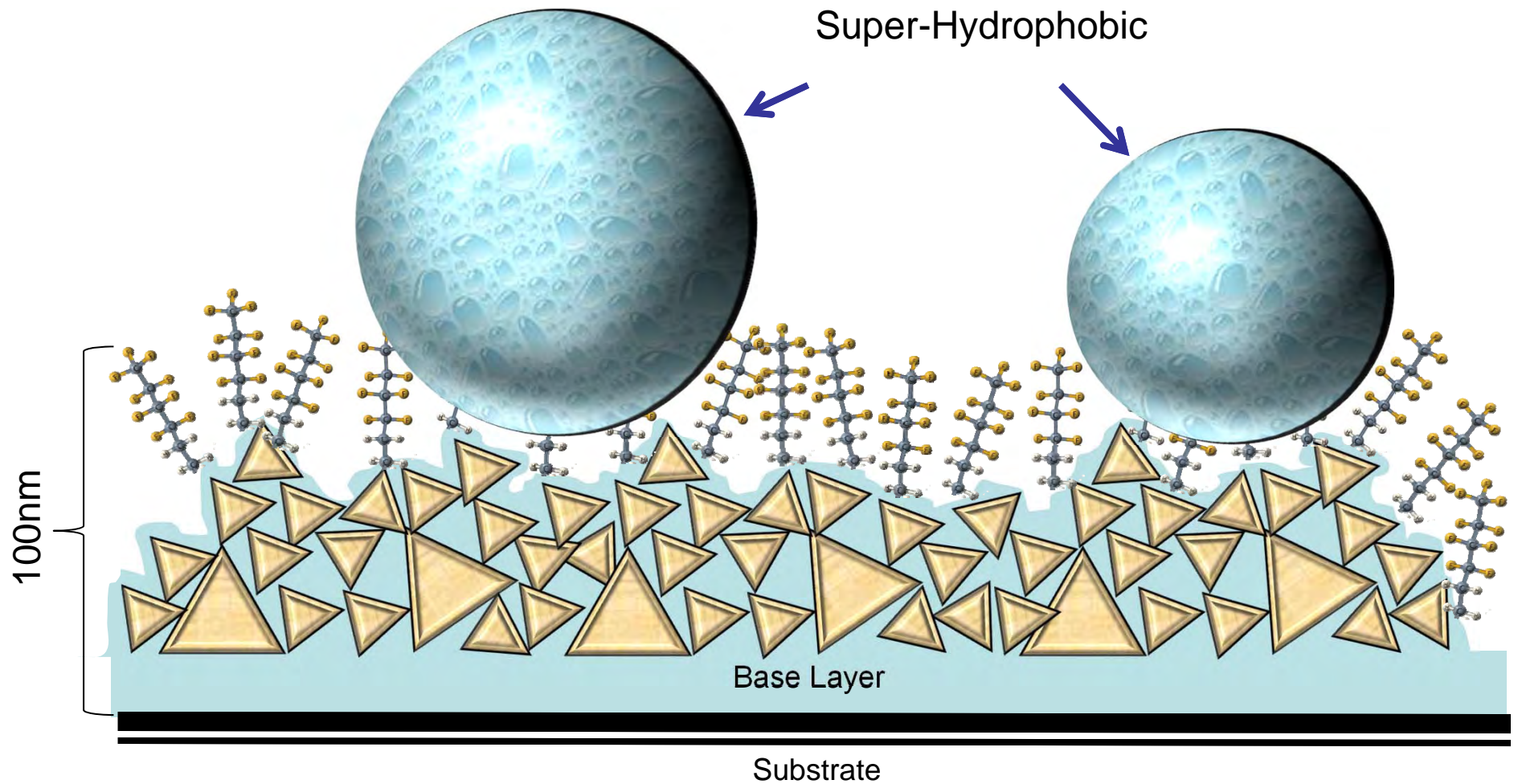


RPX Coated

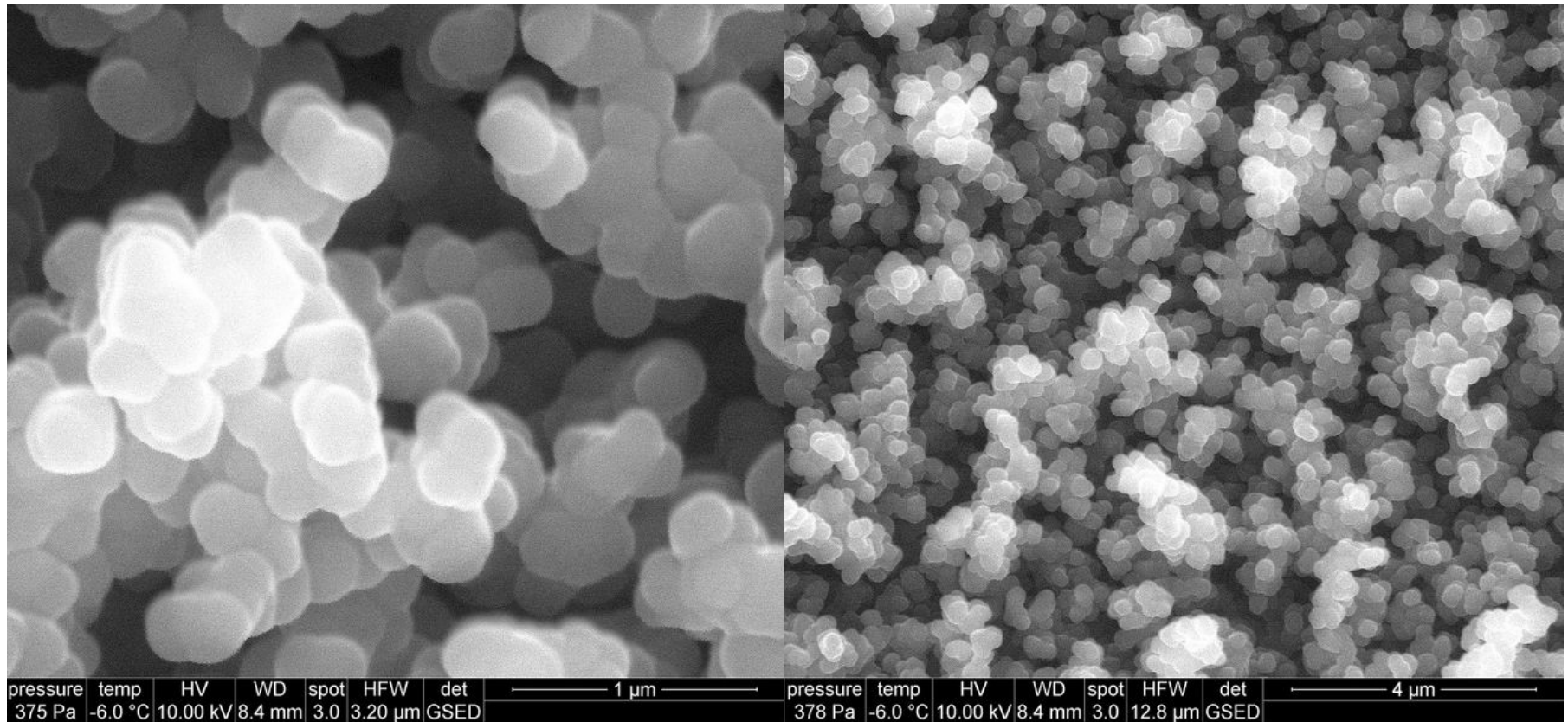
**After Neutral Salt Fog Exposure – 40 hours**



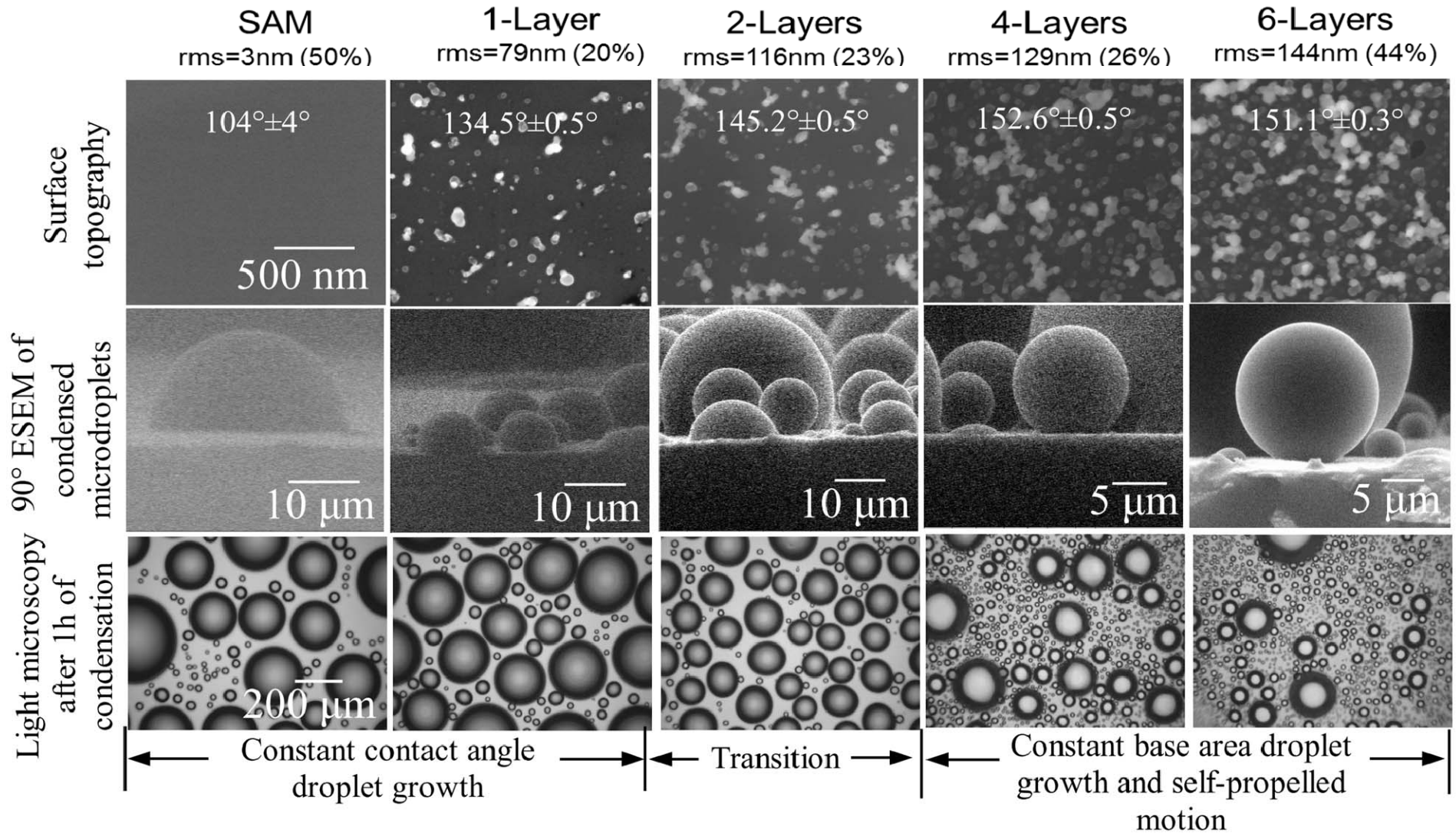
# The Film Properties



# Repellix with 100nm Linkerrix - Unscratched



# Process Layering



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# IST Standard Hardware Platforms



## -210

- Surface Modification

## RPX-540

Additional Options:

- Ozone
- Plasma



## -560

Enhanced Features:

- Dual Injection
- Plasma



## BL1

- Plasma Polymer



|              | BL1<br>(Plasma) | RGM-<br>210 | RPX-<br>540 | RGM-<br>560 |
|--------------|-----------------|-------------|-------------|-------------|
| Width        | 12"             | 12.5"       | 20"         | 20"         |
| Height       | 8"              | 10"         | 12"         | 17"         |
| Depth        | 10"             | 9.6"        | 16"         | 16"         |
| Vol. (liter) | 12              | 20          | 60          | 90          |
| Gases        | 1               | 2 ½         | 5           | 5           |



# RPX-540 Hardware Designs



Ozone

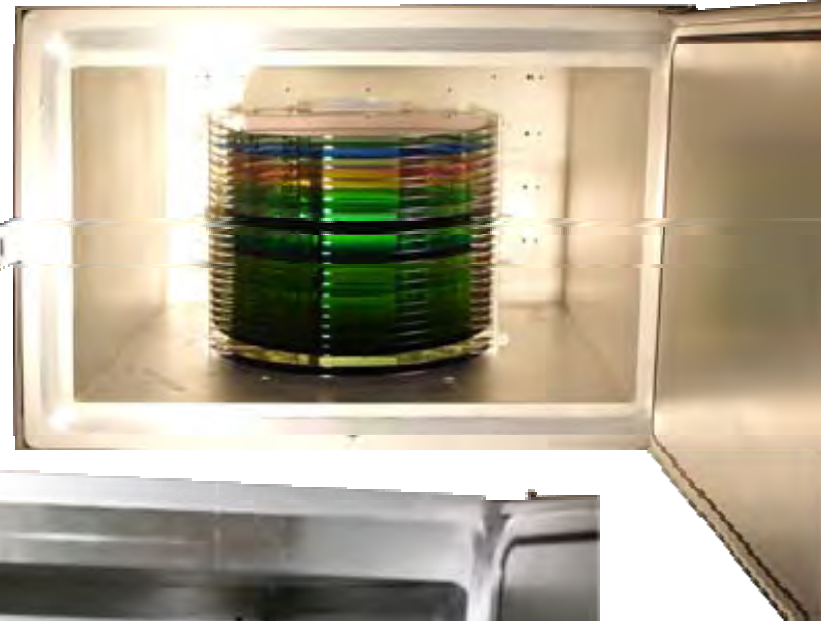


Compact design with pull out electronics modules.



# 300mm Wafers or 15" Laptop

300mm



# 3<sup>rd</sup> Gen : Chemical Delivery



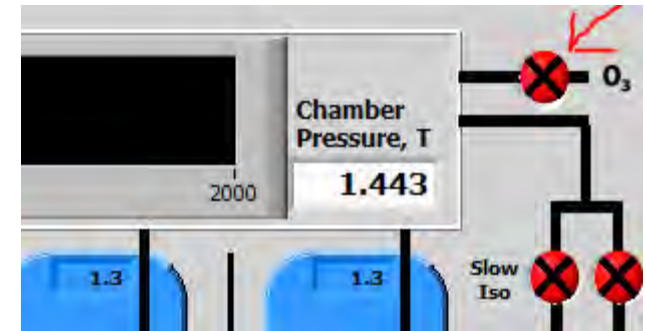
Vaporizers

Interchangeable  
Cartridges

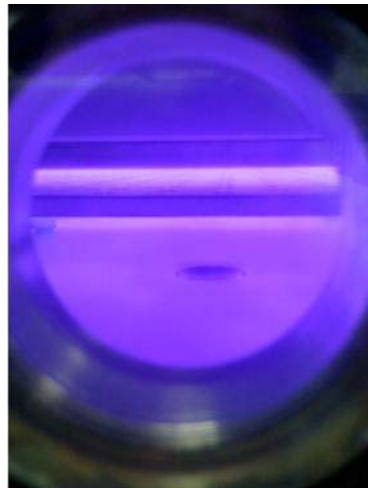


# Fully Integrated Plasma Option

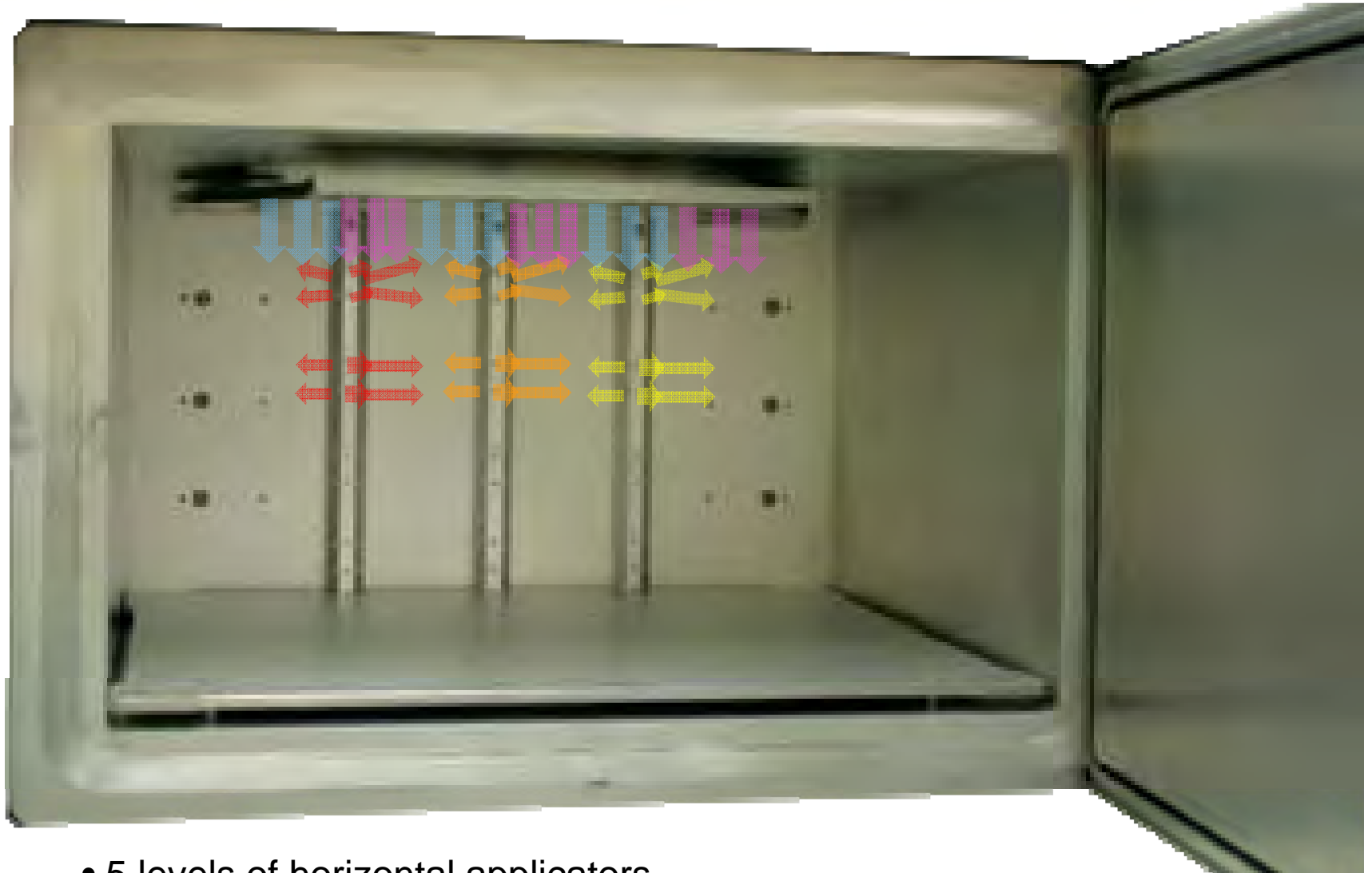
- Safety interlocked
- Use of plasma assisted reactions and surface mixing within individual process steps.
- RF Power (0-1000W) controlled within the process recipe.



| Pressure TimeLim | RF On | RF Power |
|------------------|-------|----------|
|                  | 1     | 200      |
|                  | 1     | 150      |
| 35               |       |          |
|                  | 1     | 50       |
| 60               |       |          |
|                  | 1     | 35       |



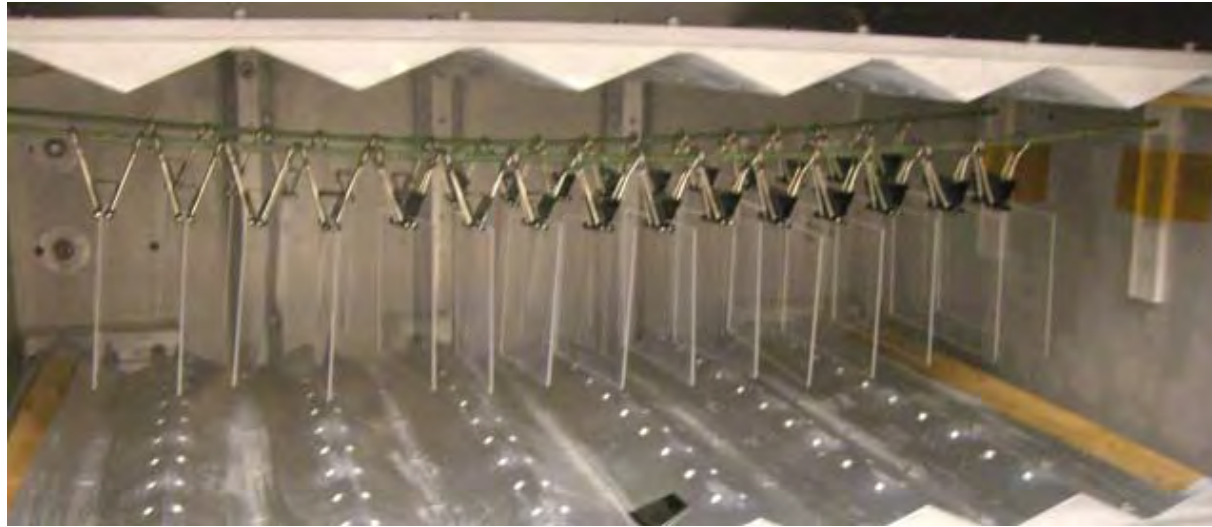
# Multi-Gas Injection Chamber Design



- 5 levels of horizontal applicators
- 3 vertical dispersion towers



# Slides & Cartridges



# The Cartridge – Safe, Reliable, Easy

***Gas Cartridge is designed for a simplistic “plug and play” operation for easy maintenance***

Cartridge Features:

- Precursor Charge
- Manual Shut-off Valve
- Heater
- Insulation
- Temperature Sensor
- Identification Interlock
- Electrical Over-temperature Safety Switch
- Lockout Safety to software



***Each Cartridge is a self contained module delivered***



# Cartridges in Volume



- **Lower COO for our customers**
- **Convenient Recycle Programs**
- **Economic Refilling**

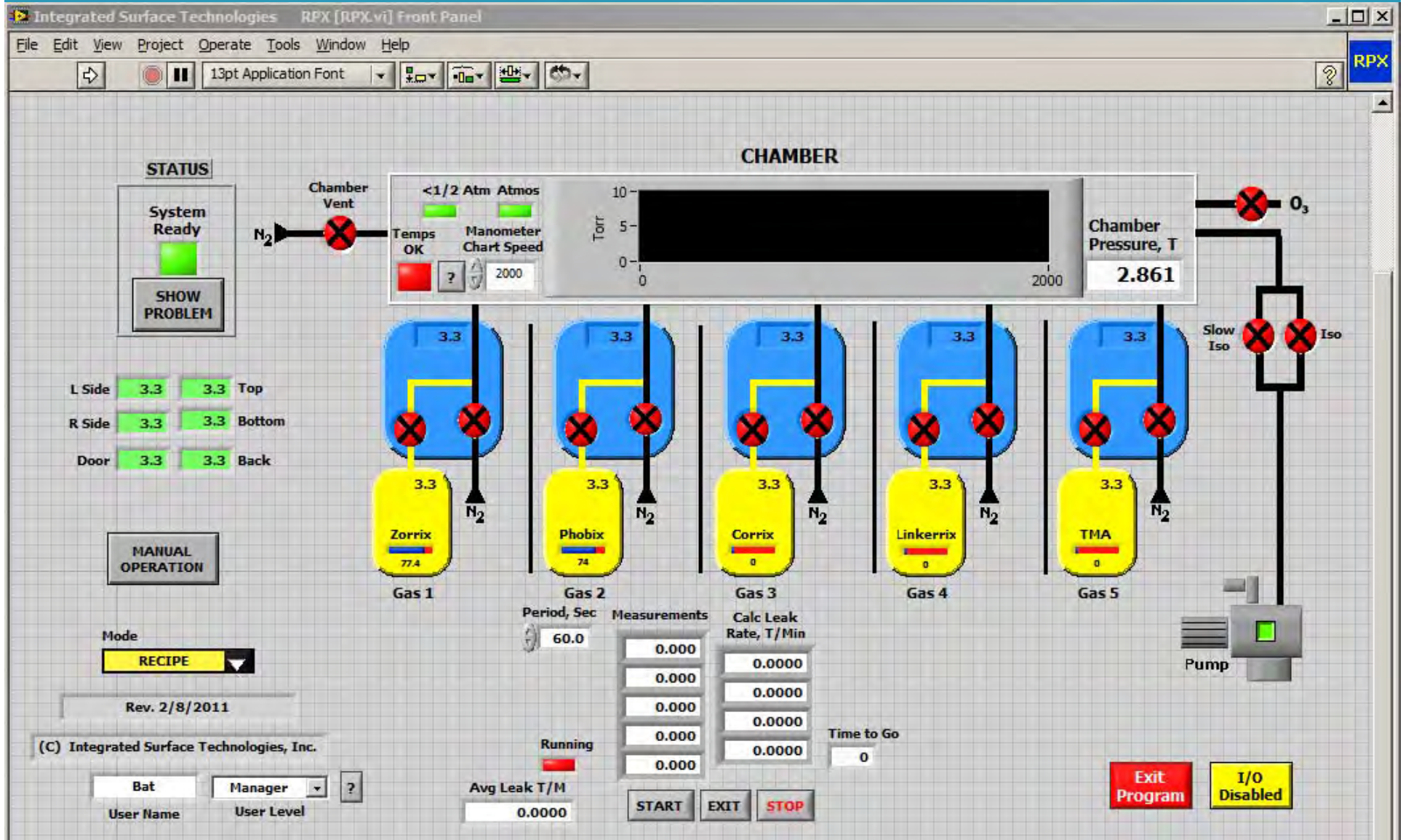


# Chemical Matrix

| Chemical              | Application Type |           |         |          |           |           |                      |             |                |
|-----------------------|------------------|-----------|---------|----------|-----------|-----------|----------------------|-------------|----------------|
|                       | Hydrophobic      | Repellix™ | Bio     | Adhesion | Bio Oxide | ALD Oxide | ALD TiO <sub>x</sub> | ALD Alumina | Anti-Corrosion |
| BCHTCS                |                  |           | •       | •        |           |           |                      |             |                |
| BCH-E-TCS             |                  |           | •       | •        |           |           |                      |             |                |
| C8-silane             | •                |           |         |          |           |           |                      |             |                |
| C12-silane            | •                |           |         |          |           |           |                      |             |                |
| F(C5)TS               | •                |           |         |          |           |           |                      |             |                |
| F(C6)TS               | •                |           |         |          |           |           |                      |             |                |
| F(C8)TS               | •                |           |         |          |           |           |                      |             | •              |
| F(C10)TS              | •                |           | •       |          |           |           |                      |             | •              |
| APTMS                 |                  |           | •       | •        |           |           |                      |             |                |
| BCTSE                 |                  |           | •       | •        | •         |           |                      |             |                |
| Epoxy                 |                  |           | •       | •        |           |           |                      |             |                |
| Glycidoxy             |                  |           | •       | •        |           |           |                      |             |                |
| Linkerrix             |                  | •         |         | •        |           | •         |                      |             |                |
| Phobix                |                  | •         |         |          |           |           |                      |             |                |
| Pyridine              |                  |           |         |          |           | •         |                      |             |                |
| TMA                   |                  | •         |         | •        |           |           |                      | •           |                |
| TIP                   |                  |           |         |          |           |           | •                    |             |                |
| PEG                   |                  |           | •       |          |           |           |                      |             |                |
| Corrix (Cu)           |                  |           |         |          |           |           |                      |             | •              |
| Corrix (Metal)        |                  |           |         |          |           |           |                      |             | •              |
| Zorrix                | •                | •         | •       | •        | •         | •         | •                    | •           | •              |
| <b>Contact Angles</b> | 90°-115°         | >160°     | 30°-70° | 30°-70°  | < 30°     | < 30°     | < 30°                | < 20°       | 50°-70°        |



# Labview Interface v2



# Example: Installation at Customer Site



Thru-the-wall Installation



# IST's Issued Patents

(12) **United States Patent**  
Chinn et al.

(10) Patent No.: **US 7,968,187 B2**  
(45) Date of Patent: **\*Jun. 28, 2011**

(54) **SURFACE COATING**

(75) Inventors: **Jeff Chinn**, Menlo Park, CA (US); **W. Robert Ashurst**, Auburn, AL (US); **Adam Anderson**, Auburn, AL (US)

(73) Assignee: **Integrated Surface Technologies**, Menlo Park, CA (US)

(\* ) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 78 days. This patent is subject to a terminal disclaimer.

(21) Appl. No.: **12/209,629**  
(22) Filed: **Sep. 12, 2008**

(65) **Prior Publication Data**  
US 2009/0107713 A1 Apr. 30, 2009

**Related U.S. Application Data**

(60) Provisional application No. 60/983,504, filed on Oct. 29, 2007, provisional application No. 61/029,801, filed on Feb. 19, 2008.

(51) **Int. Cl.** (2006.01)  
**B12B 7/12**  
**H01L 23/29** (2006.01)

(52) **U.S. Cl.** 428/339; 428/142; 428/143; 428/145; 428/148; 428/352; 257/789; 257/791; 257/635; 257/642

(58) **Field of Classification Search** 174/255, 174/258; 428/142, 143, 145, 148, 339, 352; 257/635, 642, 789, 791  
See application file for complete search history.

(56) **References Cited**

**U.S. PATENT DOCUMENTS**

5,656,830 A \* 8/1997 Zechman ..... 237/784  
6,138,349 A \* 10/2000 Vinciguelli et al. .... 22/861  
6,902,947 B3 \* 6/2005 Chinn et al. .... 438/48  
7,339,439 B2 \* 2/2008 Sachdev et al. .... 427/517  
7,638,167 B2 \* 12/2009 Kohala et al. .... 427/248.1  
\* cited by examiner

**Primary Examiner** — Cathy Lam  
(74) **Attorney, Agent, or Firm** — Daniel L. Flamm

(57) **ABSTRACT**  
A composite is provided, comprising a substrate and a film on the substrate. The film has an RMS surface roughness of 25 nm to 500 nm, a film coverage of 25% to 60%, a surface energy of less than 70 dyne/cm; and a durability of 10 to 5000 microNewtons. Depending on the particular environment in which the film is to be used, a durability of 10 to 500 microNewtons may be preferred. A film thickness 3 to 100 times the RMS surface roughness of the film is preferred.

**22 Claims, 17 Drawing Sheets**

**Durability Improvement**

- Nano-particles adhesion improved with silane linker make a more durable film.
- Subsequent — Low Surface Energy coating (ie. FOTS) applied to nano-particle surface to create super-hydrophobic properties.

Linker Silane

Low Surface Energy FOTS functional SAM

Nano Particle

(12) **United States Patent**  
Chinn et al.

(10) Patent No.: **US 8,071,160 B2**  
(45) Date of Patent: **\*Dec. 6, 2011**

(54) **SURFACE COATING PROCESS**

(75) Inventors: **Jeffrey D. Chinn**, Foster City, CA (US); **Robert W. Ashurst**, Auburn, AL (US); **Adam N. Anderson**, Moseley, VA (US)

(73) Assignee: **Integrated Surface Technologies**, Menlo Park, CA (US)

(\* ) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 749 days. This patent is subject to a terminal disclaimer.

(21) Appl. No.: **12/115,874**  
(22) Filed: **May 6, 2008**

(65) **Prior Publication Data**  
US 2009/0110819 A1 Apr. 30, 2009

**Related U.S. Application Data**

(60) Provisional application No. 61/029,801, filed on Feb. 19, 2008, provisional application No. 60/983,504, filed on Oct. 29, 2007.

(51) **Int. Cl.** (2006.01)  
**B05J 5/12** (2006.01)  
**H05K 3/00** (2006.01)  
**C23C 16/00** (2006.01)

(52) **U.S. Cl.** 427/96.6; 427/96.2; 427/97.5; 427/96.8; 427/126.4; 427/126.3; 427/204; 427/205; 427/220; 427/255.29; 427/255.31; 427/255.37; 427/419.2; 439/43

(58) **Field of Classification Search** 427/96.6; 427/407.1; 189, 96.2, 96.5, 126.1, 126.2, 427/126.4; 349/149; 977/720, 722, 779, 977/782

See application file for complete search history.

(56) **References Cited**

**U.S. PATENT DOCUMENTS**

6,913,827 B2 \* 7/2005 George et al. .... 438/402  
7,258,895 B2 \* 8/2007 Samlha ..... 427/255.28  
2002/0025446 A1 \* 2/2002 Chen et al. .... 428/543  
2007/0141114 A1 \* 6/2007 Munsener et al. .... 42/4427  
2007/0225411 A1 \* 11/2007 McGill et al. .... 435/7.4  
2006/0304008 A1 \* 12/2008 Mousaeer et al. .... 351/159

**OTHER PUBLICATIONS**

Bourlino et al., "Clay-Organosiloxane Hybrids: A Route to Cross-Linked Clay Particles and Clay Monoliths", Chem. Mater. 2004, vol. 16, pp. 2404-2410.  
Ashart et al., "Vapor Phase Anti-Stiction Coatings for MEMS", IEEE Transactions on Device and Materials Reliability, vol. 3, No. 4, Dec. 2003, pp. 173-179.  
\* cited by examiner

**Primary Examiner** — Dah-Wei Yuan  
**Assistant Examiner** — Jose Hernandez-Diaz  
(74) **Attorney, Agent, or Firm** — Microtechnology Law & Analysis; Daniel L. Flamm

(57) **ABSTRACT**  
A method of forming a film is provided. Nanoparticles are deposited on a surface of a substrate using a liquid deposition process. The nanoparticles are linked to each other and to the surface using linker molecules. A coating having a surface energy of less than 70 dyne/cm is deposited over the film to form a coated film. The coated film has an RMS surface roughness of 25 nm to 500 nm, a film coverage of 25% to 60%, a surface energy of less than 70 dyne/cm; and a durability of 10 to 5000 microNewtons. Depending on the particular environment in which the film is to be used, a durability of 10 to 500 microNewtons may be preferred. A film thickness 3 to 100 times the RMS surface roughness of the film is preferred.

**5 Claims, 17 Drawing Sheets**

#3 US Patent: 8,221,828

# Working with IST

- As a Supplier:
  - IST is provider of custom processing equipment for variety of surface engineering films (Super-Hydrophobic, Adhesion Promoters, Wetting solutions, Plasma.)
- As a Service Provider
  - Contract Coating Services
    - IST provides rapid turn coatings as a contract service for “You” R&D. and product lines.
- As a Partner
  - Technical Consult
    - IST has extensive expertise in materials and thin film technologies and can advise “You” on case-by-case scenarios.



# Contact Us

- Fred Helmrich ([fred@insurftech.com](mailto:fred@insurftech.com))
- Jeff Chinn, Ph.D., CTO  
Integrated Surface Technologies  
1455 Adams Drive, Suite 1125  
Menlo Park, CA 94024  
[jeff@insurftech.com](mailto:jeff@insurftech.com)  
+1-650-324-1824

