Marvell NanoLab Summer Internship 2011

UC Berkeley

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What brought me here?

A:

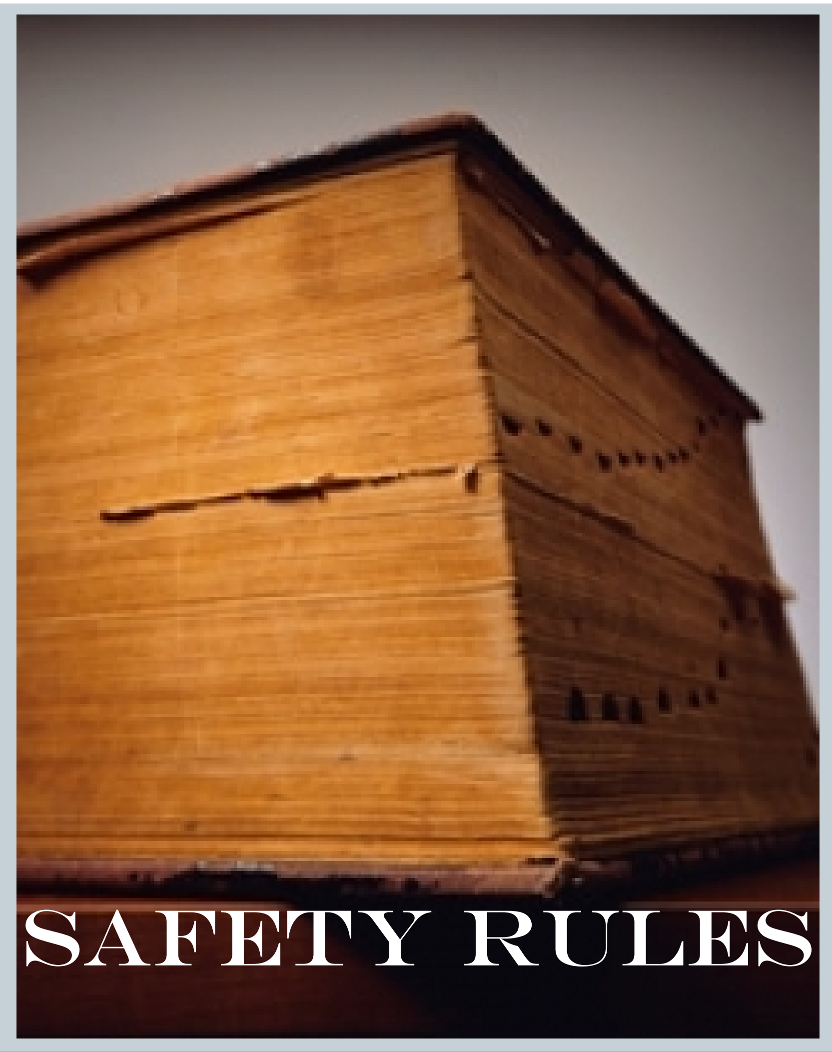
My school interests;
Math & Science
and
A father with an
Engineering degree

My next logical question:
Would I like math & science **outside** of school, and quite possibly, **engineering**?

Day

1

The Essentials



Experiment Objectives

PART A

- Modify silicon surface wetting properties
- Improve photoresist adhesion to oxides via HMDS application
- Test various HMDS application methods for best deposition

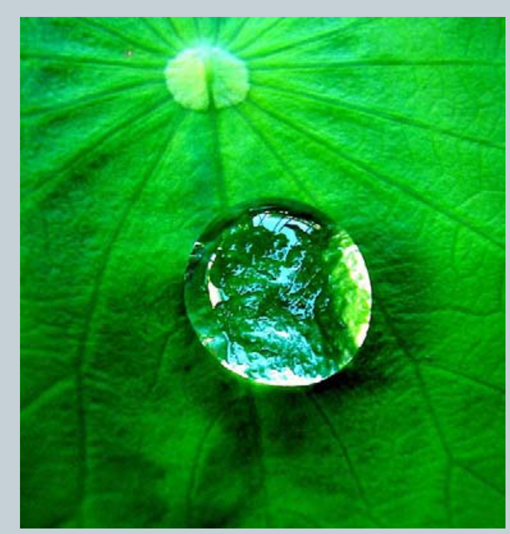
PART B

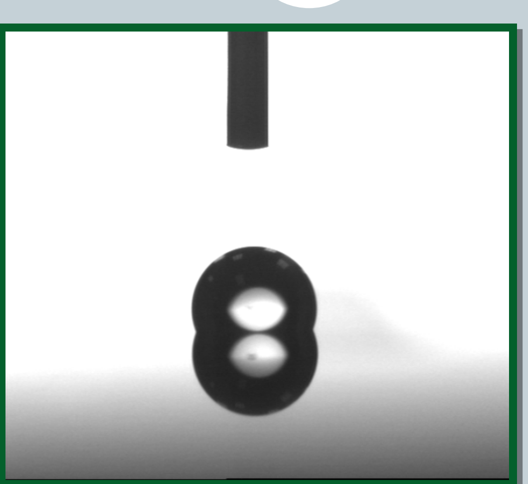
- Promote adhesion and reduce stiction on MEMS devices via mono-molecular film coatings
- Modify wetting properties on BioMEMS devices via mono-molecular film coatings

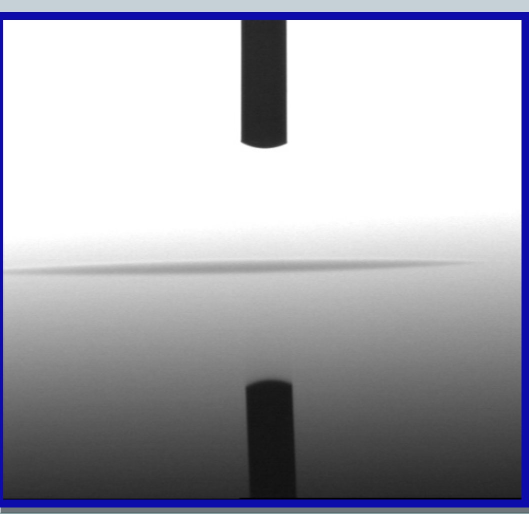
What are Wetting Properties?

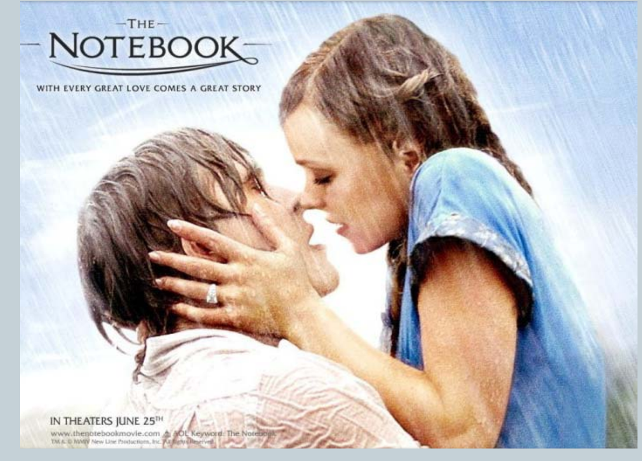
Hydrophobic

Repels water







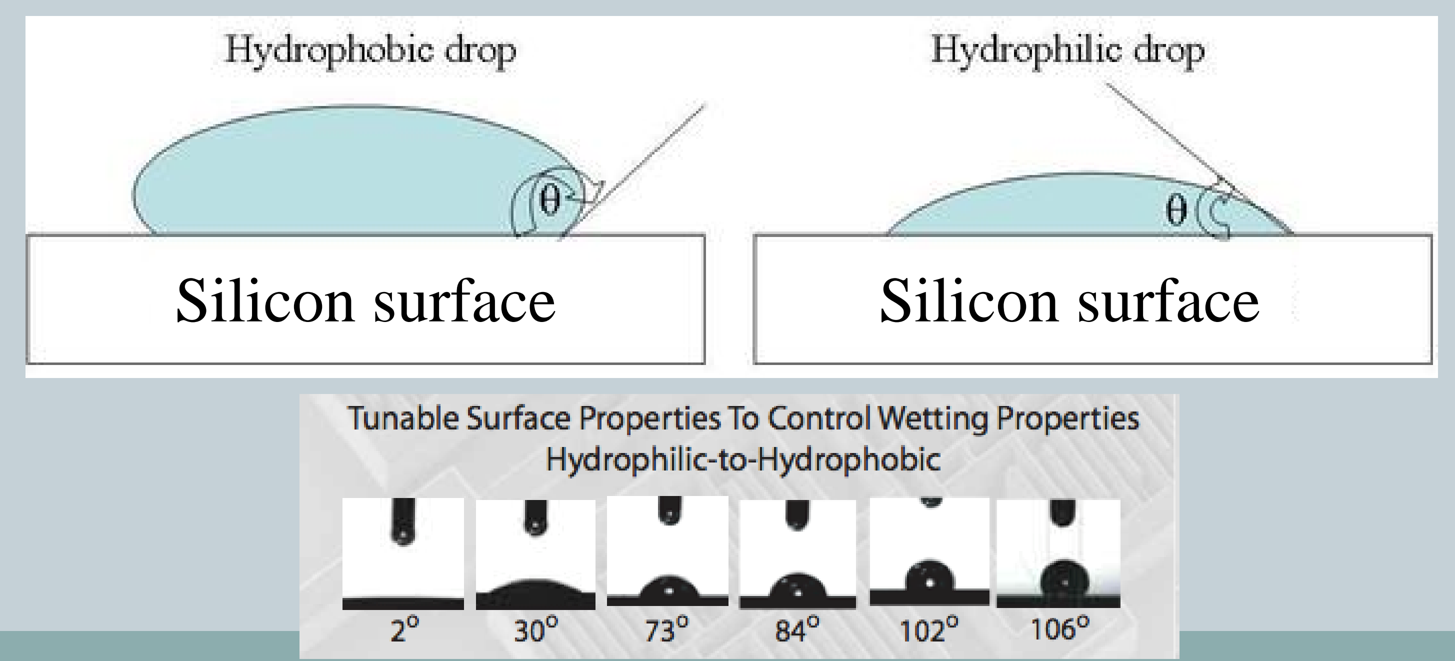


Hydrophilic

Attracted to, and tends to be dissolved by water

Kruss Contact Angle Measurement System

- Uses a sessile water drop method to estimate wetting properties on surfaces
- Measures the angle between the baseline of the drop and the drop boundary

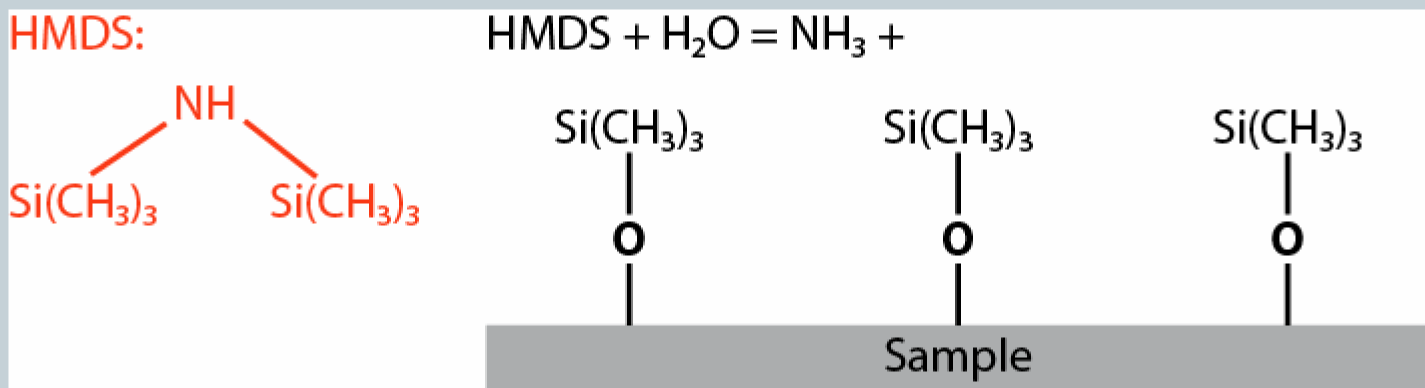


Part A

MEASURING CONTACT ANGLES

HMDS

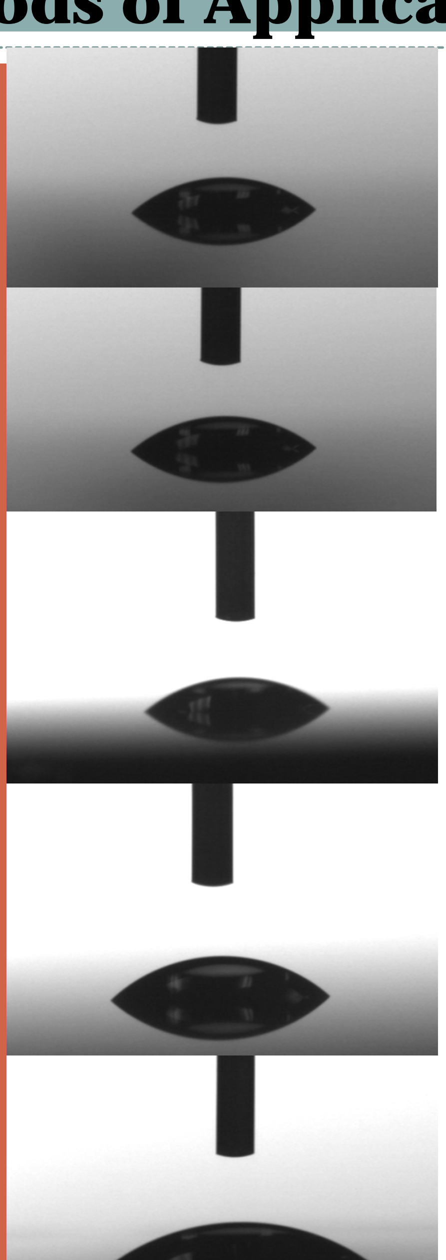
- HMDS = Hexamethyldisilazane
- Organosilicon compound
- Used to improve photoresist adhesion to oxides
- Reacts with the oxide surface forming a strong bond, but at the same time leaving free bonds to react with the photoresist



HMDS Methods of Application

#1 Msink3 Bubbler Tank

- Does not dehydrate wafers
- Manually timed cycle – 1 minute HMDS treatment
- NO heat



Immediately
42.2

1 hour later
42.1

1 day later
39.0

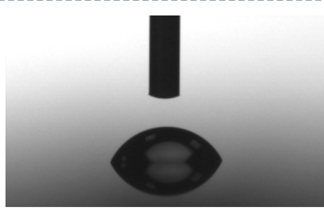
7 days later
40.1

21 days later
37.1

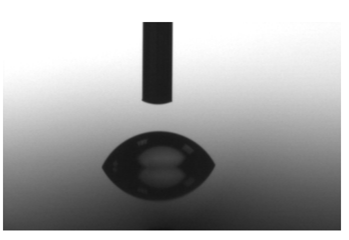
HMDS Methods of Application

#2 Svgcoat6

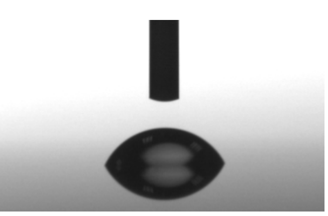
- 1 minute HMDS treatment with N2 pump and purge cycles before application
- hot plate 100°C



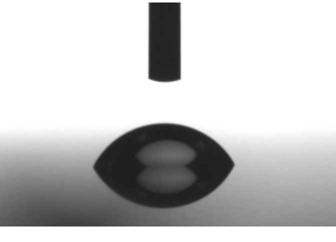
Immediately
65.2



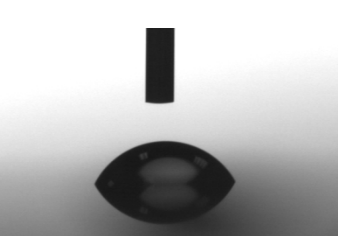
1 hour later
64.5



1 day later
63.7



7 days later
62.3

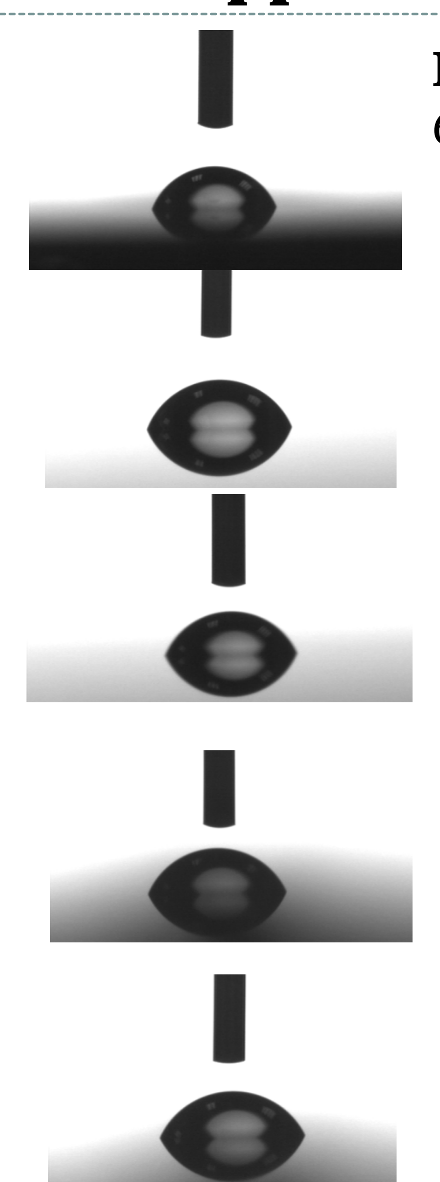


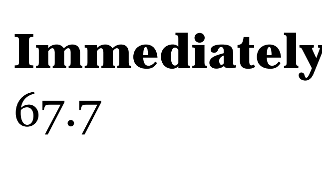
21 days later
62.7

HMDS Methods of Application

#3 Primeoven Program 0

- 1 minute HMDS treatment with three N2 pump and purge cycles before application
- 90°C





Immediately
67.7

1 hour later
67.8

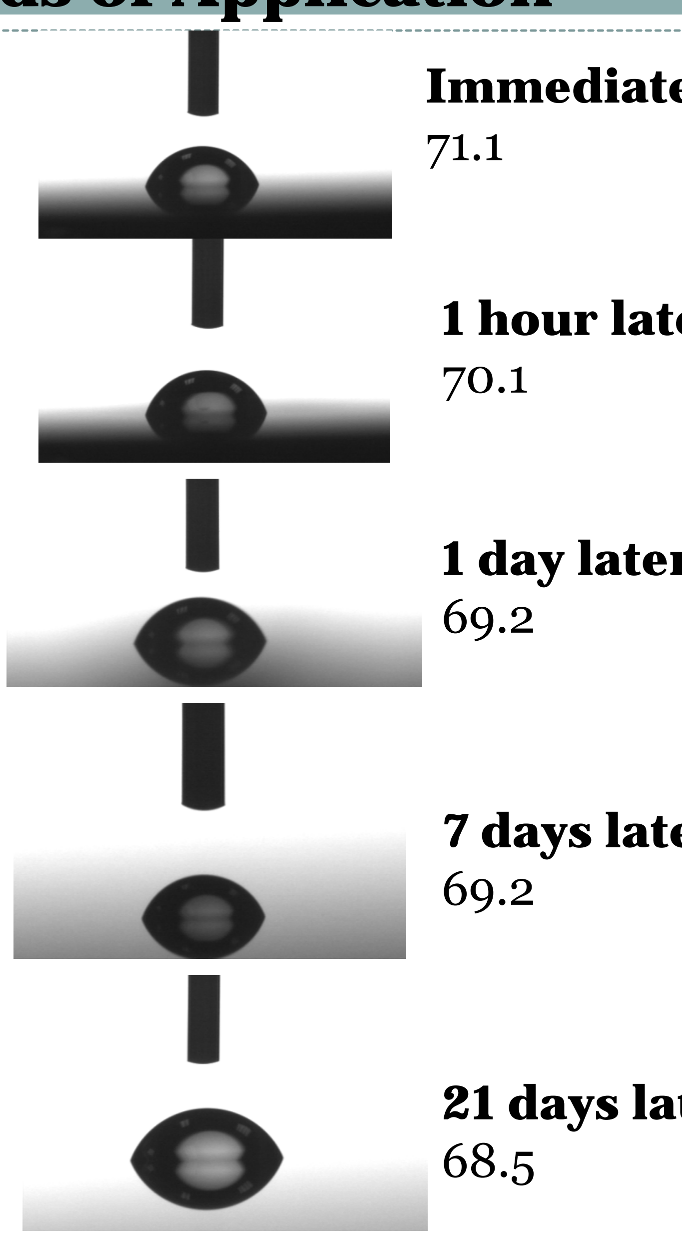
1 day later
66.6

7 days later
67.3

21 days later
64.7

#4 Primeoven Program 2

- 2 minute HMDS treatment with one long pump down before application
- 90°C



Immediately
71.1

1 hour later
70.1

1 day later
69.2

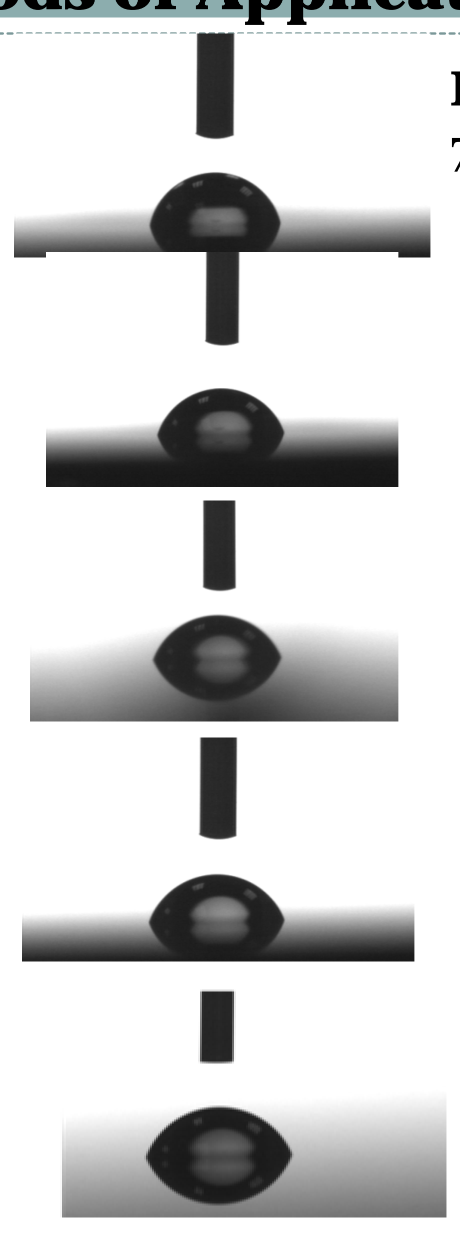
7 days later
69.2

21 days later
68.5

HMDS Methods of Application

#5 Primeoven with Oxide Wafer

- 2 minute HMDS treatment with one long pump down before application
- 1000A oxide wafers
- 90°C



Immediately
76.5

1 hour later
70.9

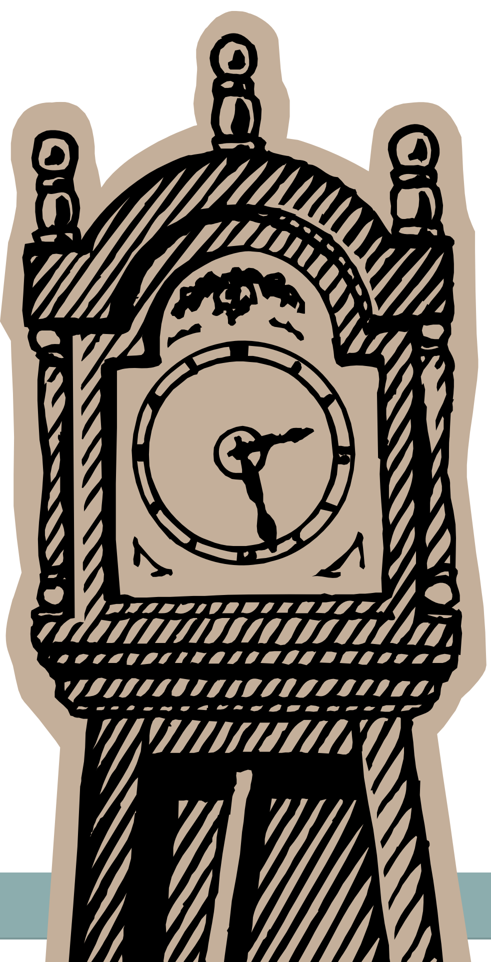
1 day later
69.2

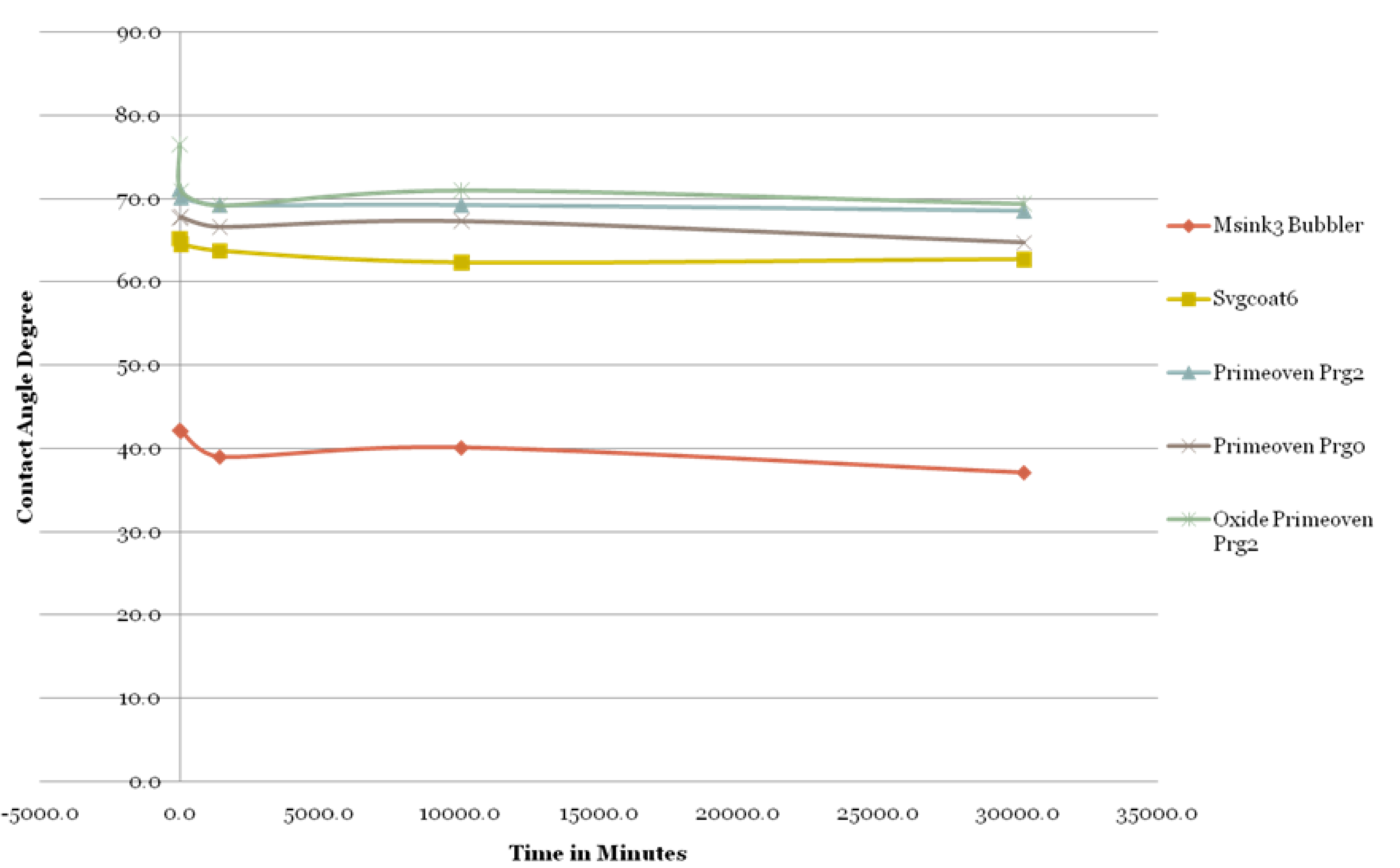
7 days later
71.0

21 days later
69.4

HMDS Application Method	**Contact Angle Measurement *Taken Immediately***	**Kruss Contact Angle Image**
Msink3 Bubbler for 1 minute	42.2	
Svgcoat6 for 1 minute	65.2	
Primeoven Program 0 for 1 minute	67.7	
Primeoven Program 2 for 2 minutes	71.1	
Primeoven Program 2 *on an Oxide Wafer* for 2 minutes	76.5	

Over Time…




HMDS Contact Angle Measurement
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
Contact Angle Degree
-5000.0
0.0
5000.0
10000.0
15000.0
20000.0
25000.0
30000.0
35000.0
Time in Minutes
Msink3 Bubbler
Svgcoat6
Primeoven Prg2
Primeoven Prg0
Oxide Primeoven Prg2

HMDS Application Conclusions

- Since the Svgcoat6 and Primeoven methods were baked, pumped and purged, the contact angles didn't degrade in 3 weeks because HMDS bonded with the oxide surface and sealed out moisture.
- Therefore, after HMDS treatment in the Svgcoat6 and Primeoven, it may still be possible to coat the photoresist on the wafers after 3 weeks.
- The Msink3 method did not bake the wafers nor did it pump out the extra water vapor. Therefore, the contact angle changed over time as the wafers absorbed some moisture.

Part B

MEASURING CONTACT ANGLES

AMST Machine

- AMST is a molecular vapor deposition system that can deposit single layer mono-molecular films. Monolayer deposition is used to change the surface properties of MEMS and BioMEMS structures.
- Room temperature vapor deposition

Sample Applications:

- Deposition of FOTS: increases surface hydrophobicity desired by MEMS devices
- Deposition of organosilane monolayers to enable covalent attachment of specific receptor molecules to BioMEMS sensor surfaces

AMST Available Gases

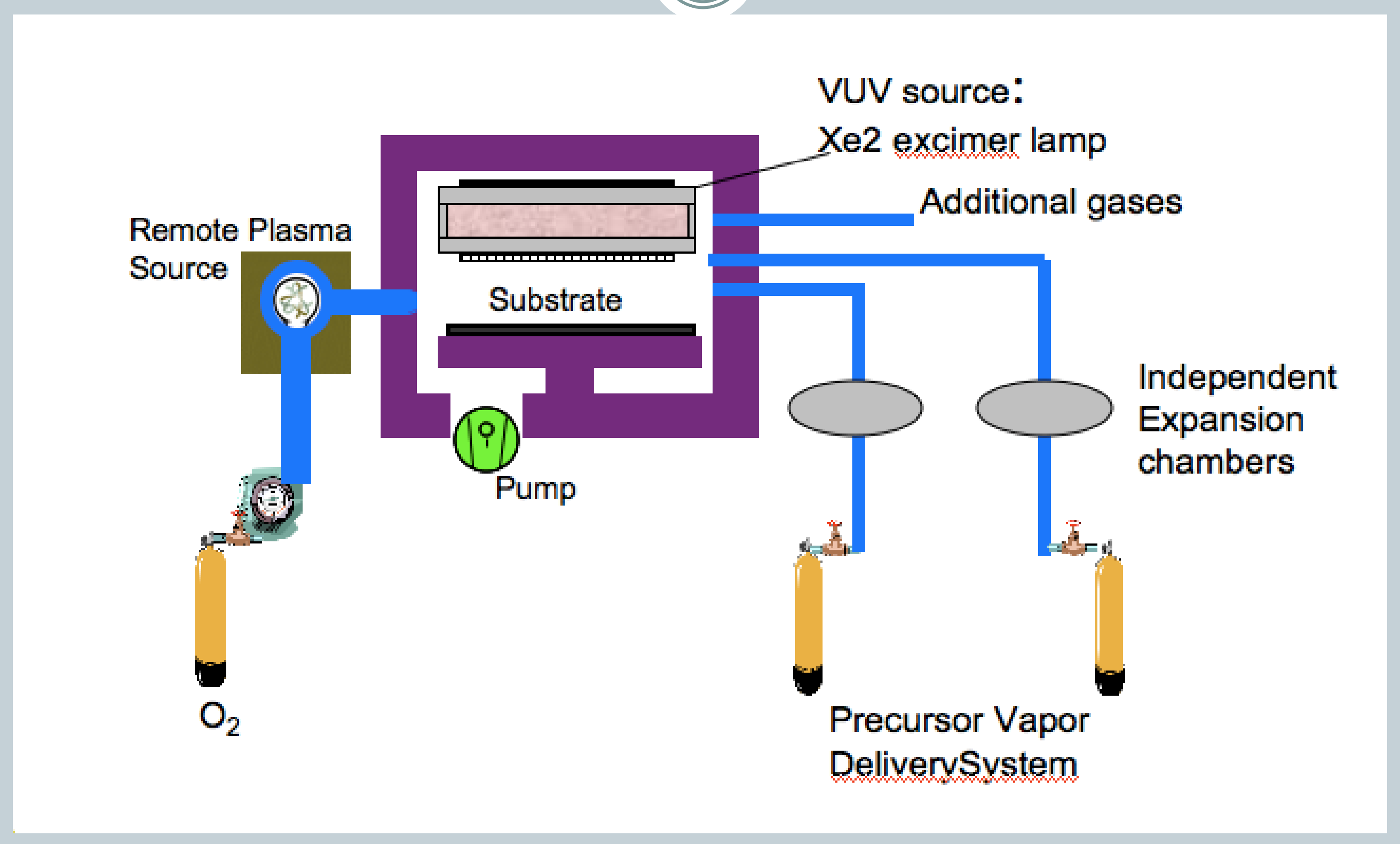
- **Line 1** - deionized water vapor

  A small amount of water vapor is required for reaction of most chlorinated or oxy silanes to react with silica surfaces
- **Line 2** - labmember proposed chemistries, our experiment used

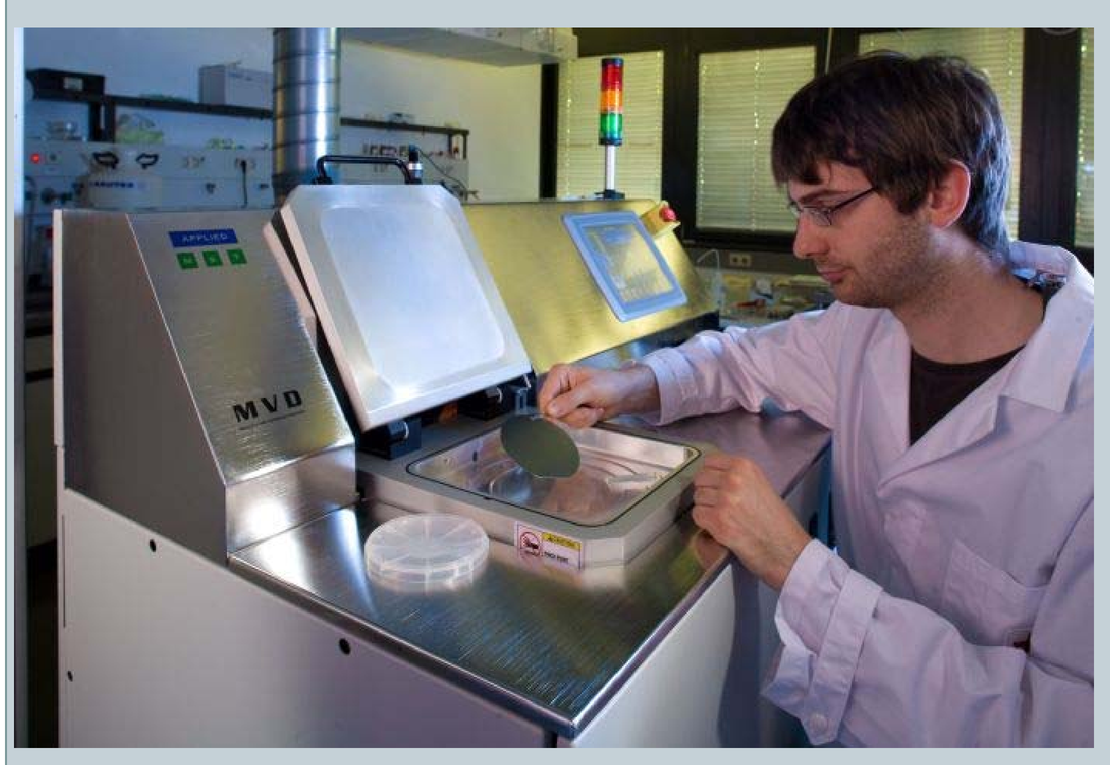
  Gamma-methacryloxypropyltrimethoxysilane (Gamma-MPS)
- **Line 3** – Fluoro Octo Trichloro Silane (FOTS)
- **Oxygen**

  Used for substrate surface condition and chamber cleaning

AMST Configuration



Steps for Using the AMST



I. Vent chamber
II. Open & Load wafer
III. Run Process
IV. Starts with Purge, followed by RF treatment, Vapor Injection, Process Reaction & final Purge cycles

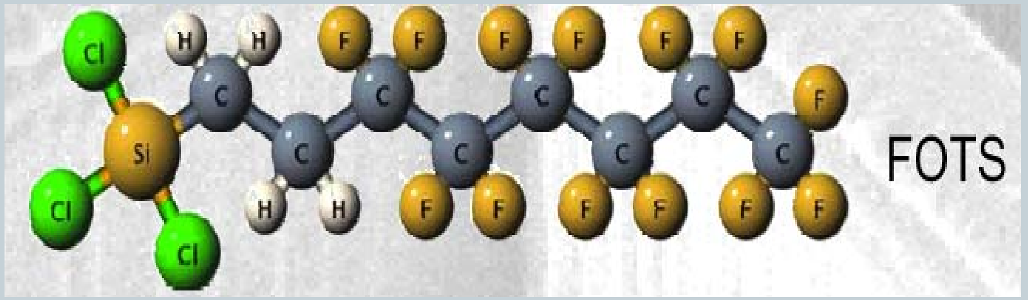
   ≈25 minutes
V. Vent chamber
VI. Open & Remove wafer

Plasma Clean vs. Oxygen Clean Line

RF Plasma Clean	Clean Line
• Oxygen plasma to clean and pre-treat the substrate surface • Plasma consists of a collection of free moving electrons and ions • Removes impurities and contaminants from surfaces through the use of an energetic plasma	• Pump out lines by opening valves • Turn on Ozone (O_3) generator • Flushes line and chamber to clean and prevent cross-contamination and ensure good film for deposition

MVD Coating with FOTS

- FOTS = fluoro octa trichloro silane
- FOTS treatment on SiO2 renders surface hydrophobic
- Water contact angle of 110°
- Proven anti-stiction coating for released MEMS



RECIPE

Pretreatement		Coating conditions	
RF Power	200 W	Partial pressure of FOTS	4 T
Oxygen	100 sccm	Partial pressure of water	0.7 T
Cleaning time	5 min	Reaction time	15 min

MVD Coating with Gamma-MPS

- Gamma-MPS = gamma-methacryloxypropyltrimethoxysilane
- Gamma-MPS treatment on SiO2 improves hydrophobicity
- Water contact angle of 60°
- Used in coatings, adhesives and sealants to provide superior adhesion and durability
- Adhesion promoter for parylene

Gamma-MPS

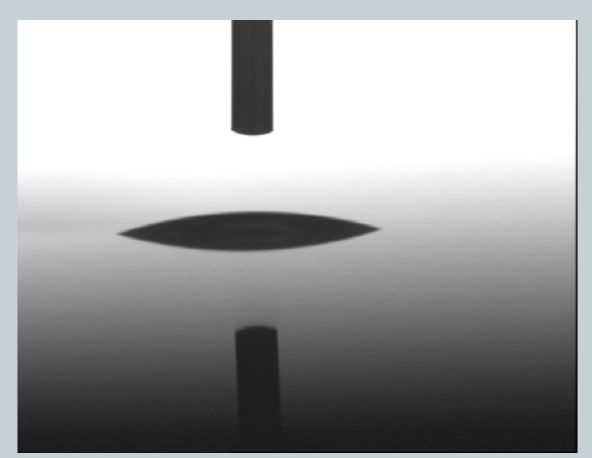
RECIPE

Pretreatement	
RF Power	200 W
Oxygen	80 sccm
Cleaning time	5 min

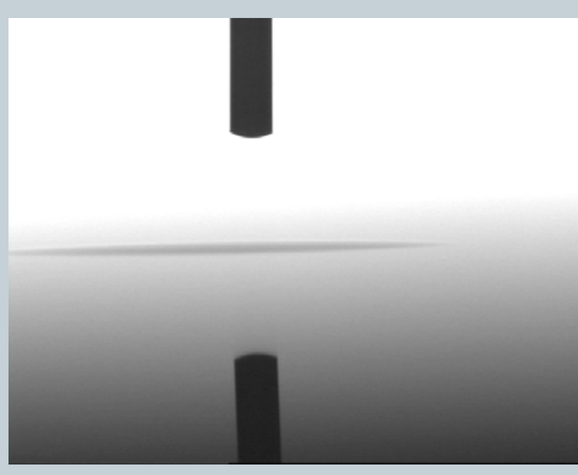
Coating conditions	
Partial pressure of Gamma	0.5 T
Partial pressure of water	4 T
Reaction time	15 min

FOTS Contact Angle

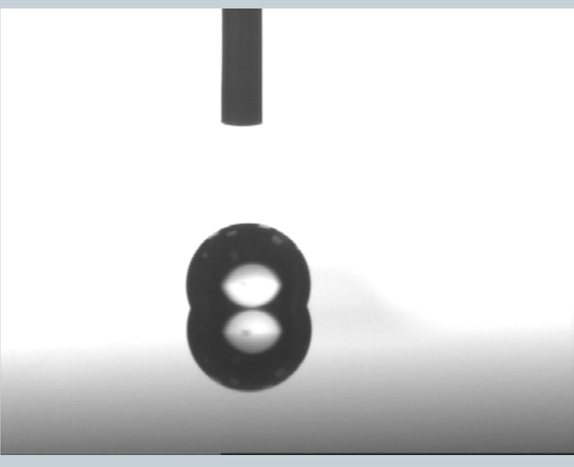
- Bare silicon wafers from the vendors have thin layers of native oxide
- After O2 plasma clean a thin layer of oxide of forms



Silicon wafer out of box
20.1°



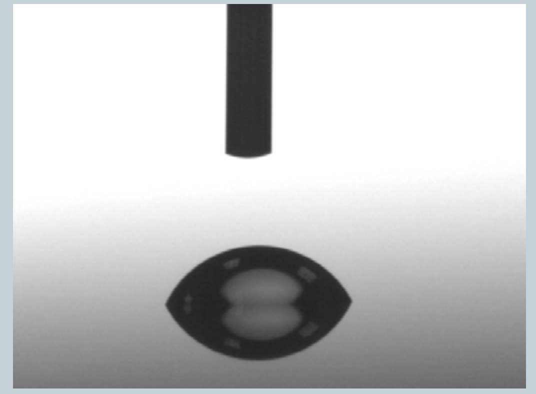
After AMST Plasma clean
0.0°



After Plasma clean & FOTS
110.2°

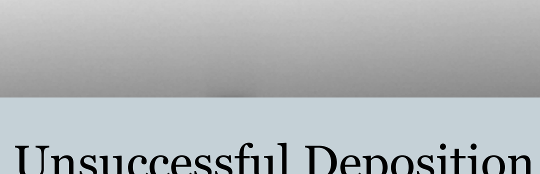
Unsuccessful Gamma-MPS Deposition

1. Performed 1 hour ozone clean on AMST
2. Purged Line 2 valve 10 times to flush out gas



Successful Deposition

1 process later…



Unsuccessful Deposition

Line 2 pressure not constant

Leak Rate of 16.1 mTorr/min

Acknowledgements

- Thank you to everyone at the Nanolab for your help and guidance, and for making this a great summer.
- Thanks to Jimmy Chang, Anna Szucs, Sia Parsa, Rosemary Spivey, and the equipment engineers.
- Special thanks to Marilyn Kushner for taking me to Semicon, for the opportunity and the experience.
- Thank you Katalin Voros for this amazing summer opportunity.
- Special thanks to Kim Chan, an incredible mentor for whom I am so grateful!