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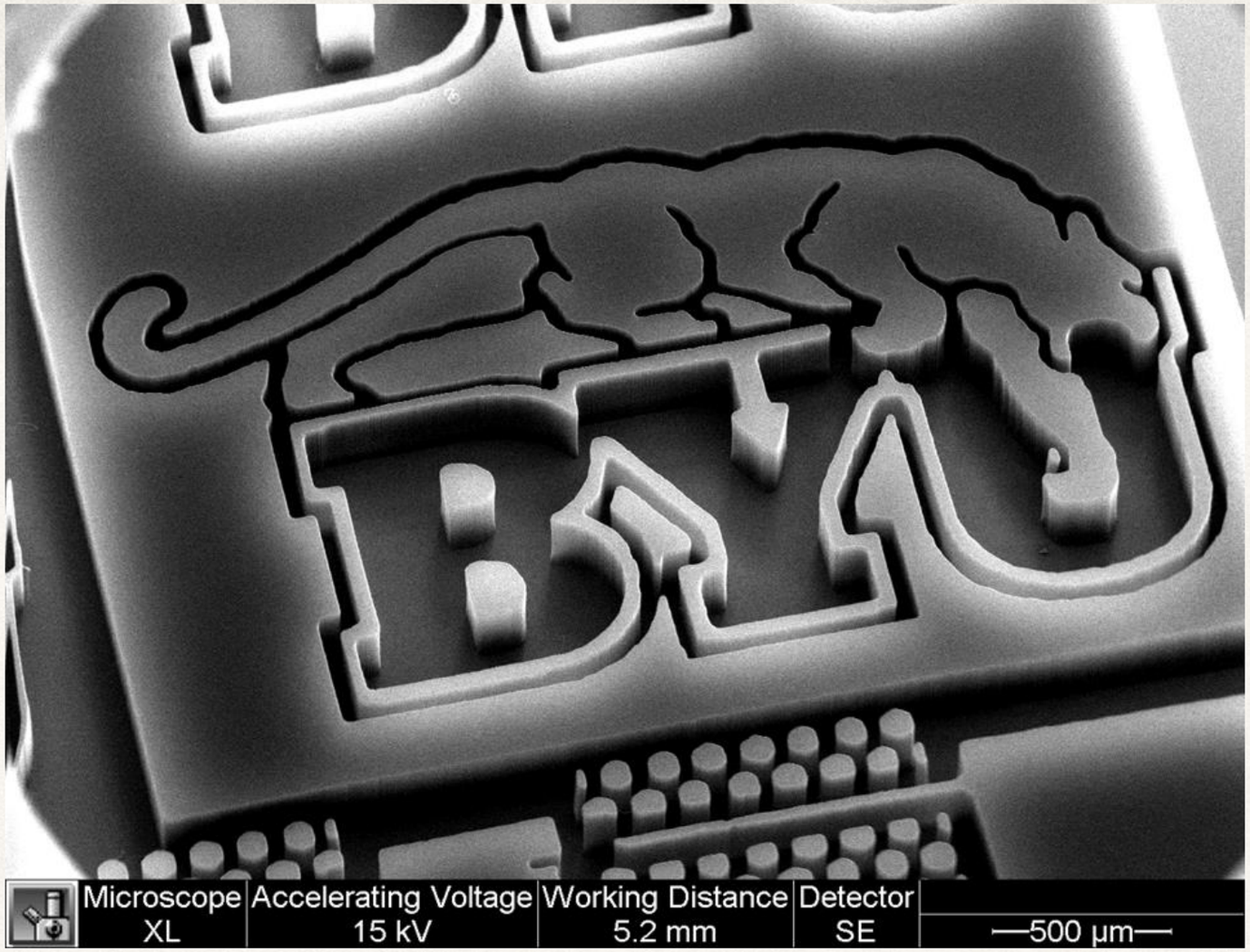

Infiltration of CNT-M Microstructures using CVD and ALD

Presented by: Collin Brown, Jason Kyle Anderson

October 31st, 2013

Acknowledgments

Jason Kyle Anderson for his help in getting the system to work

Dr's David Allred, Richard Vanfleet, and Robert Davis for their help and direction in this project

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Richard Hansen for his work on CNT-M

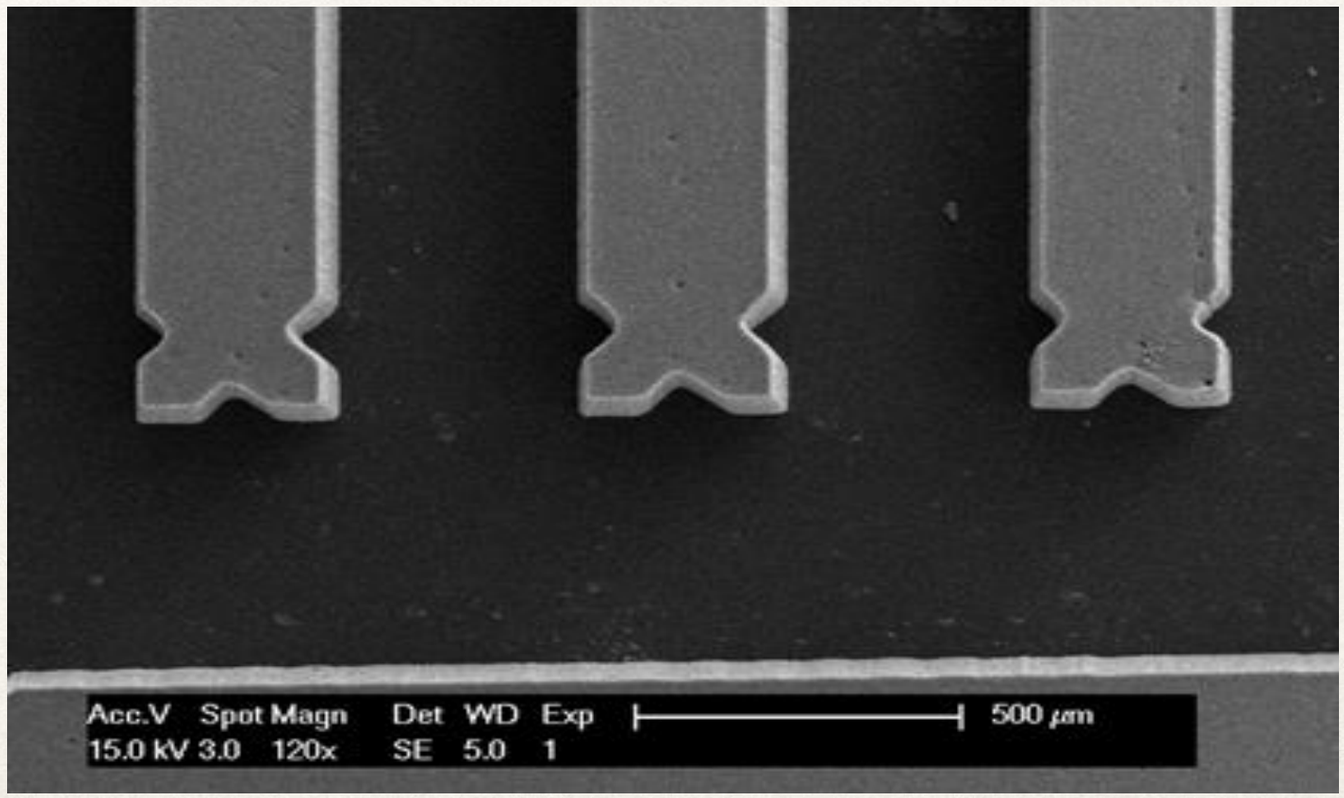
David McKenna for constructing the CVD system

Outline

- Purpose of Deposition Techniques
- Overview of CNT-M
- Theory of Deposition
  - Chemical Vapor Deposition (CVD)
  - Atomic Layer Deposition (ALD)
- Conversion of CVD system to ALD
- Initial Results
  - Initial Characterization with SEM, TEM, and XEDS

Purpose

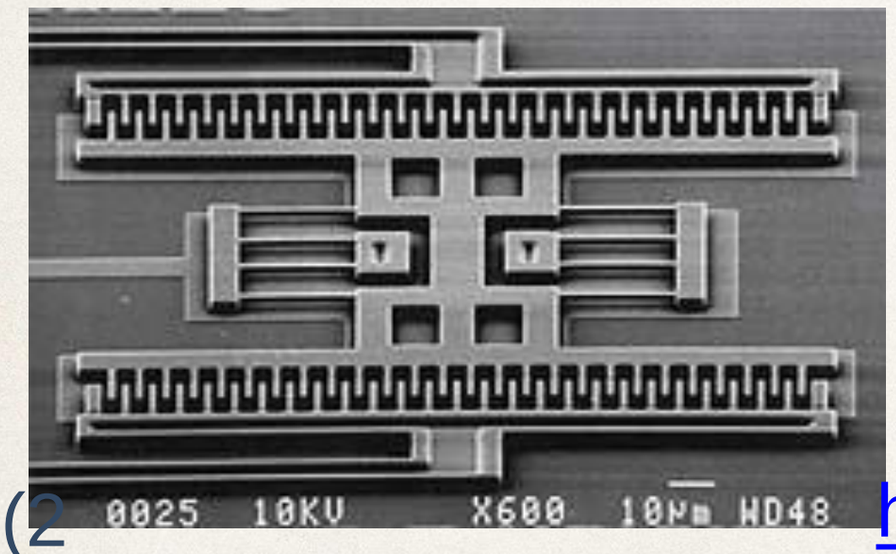
- Create mechanical and electrical components on a micro scale - NEMS/MEMS



Why does this matter?

- High sensitivity inertial sensors
- X-ray collimator

(1)

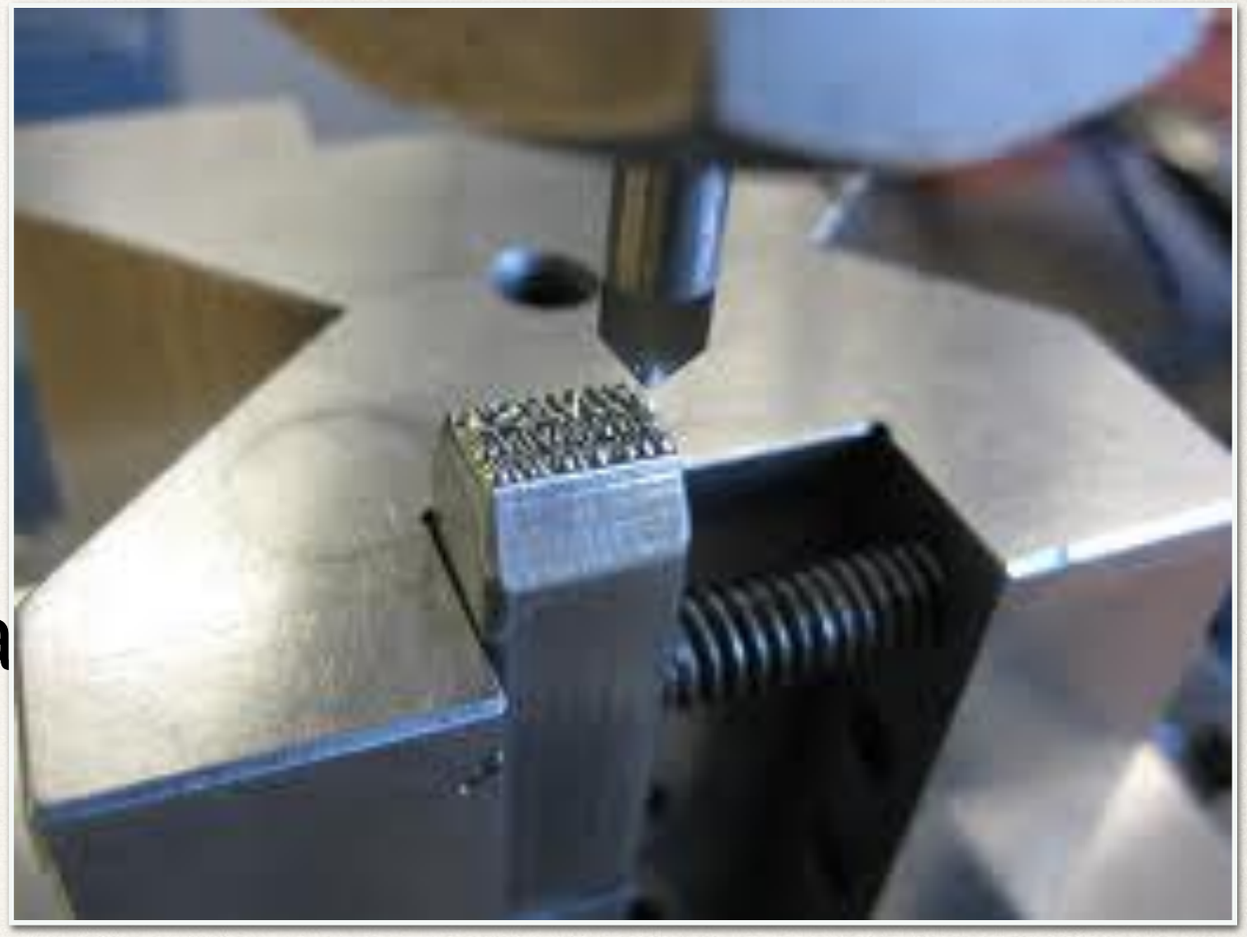


(2) http://www.ducksters.com/games/wii-sports-bowling.php

Traditional Fabrication Method

Etching-

Chemically or mechanically removing selective parts of a solid metal



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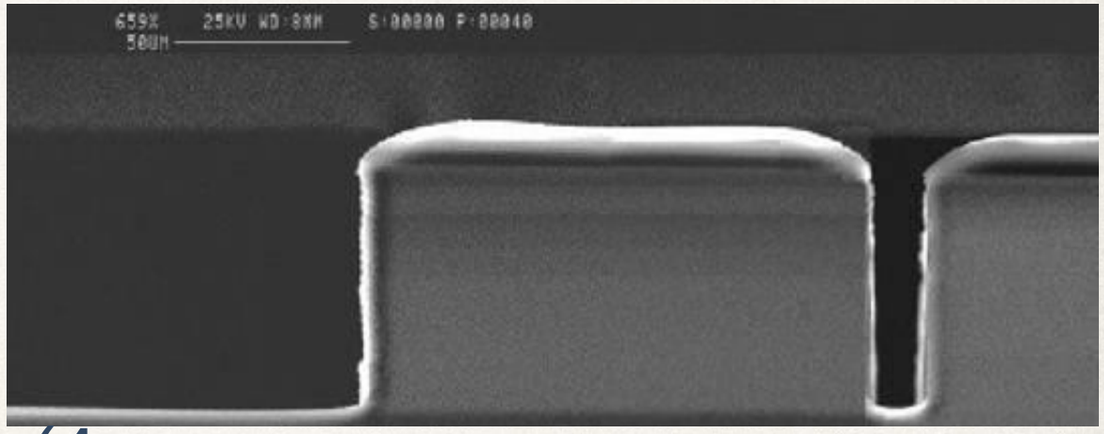
Limitations of Chemical Etching

- Limited selection of materials - Tungsten, Gold, Silicon, Aluminum
- Poor aspect ratio (height to width)
- (~5:1) aspect ratio due to gas transportation limits



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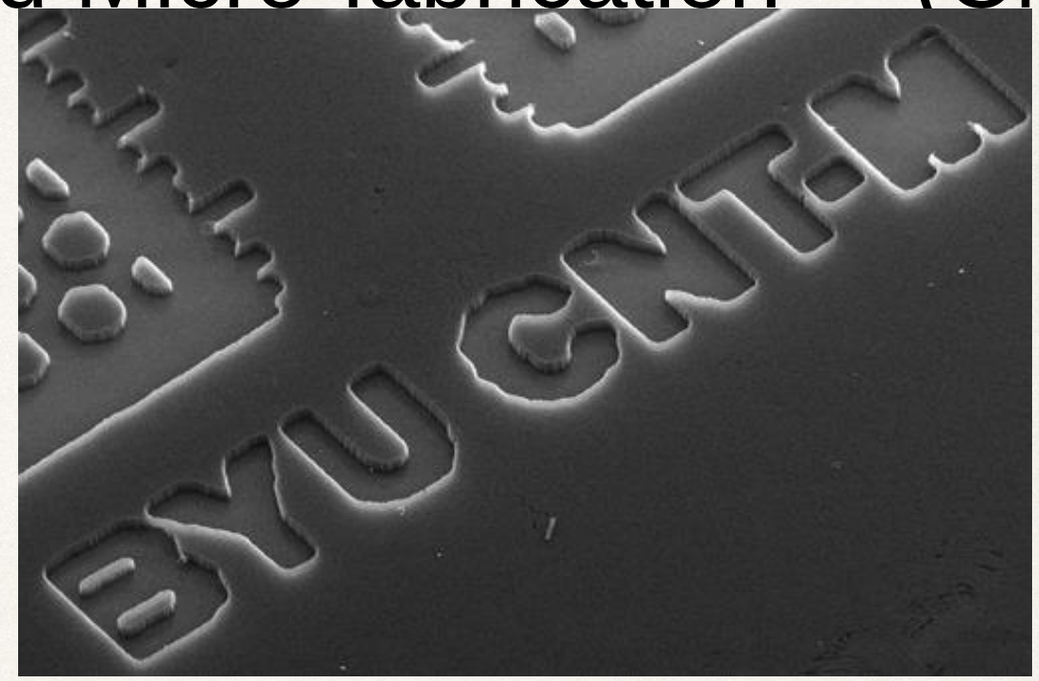
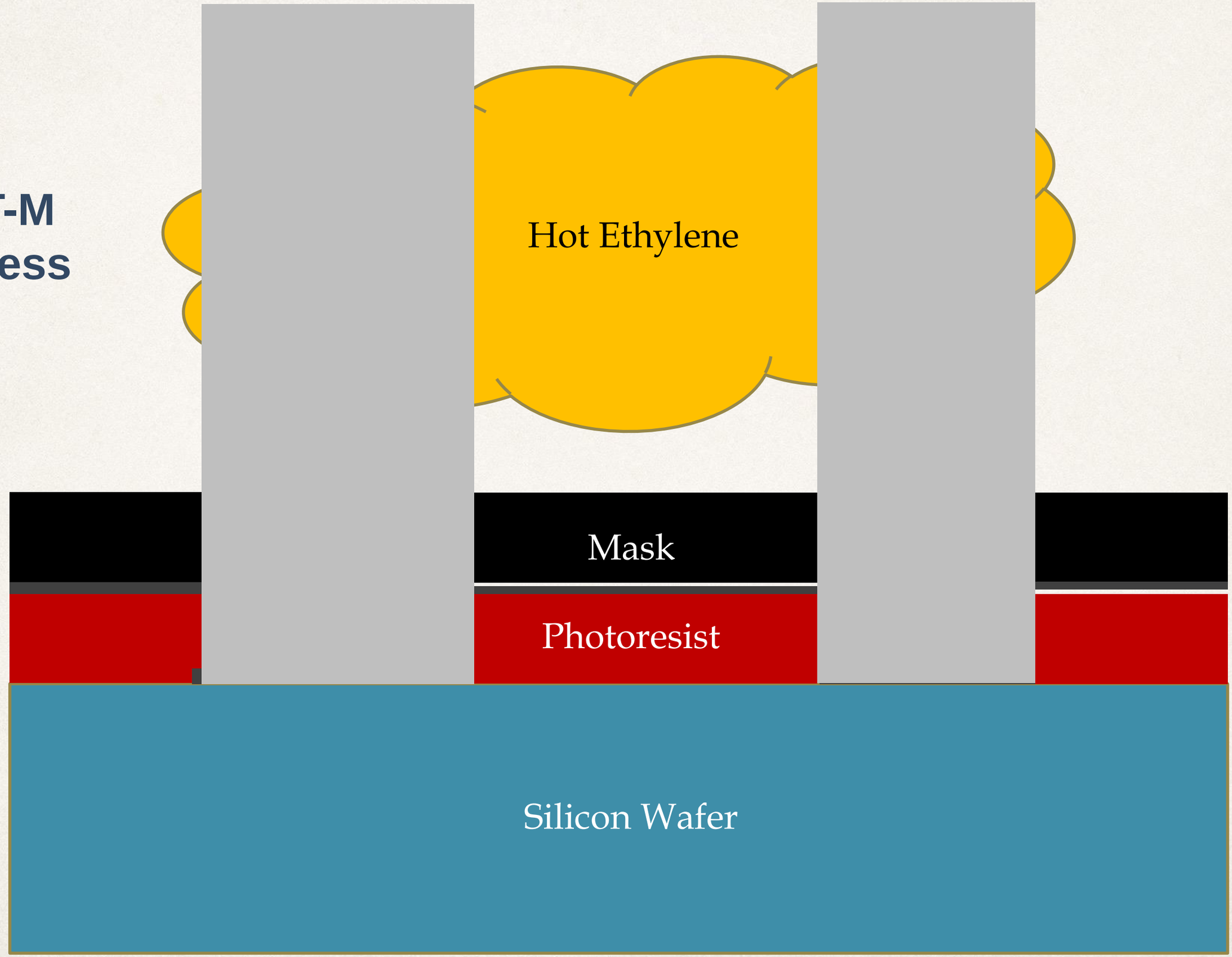
New Method for NEMS/MEMS

- Carbon Nanotube Templated Micro-fabrication (CNT-M)



- Easily Controlled
- High Aspect Ratio (200:1)
- features >500 microns tall by 2-3 microns across
- Electrically conductive

CNT-M Process



Chemical Vapor Deposition (CVD)

- When used to infiltrate a CNT, called Chemical Vapor Infiltration (CVI)
- Flow reactants into chamber constantly, usually at high temperature
- Heat the solid precursor so it will volatilize
- Flow an inert gas to carry the precursor to the sample
- Problems
  - Uneven Infiltration
  - Capping

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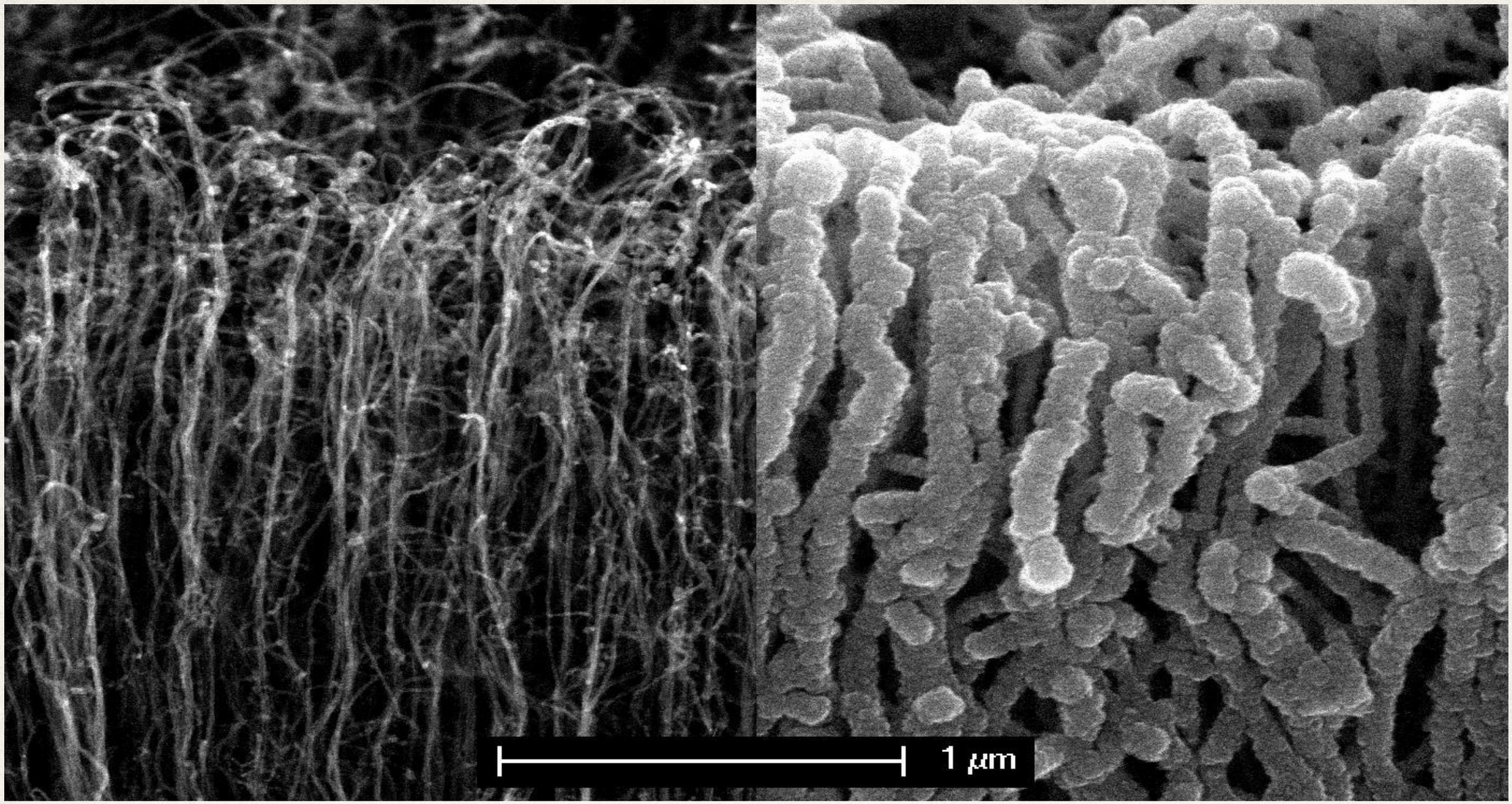

(6)

Before
infiltration

After
infiltration

Forest - 1% Carbon

Carbon Nanotube Forest Infiltrated with $Mo(CO)_6$
by Richard Hansen

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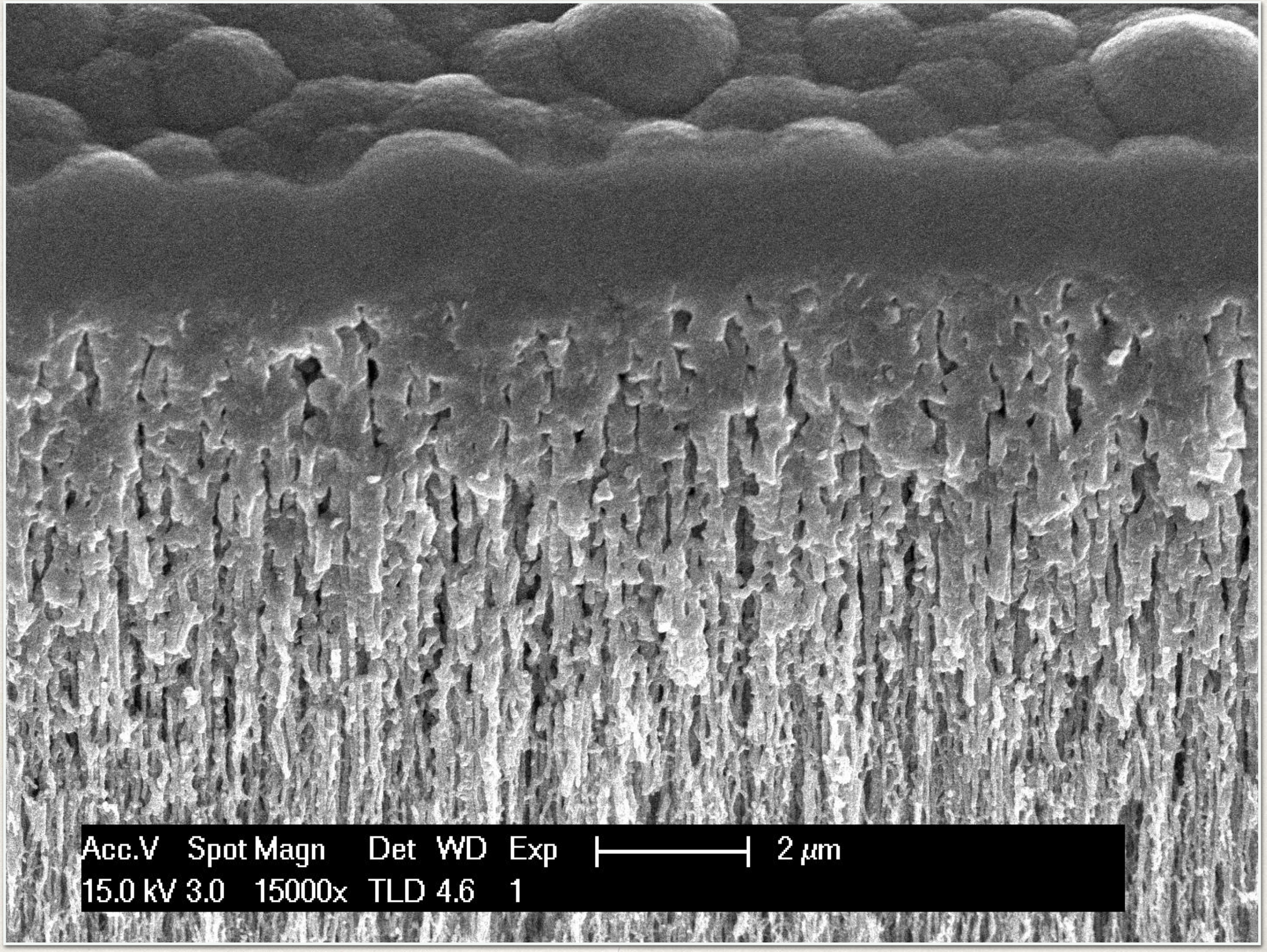
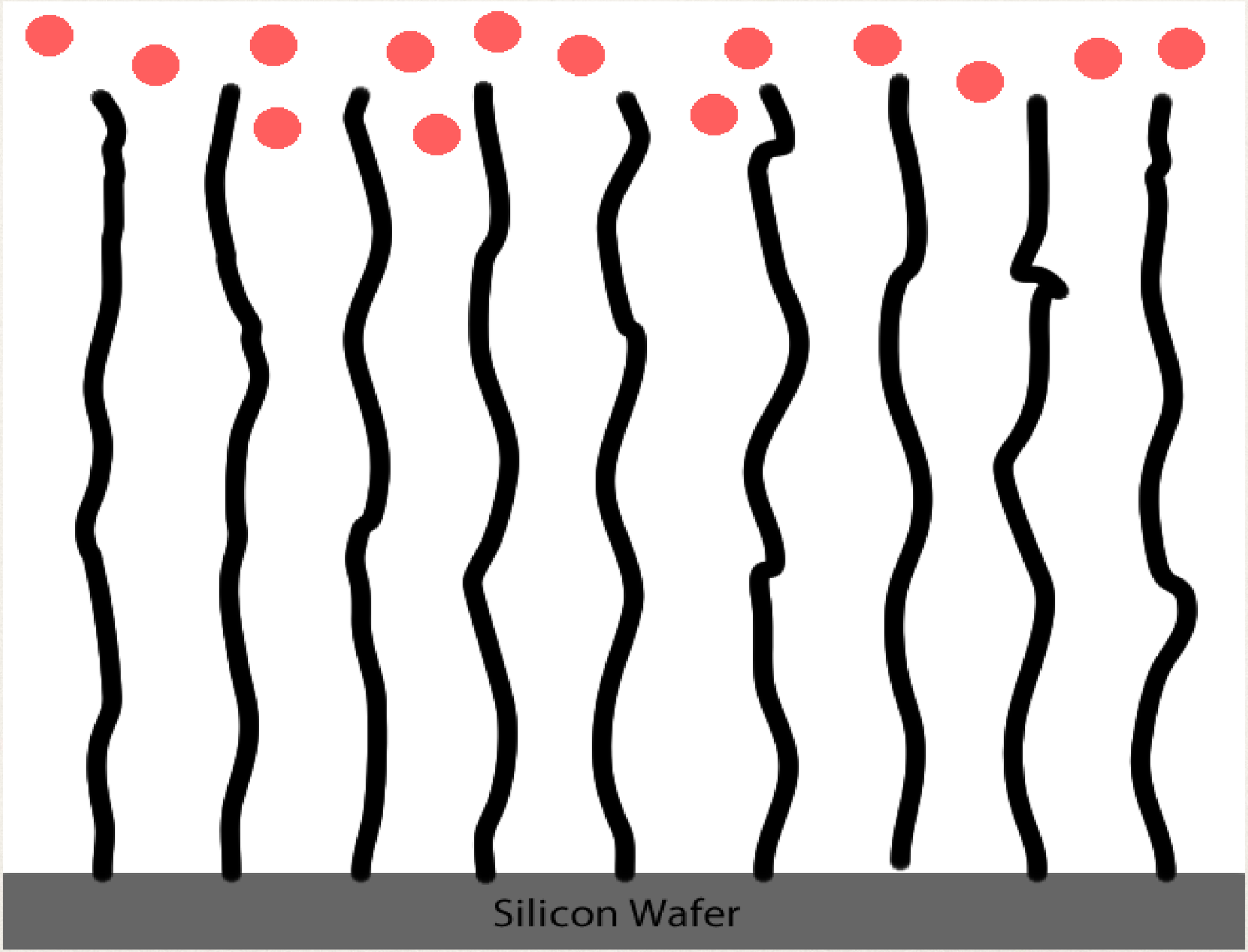
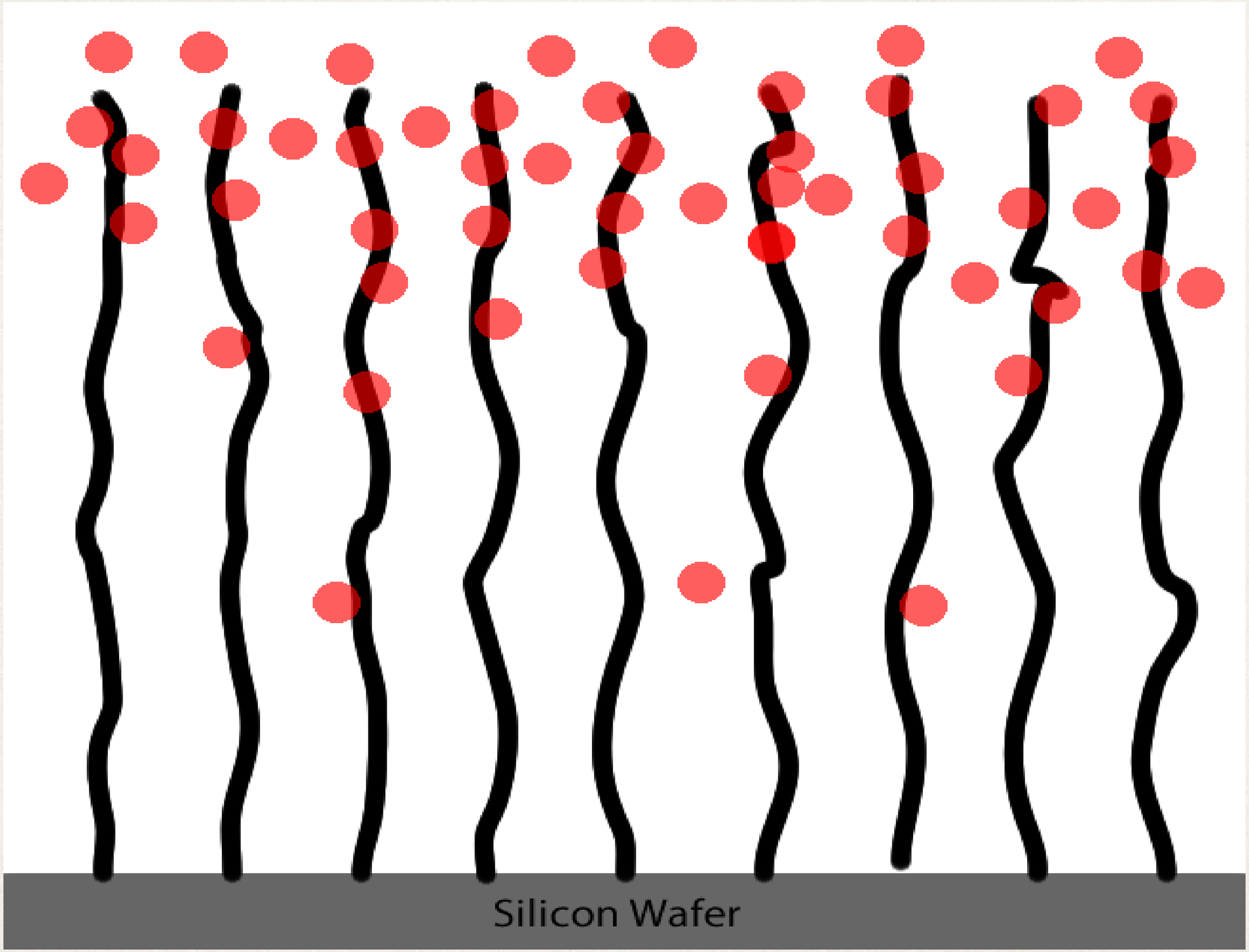



Silicon Wafer


Silicon Wafer

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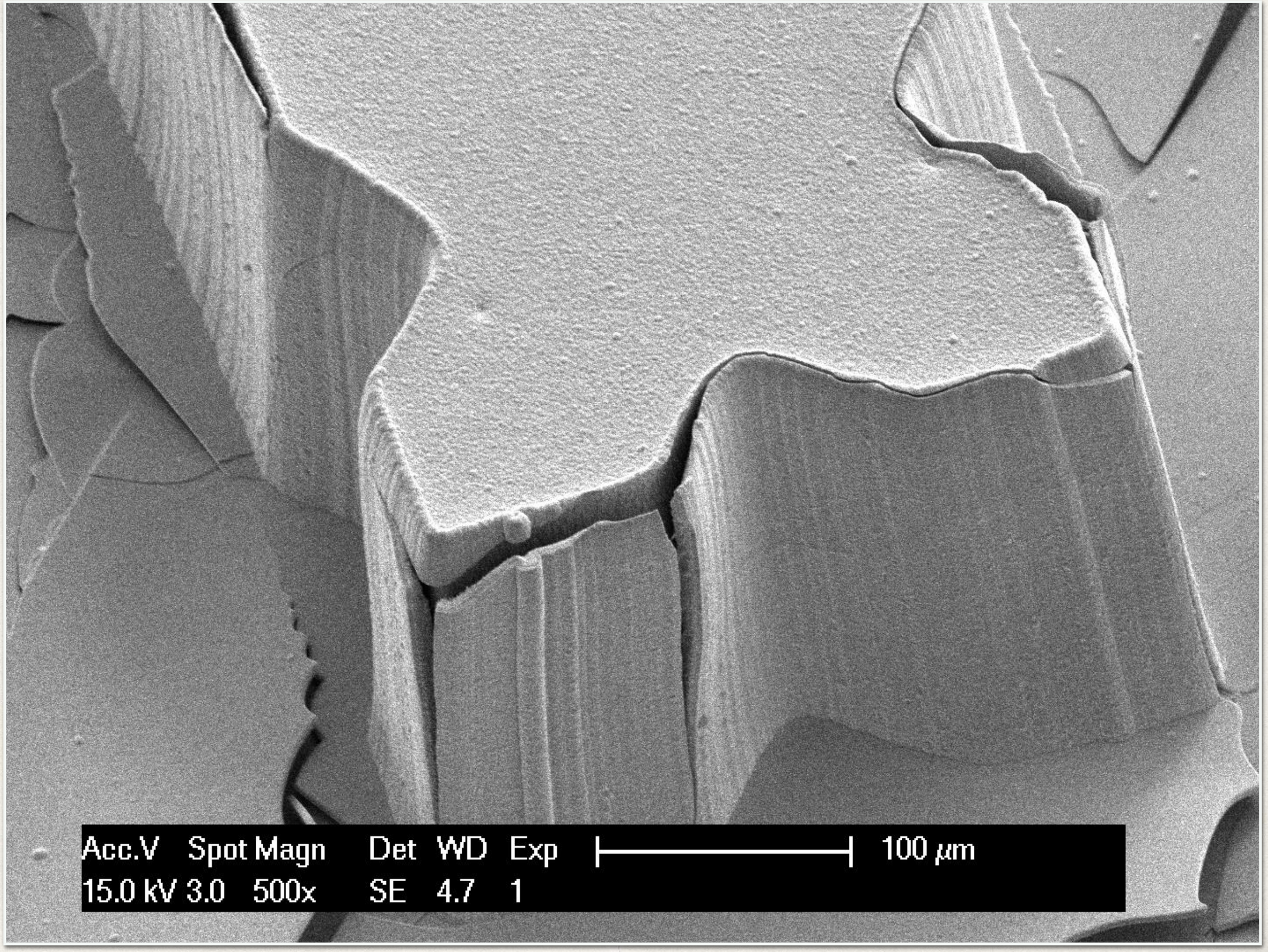
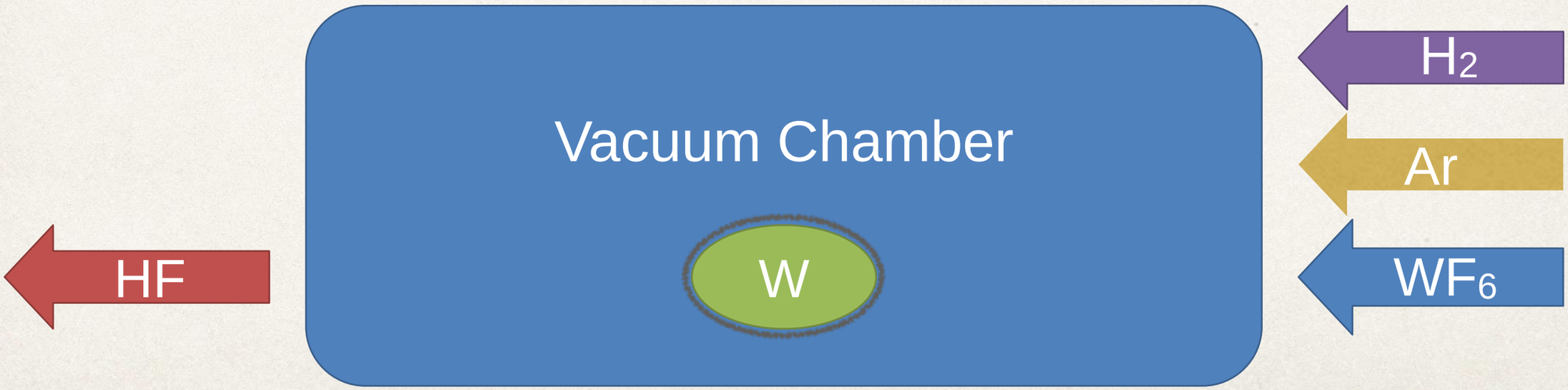


Atomic Layer Deposition (ALD)

- Propose to try with Tungsten in nanotube forests, so Atomic Layer Infiltration (AFI)
- Self-limiting layer by layer
- Hope to achieve:
  - uniform material properties
  - eliminate crusts

Tungsten ALD with WF_6

- Gaseous form of WF_6 self-limits deposition
- Flow H_2, monatomic Hydrogen from Plasma, or Silane (SiH_4)
- H reduces WF_6 leaving a layer of W
- HF gas molecules created



Converting the System

- Modified from CVD
- Hooked in gas lines for WF_6
- Seal cabinet,install cabinet exhaust system, and insert sensors and filters for HF.
- Automate Labview™ program to cycle valves for ALD

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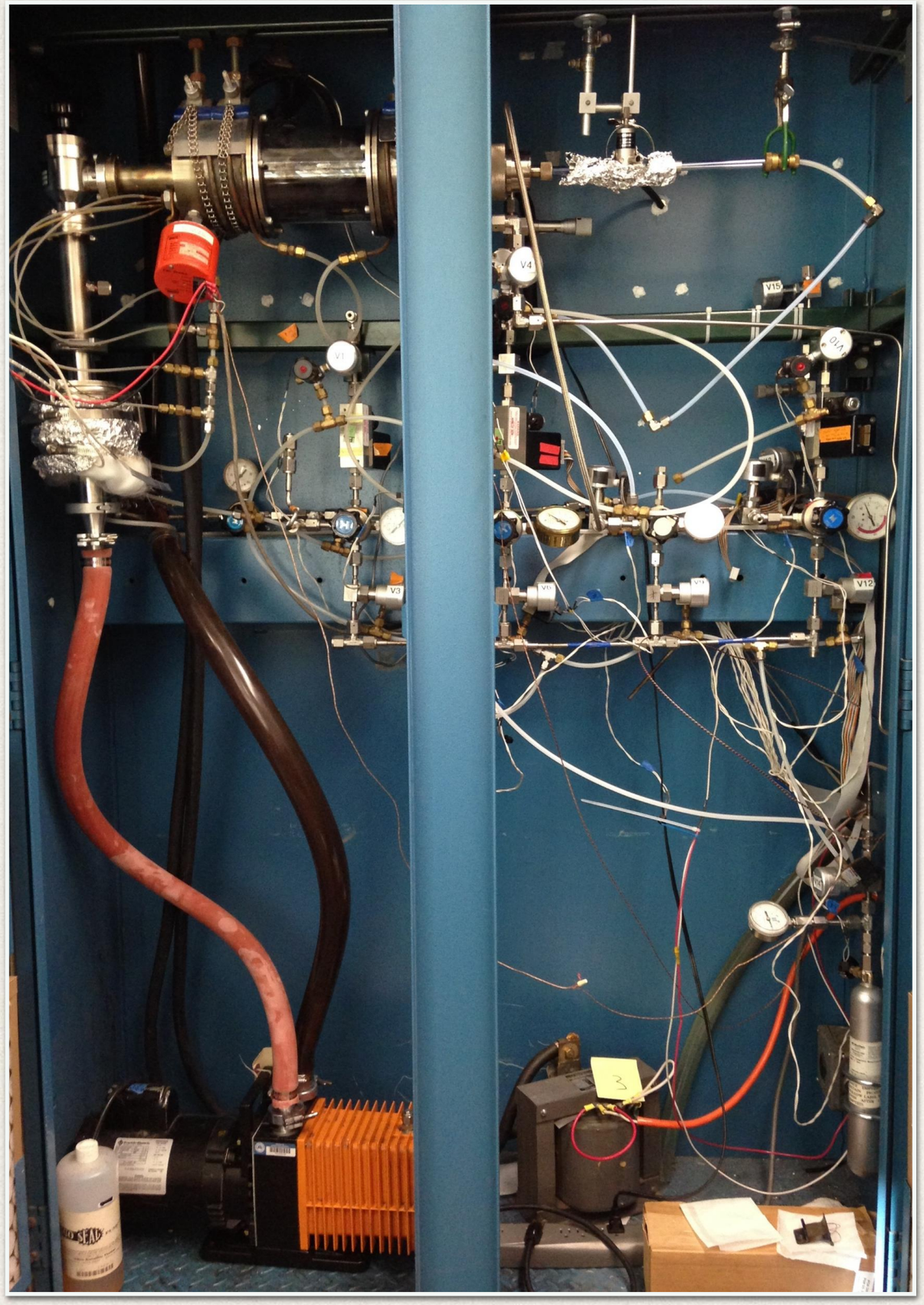
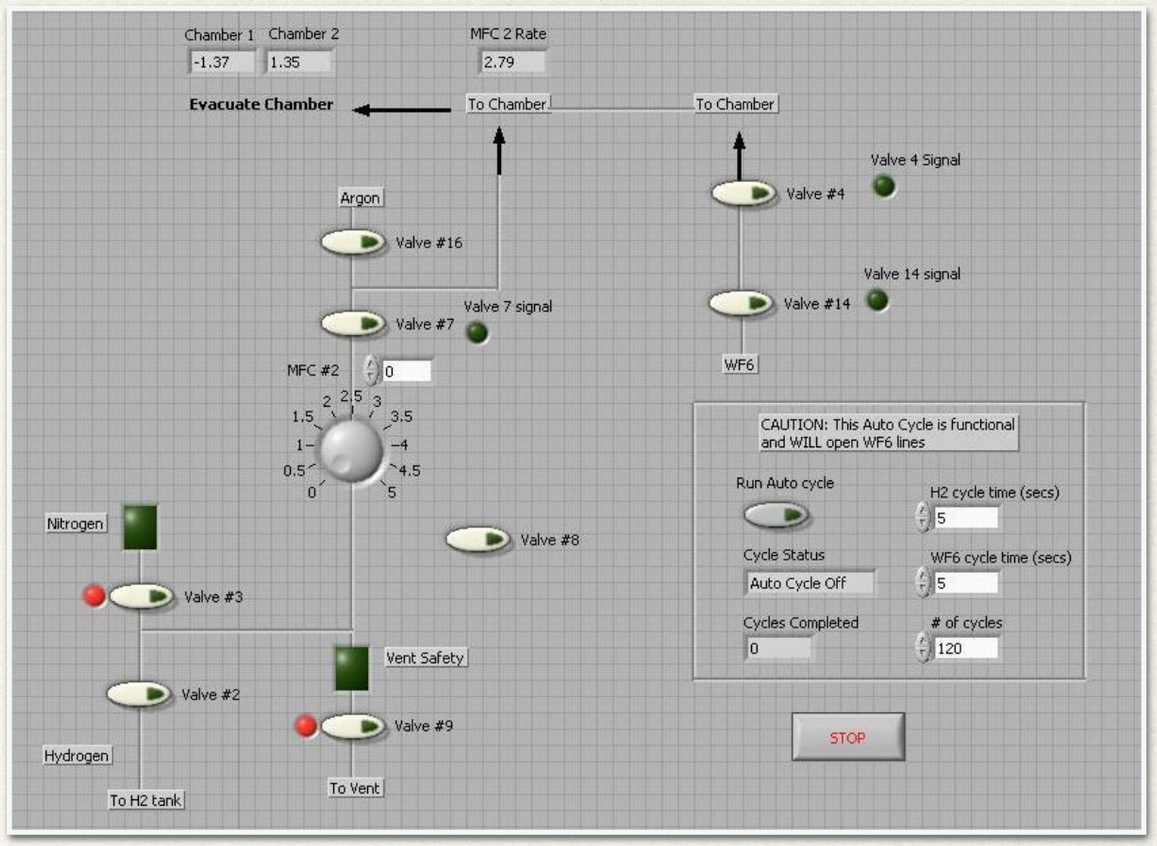
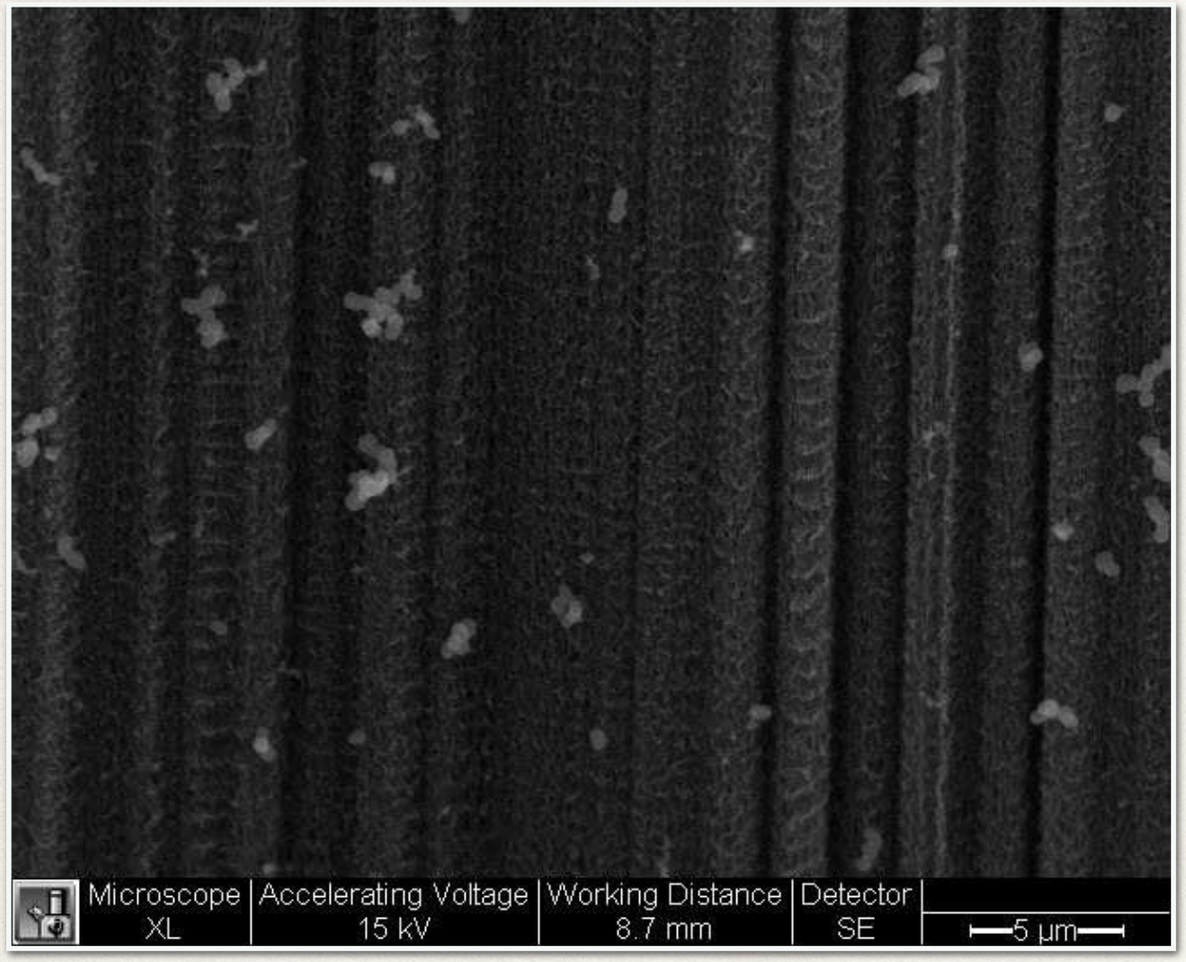
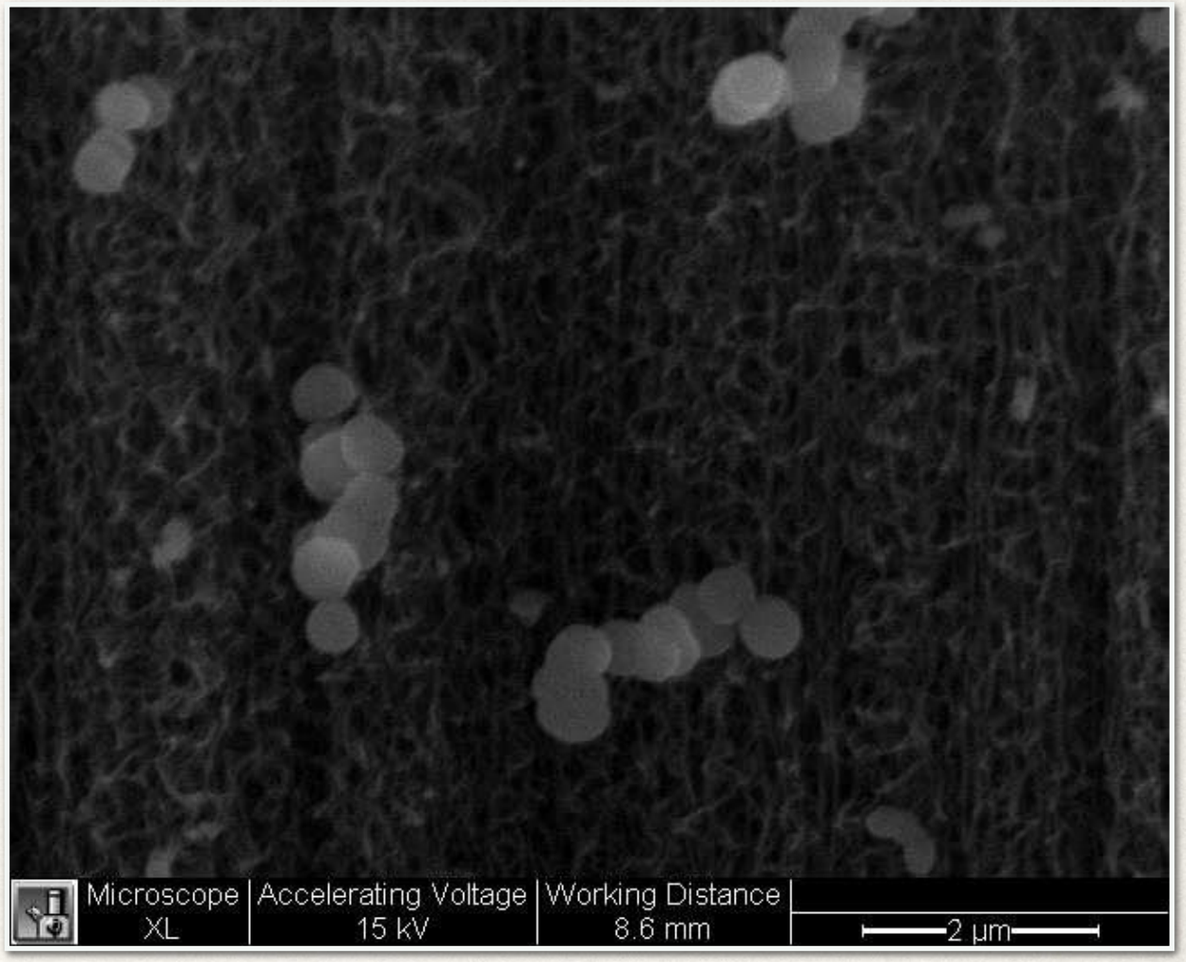




Initial Results





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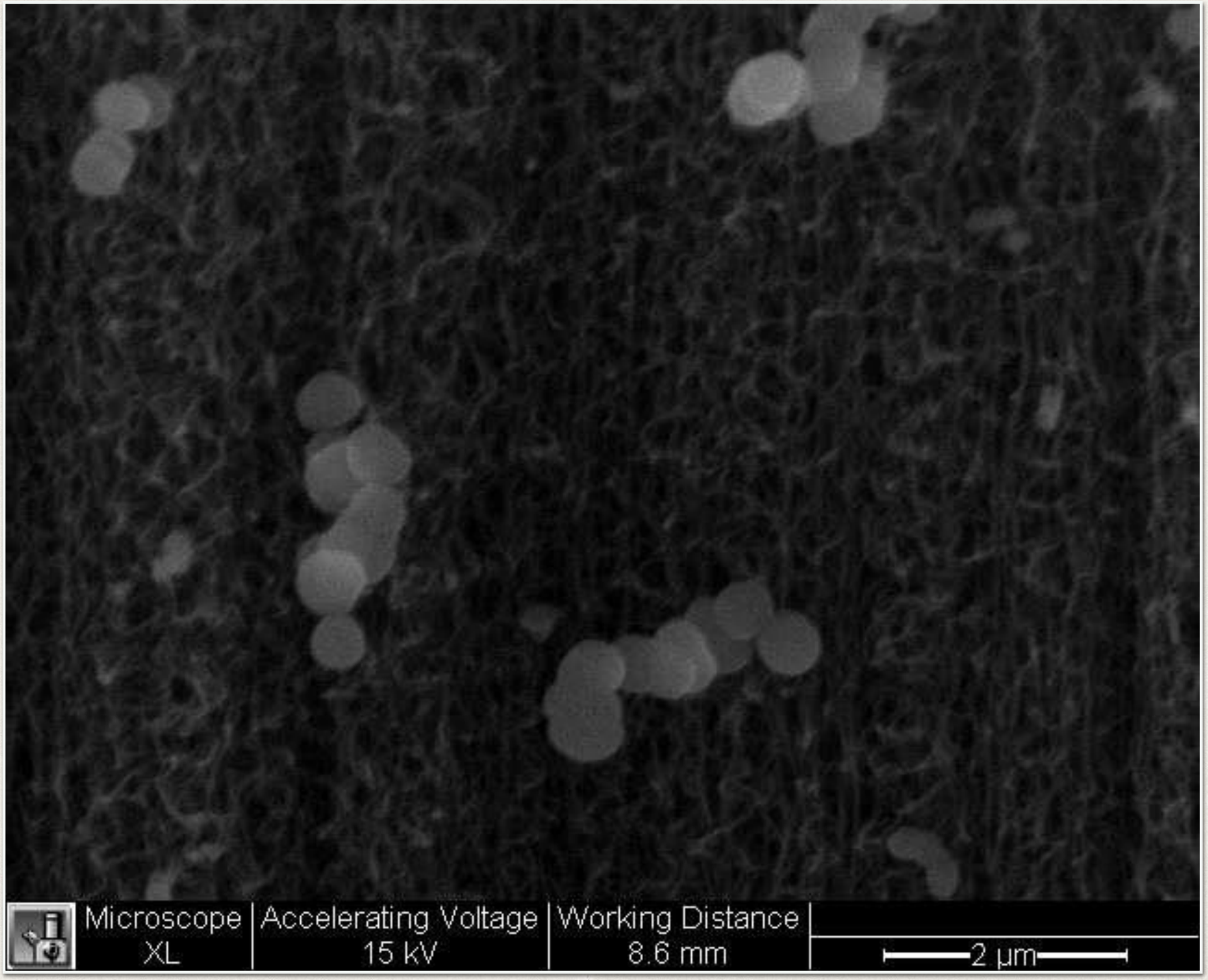
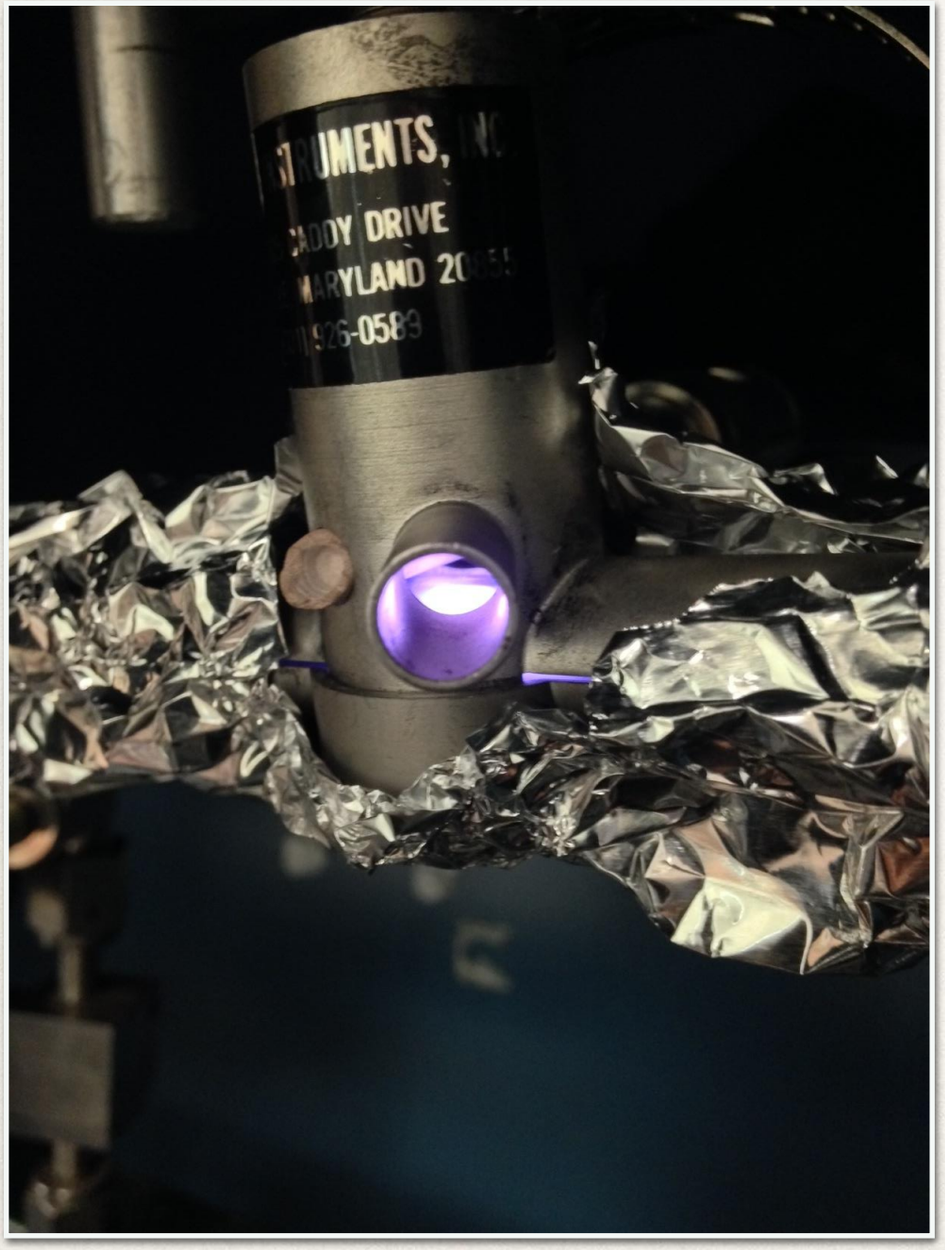
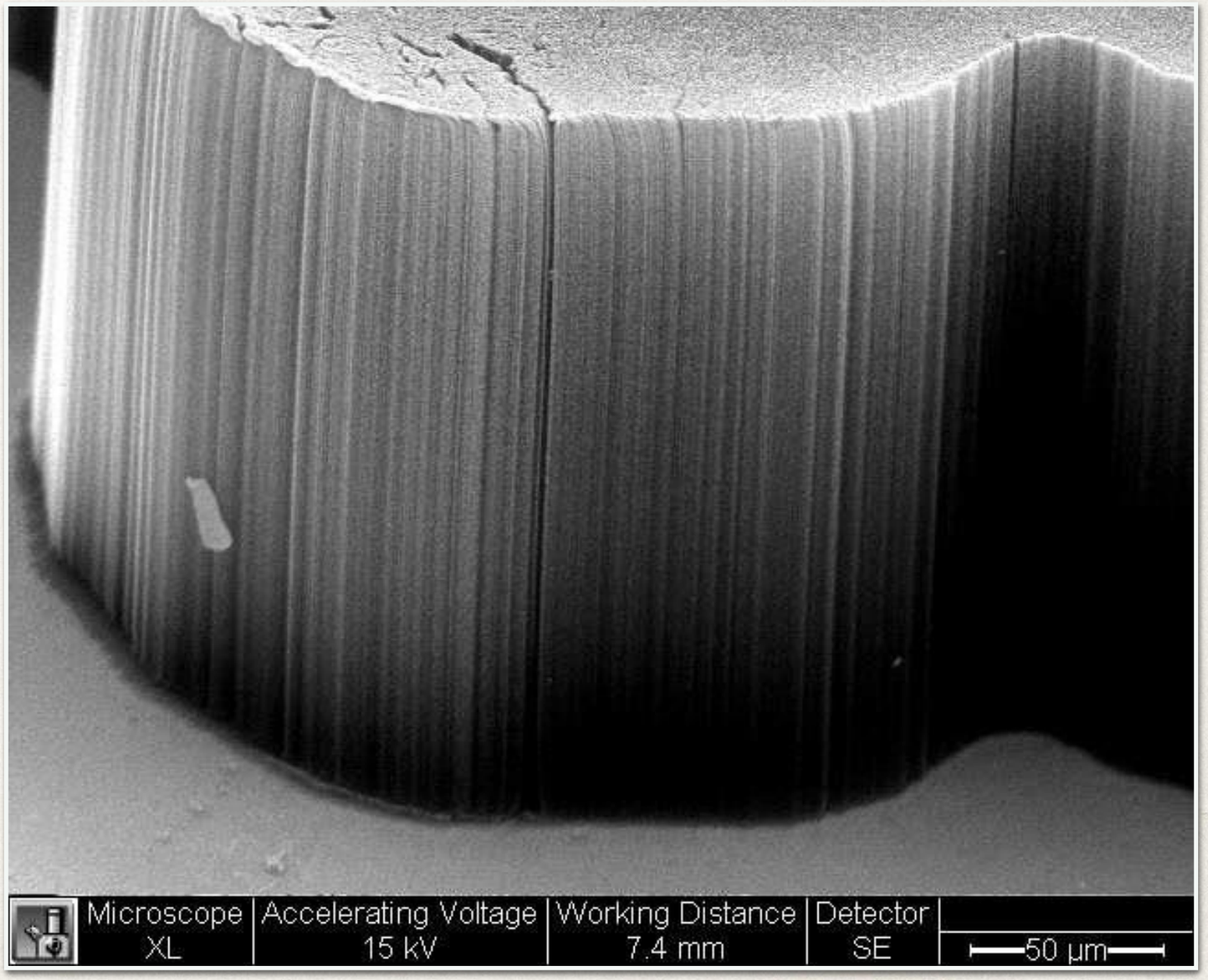


Plasma - RF generated

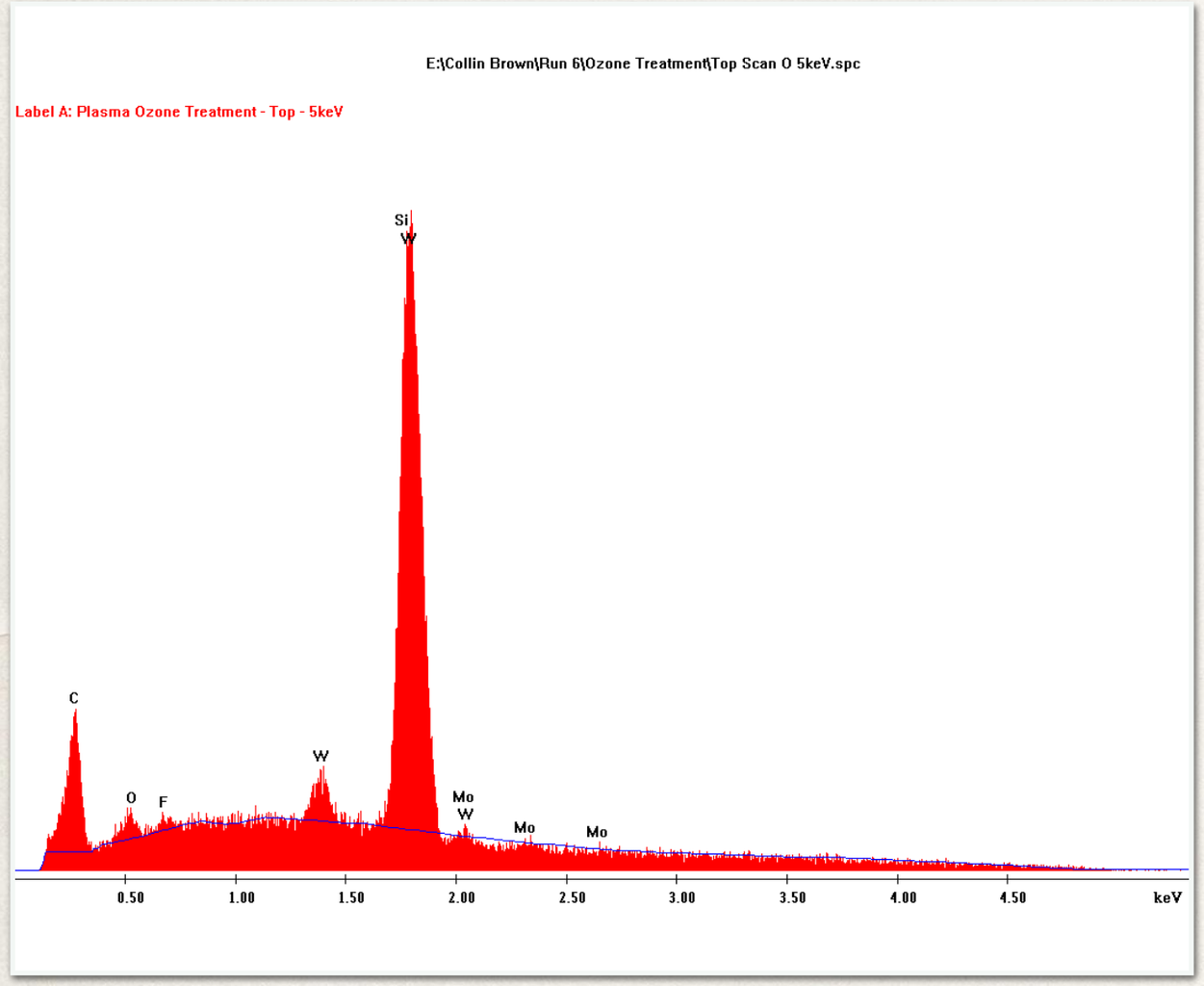




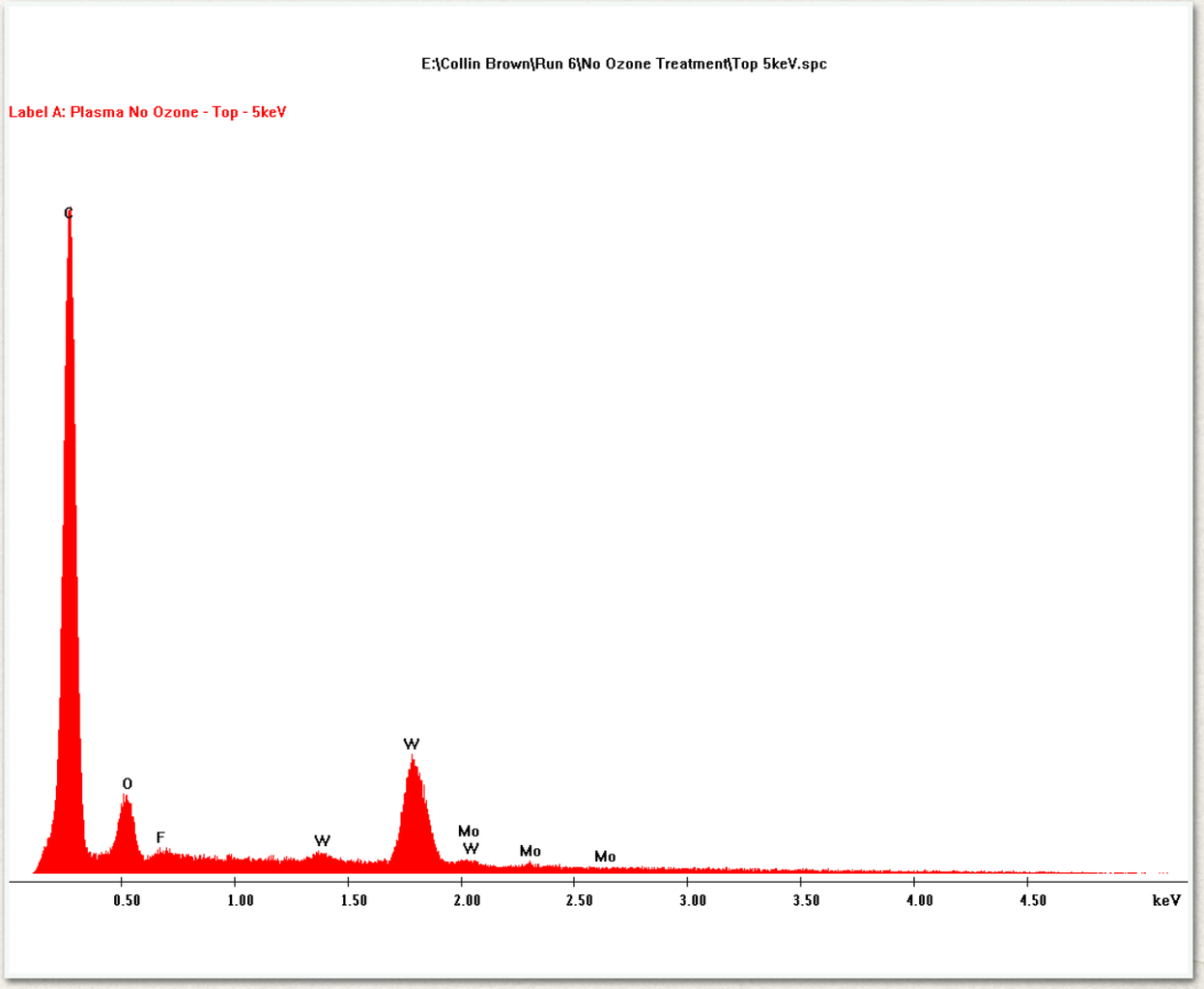
Latest Deposition - Pseudo ALD

- Constant H •(40 sccm) - from a Hydrogen Plasma
- 5 seconds of WF_6 (0.0142 l at 5 psi, about 2.0 x 10^{-4} mole)
- 120 cycles
- 235 degrees C - sample temperature
- Ozone treated 1 of the samples for 20 min, for better nucleation

Ozone treatment vs No Ozone treatment



Ozone Treatment



No Ozone Treatment

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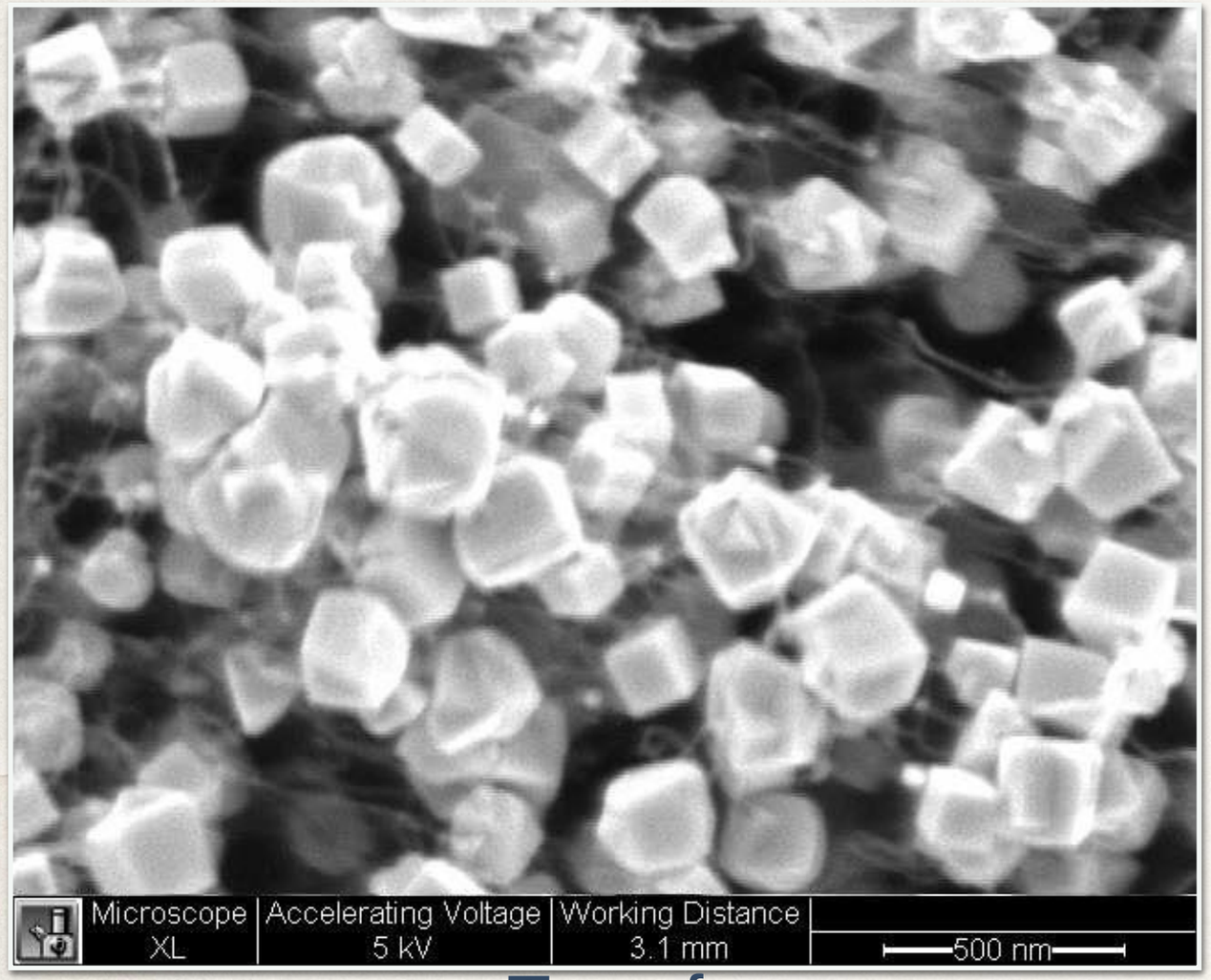
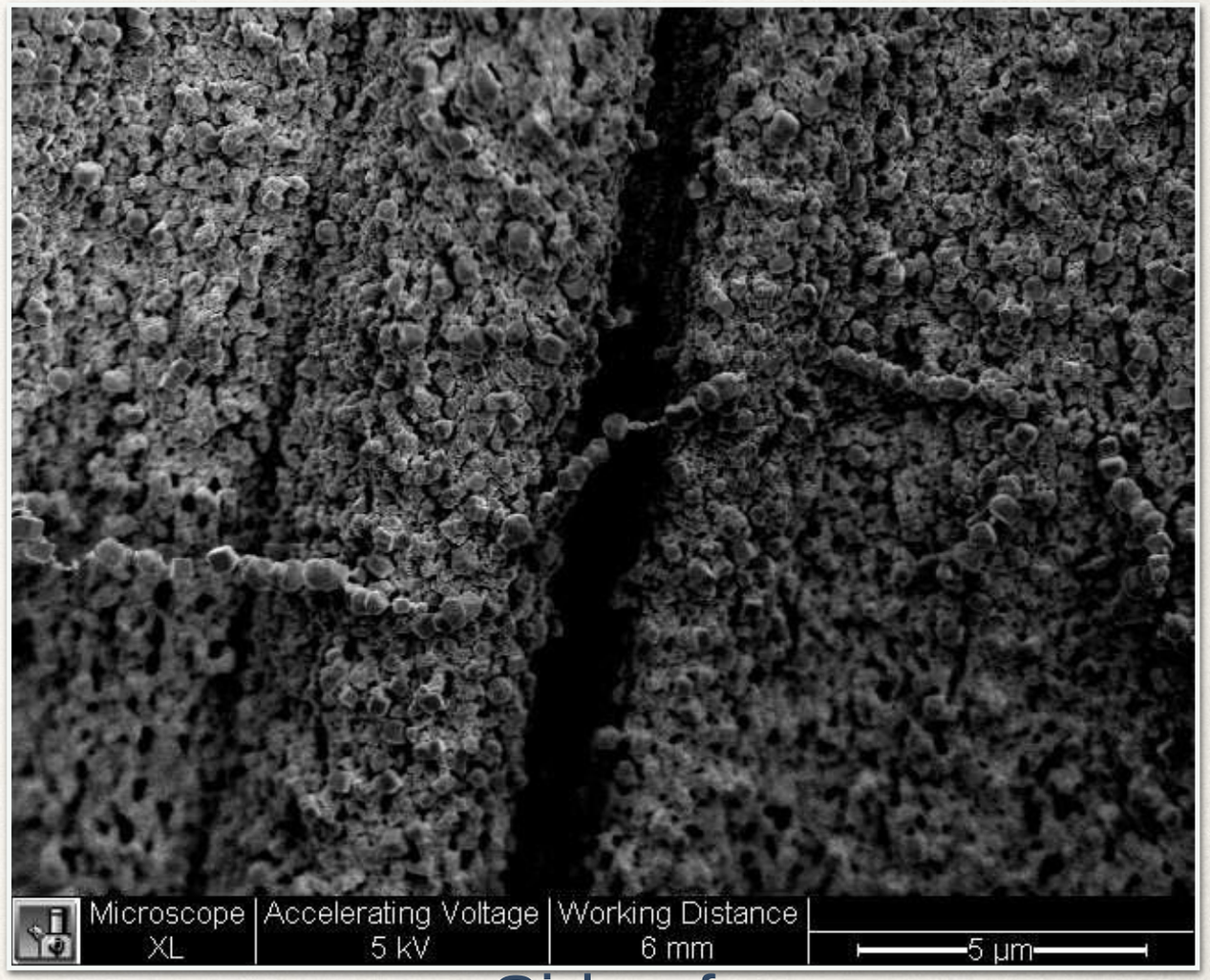


Top of Sample



Side of Sample

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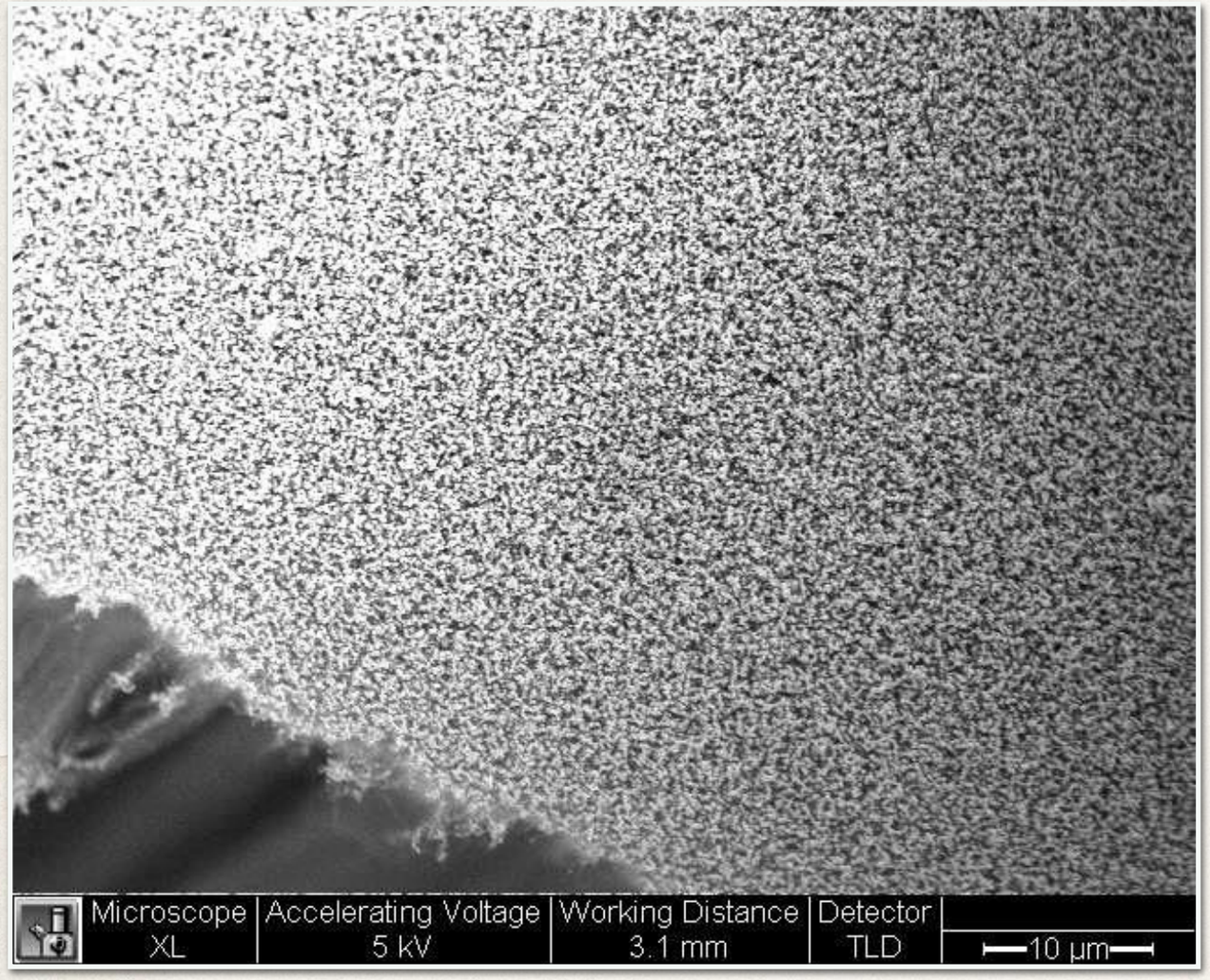


Top of Sample - Break in Lower Left

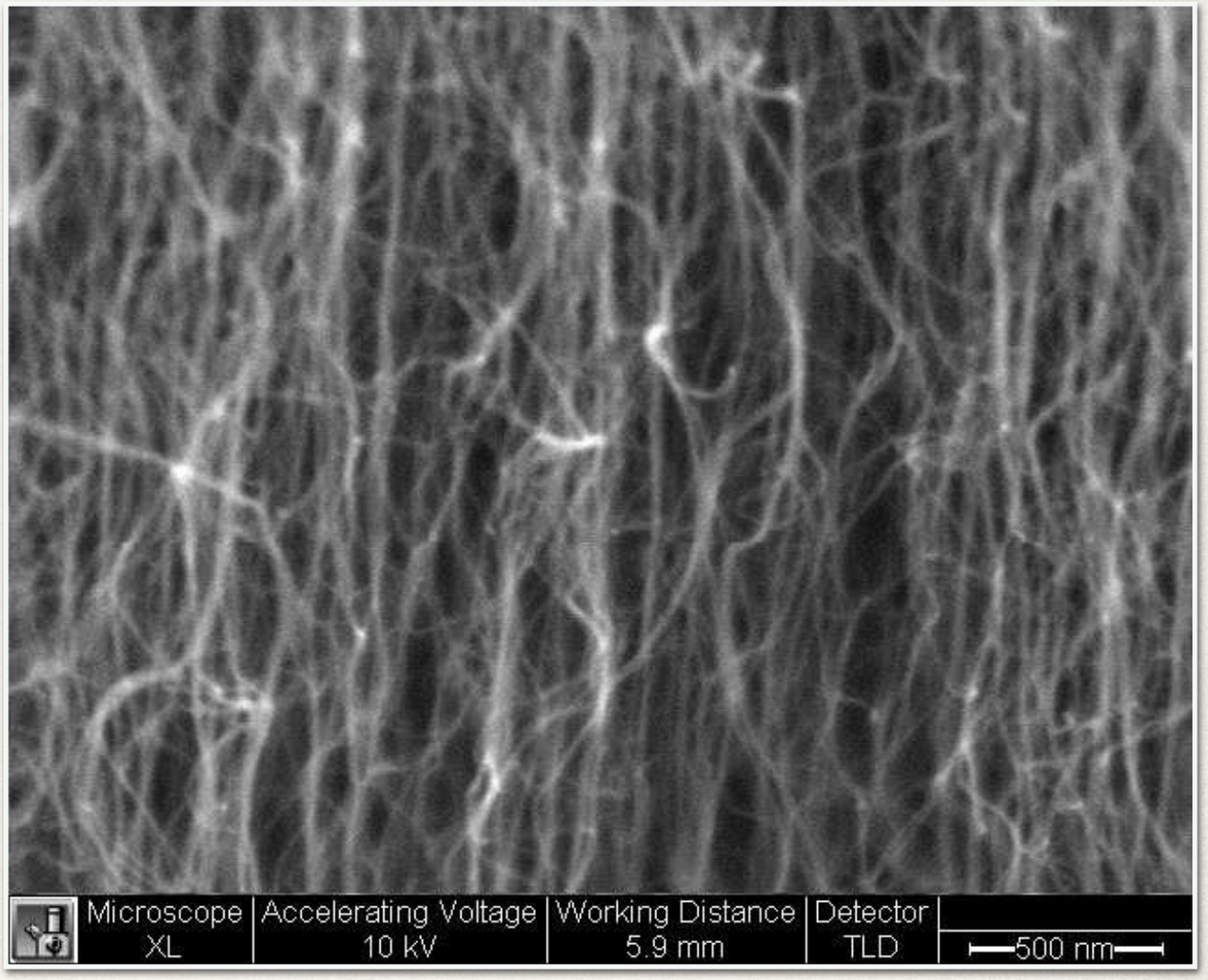
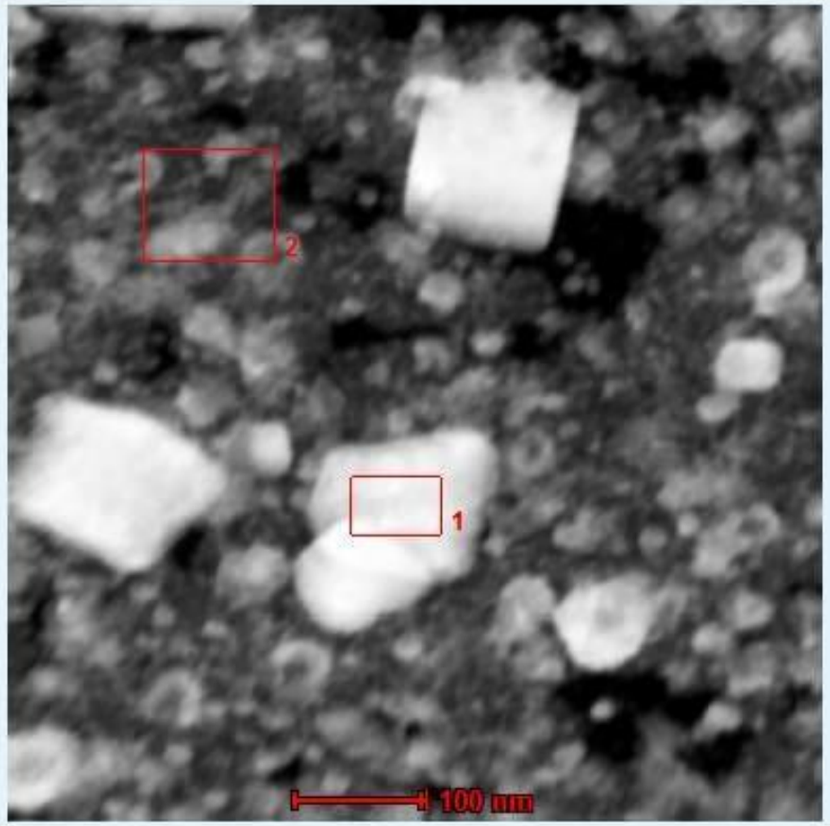
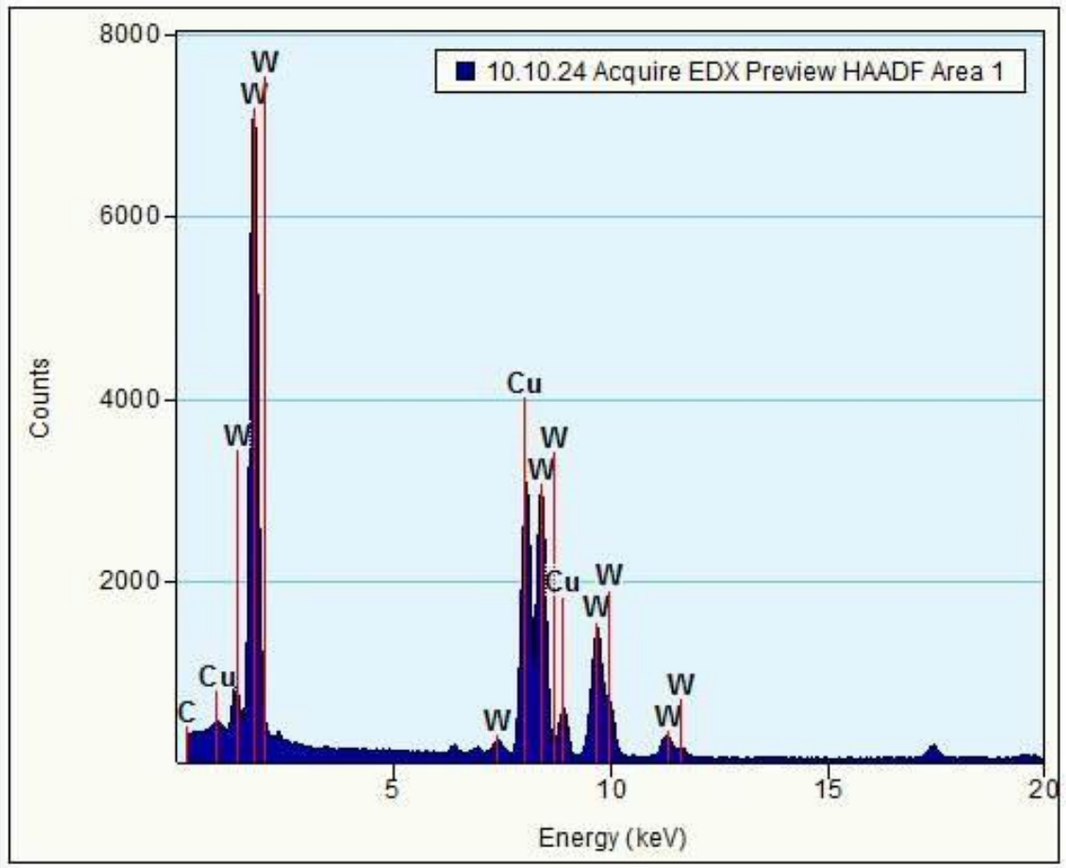
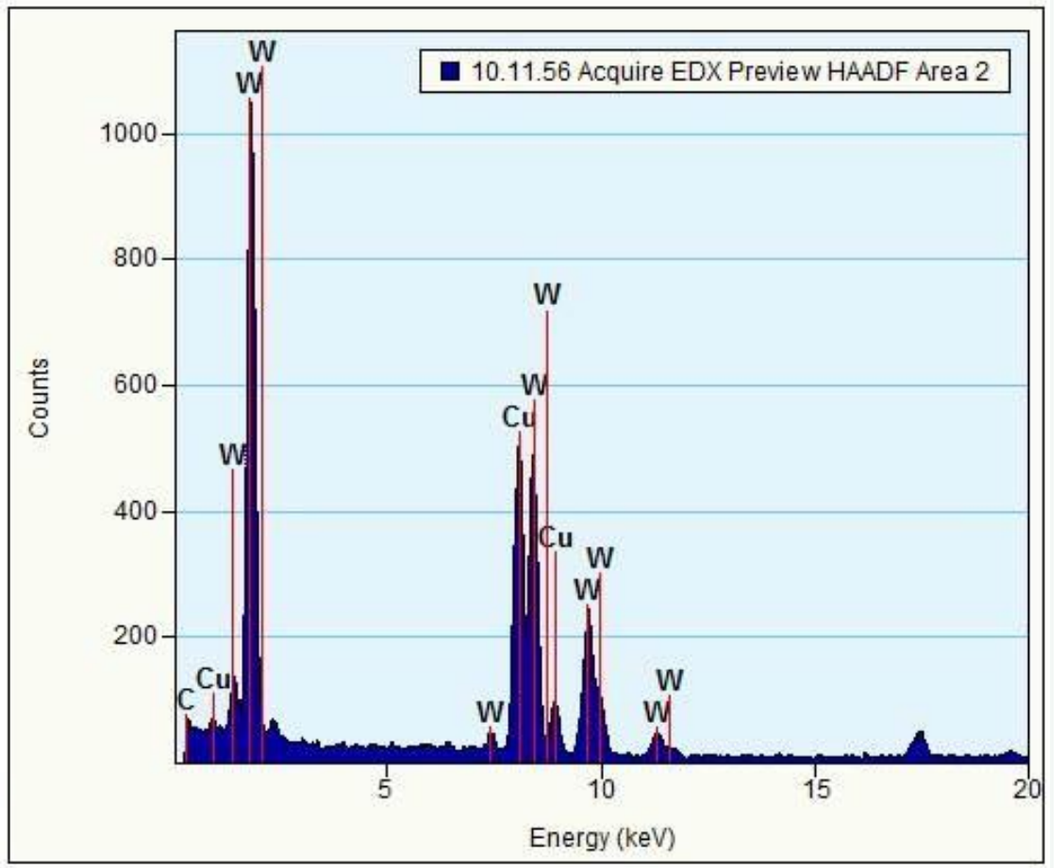


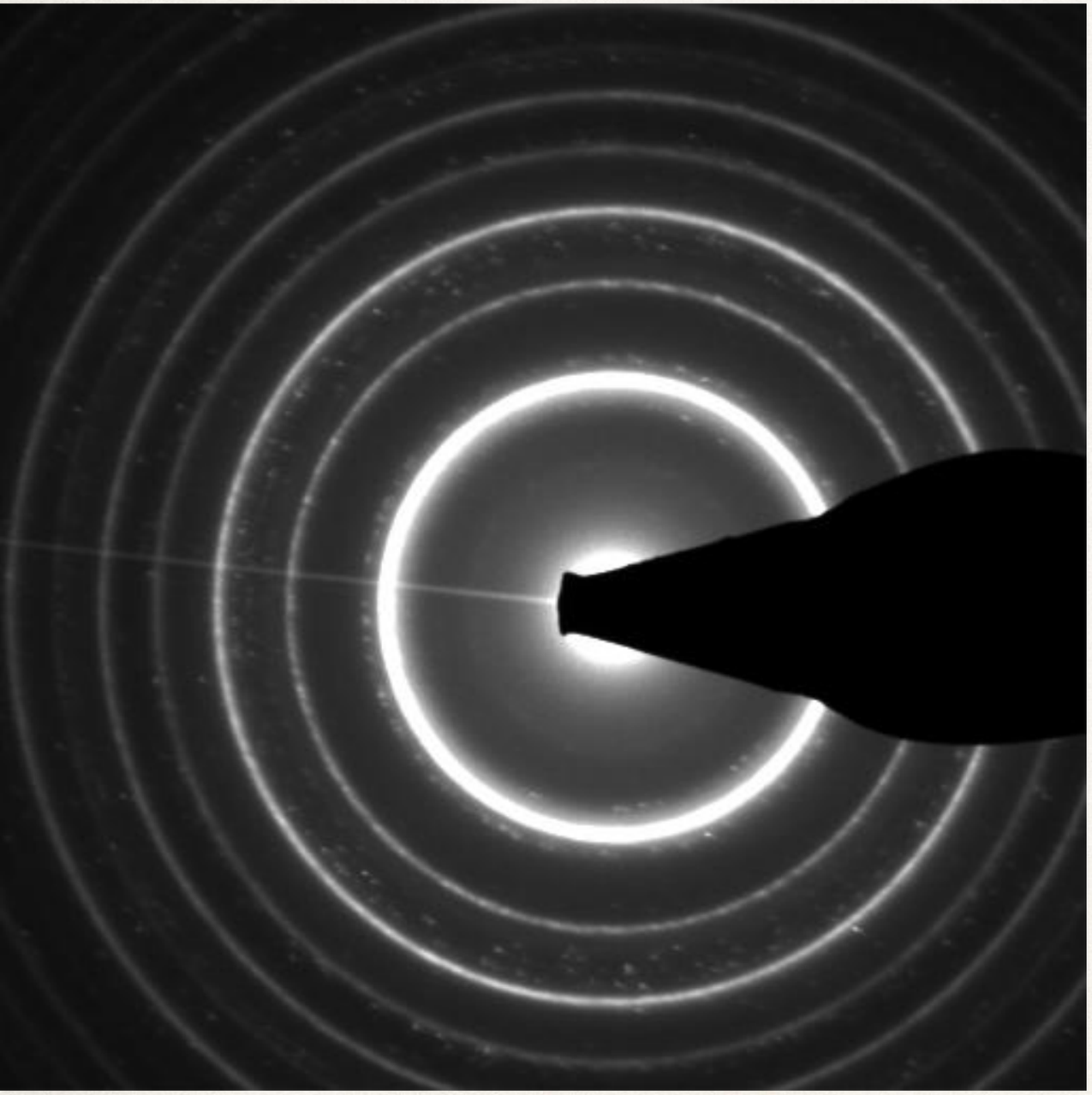
Image of Fresh Surface from Break

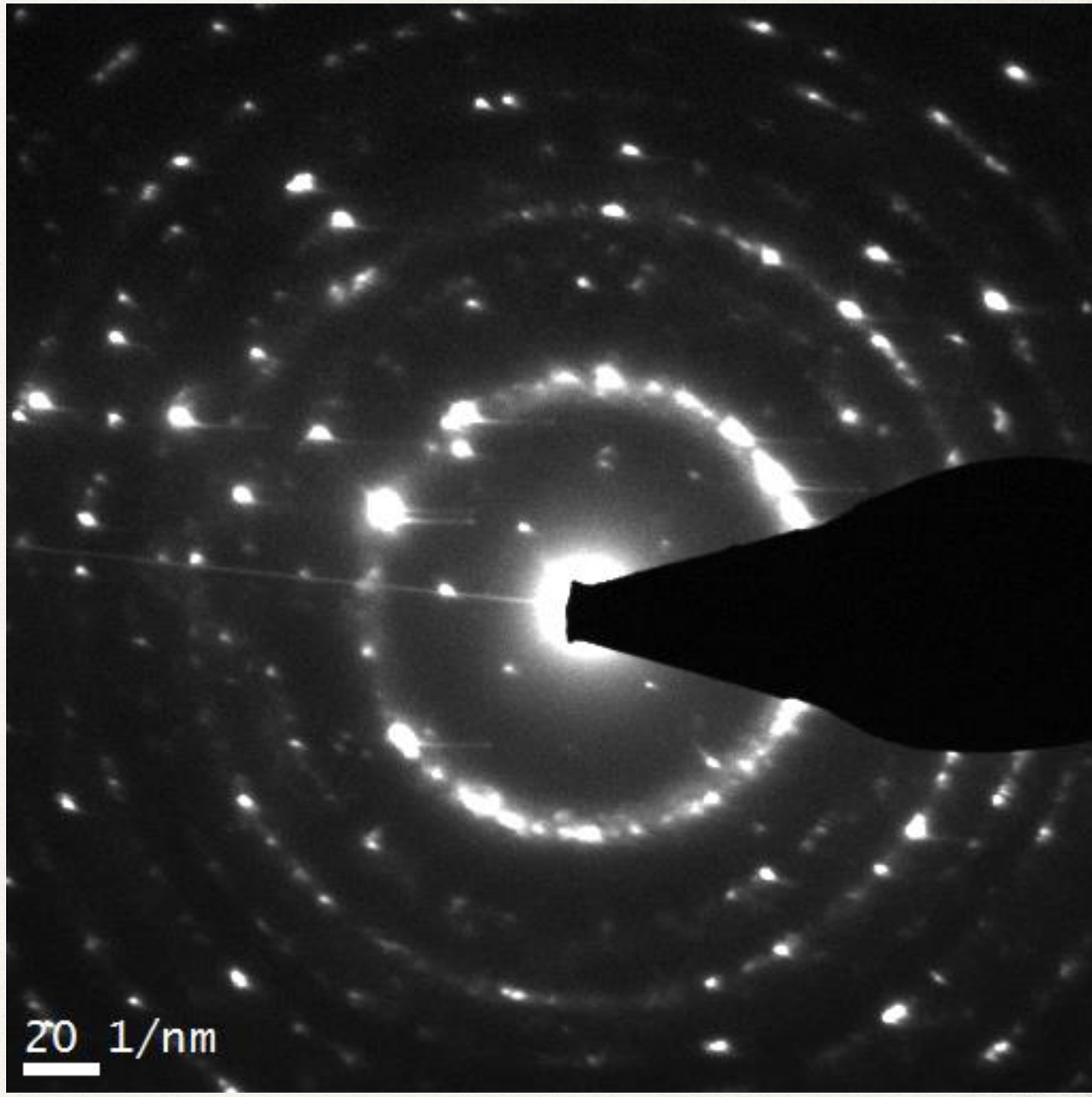


No obvious difference between chemical makeup of small or large stuff









- scattered peaks have a lattice spacing too large for BCC tungsten
- may be an A15 compound, something like Beta-Tungsten

Future Plans

- Optimize procedure (Intervals, flow rates, etc.)
  - Silane vs. Hydrogen
  - Ozone treatment vs. No Ozone treatment vs. *In Situ* Ozone treatment
  - Annealing
- Characterization by SEM & TEM cross sectioning, Also X-ray characterization at ALS, FIB
  - Capping
  - Grain size uniformity
- Electrical conductivity
- Mechanical Properties

Sources

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