SAND2015-6197C

STAR Fellowship in CINT's Electron Microscopy Lab

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Center for Integrated Nanotechnologies
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2015 July 29

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CINT

http://sstp.org/wp-content/uploads/CINT.jpg

Department of Energy Nanoscale Science Research Center

https://nsrcportal.sandia.gov/

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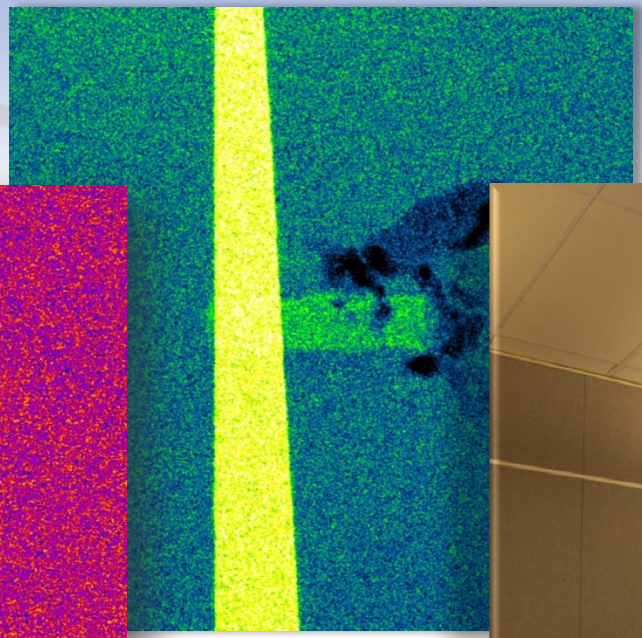
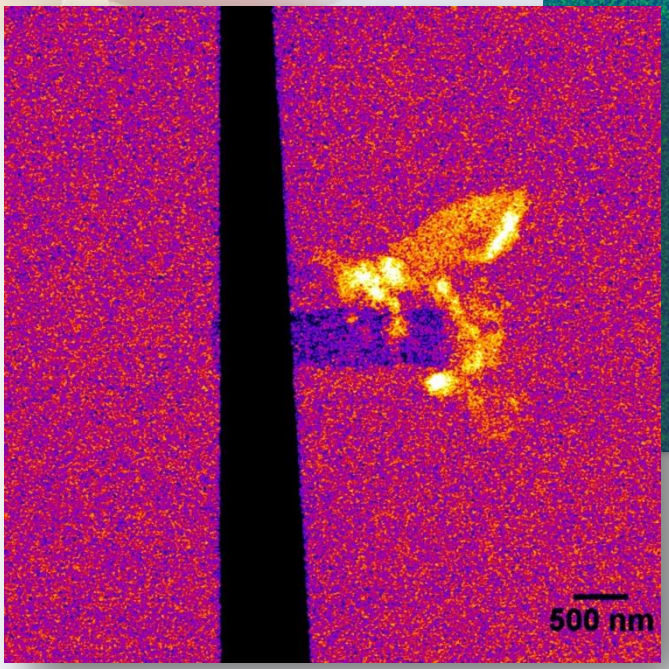
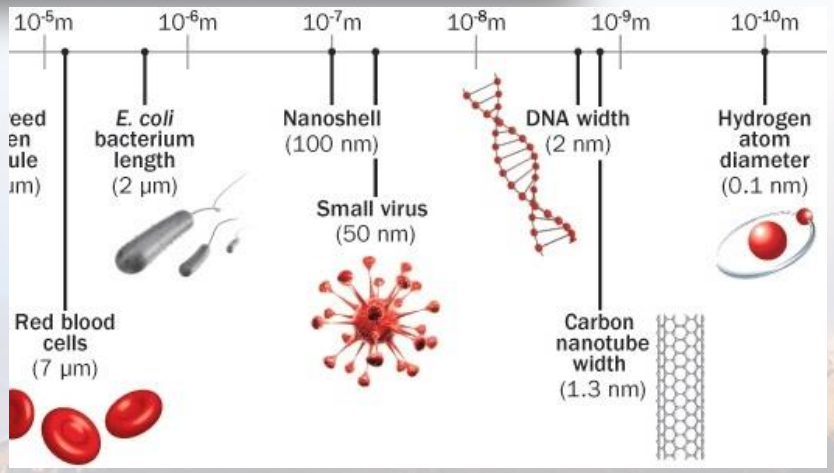
TEM



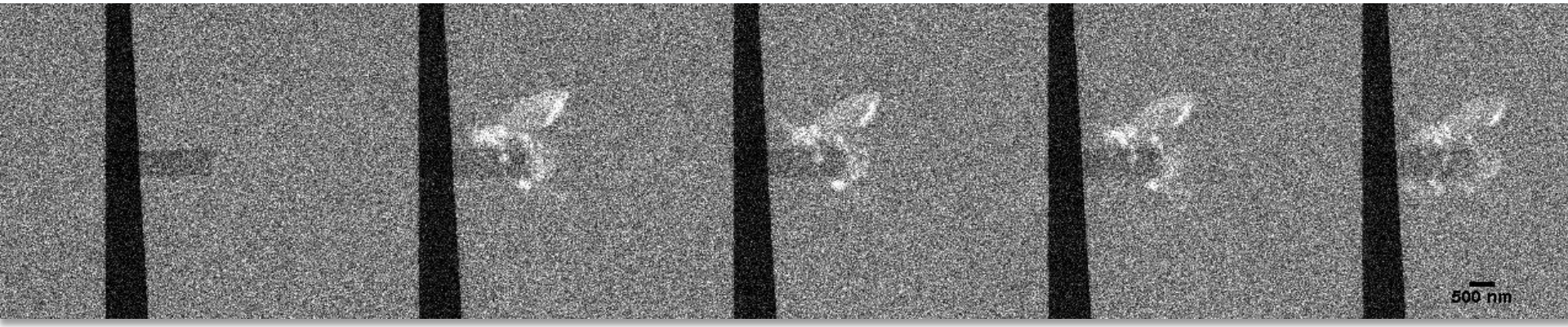




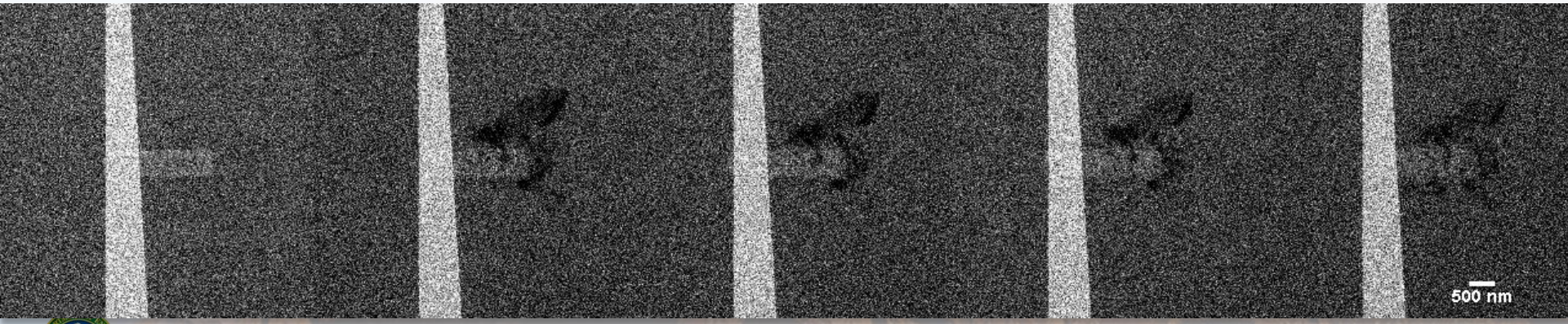
https://www.sciencenews.org/pictures/120410/nanotubes_sizes_zoom.gif

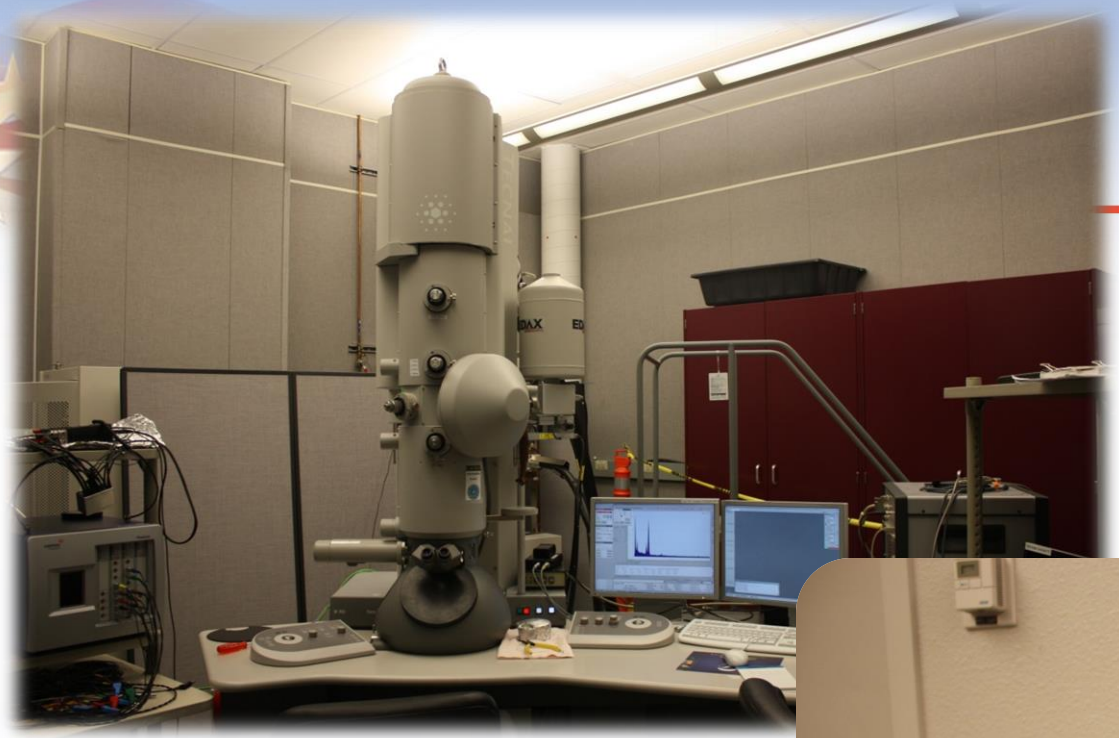
Image processing

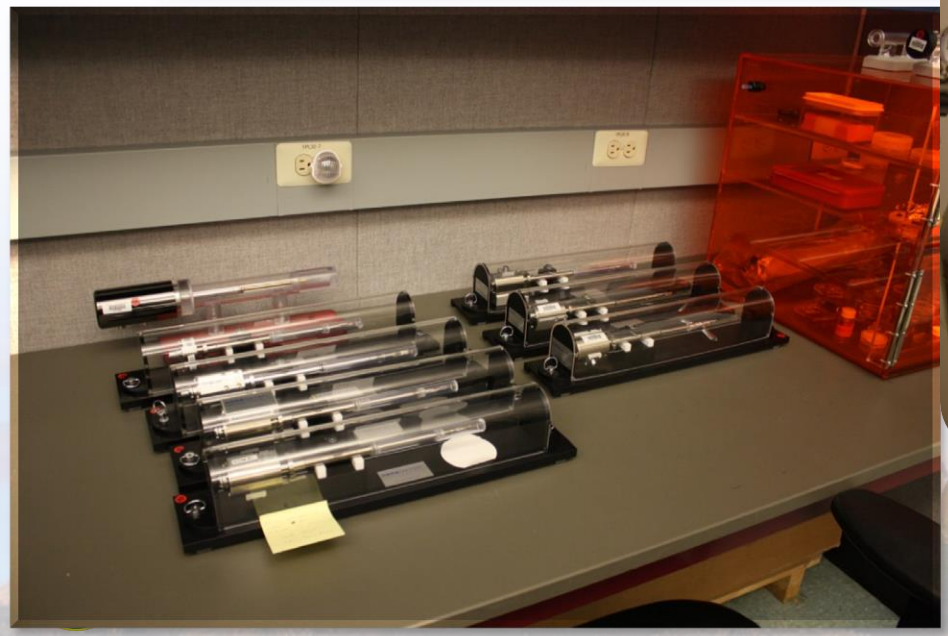
Bright Field

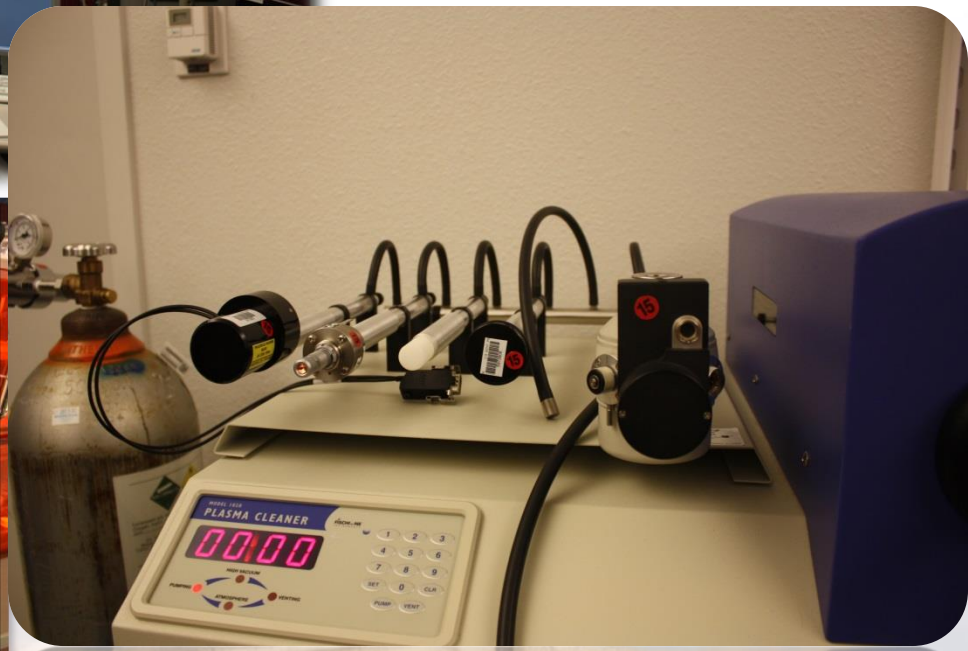


Dark Field









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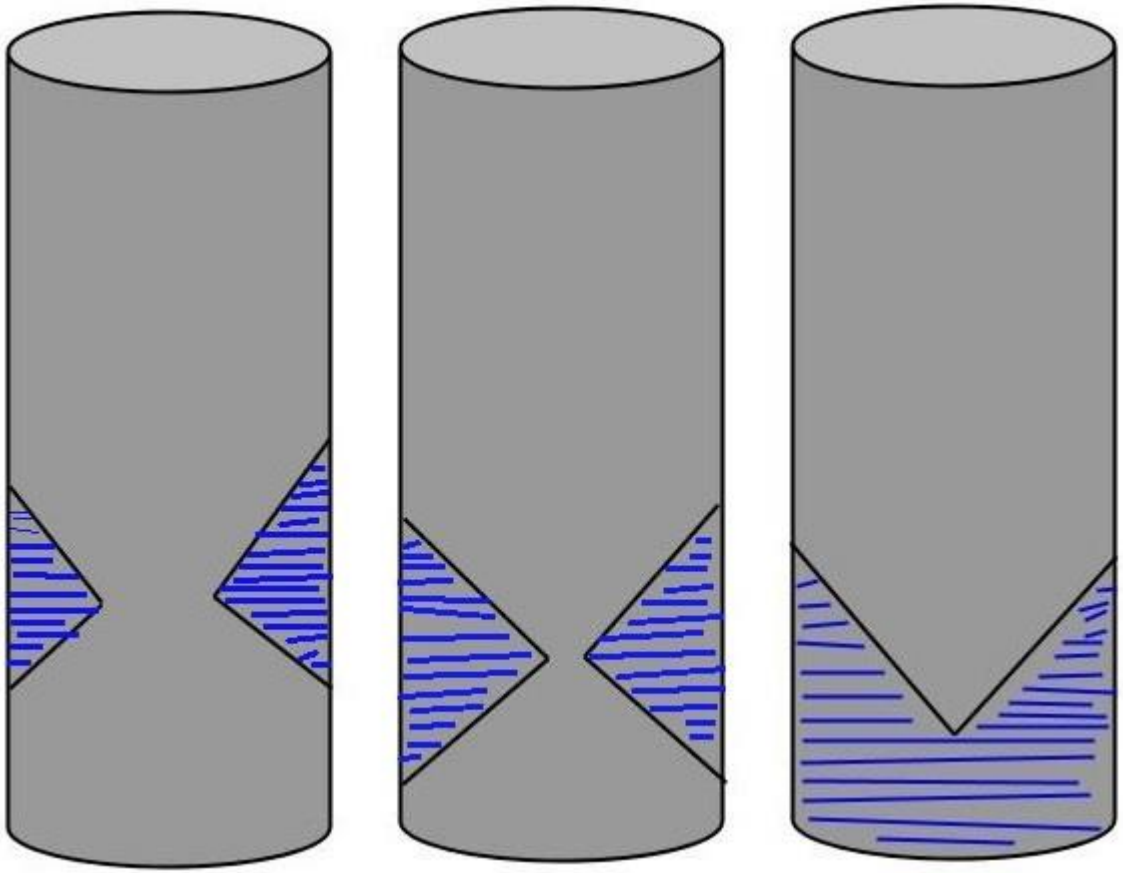
CAUTION
OPEN DOOR
SLOWLY


Leica

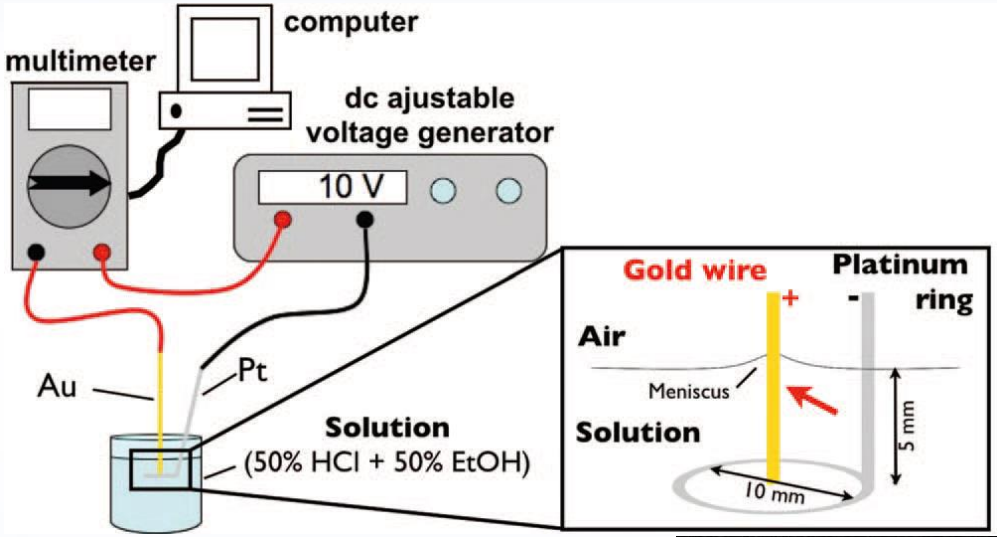

EM UC7

Electrochemical Etching of Probes

- Using a mixture of chemicals and applying a current to the solution to etch away the material from a thin wire to form a very sharp point



Background



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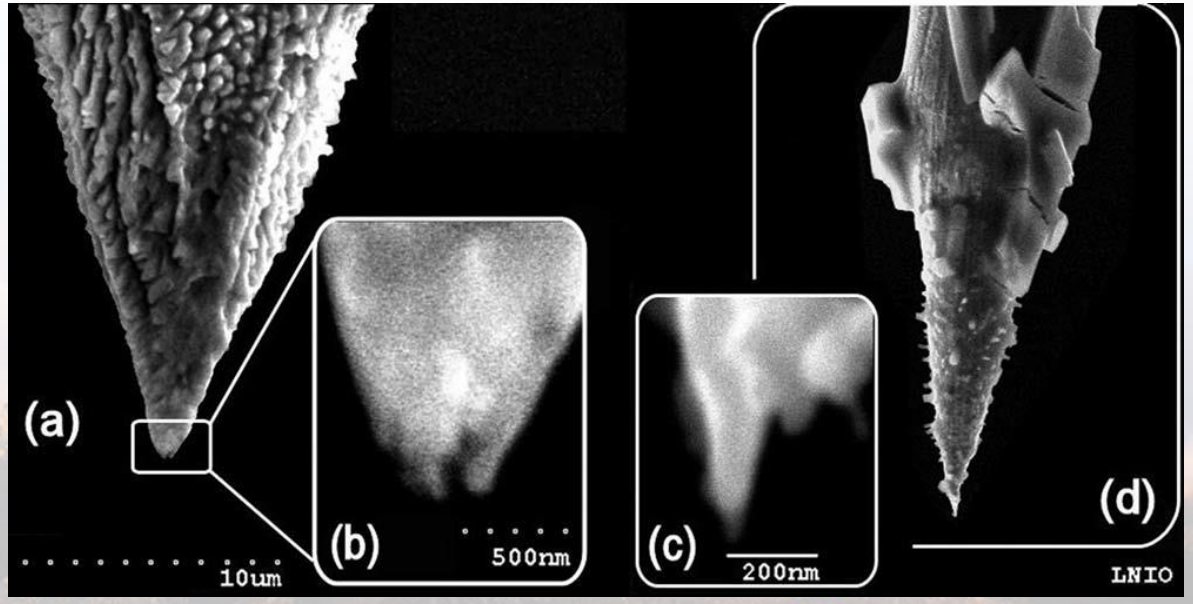
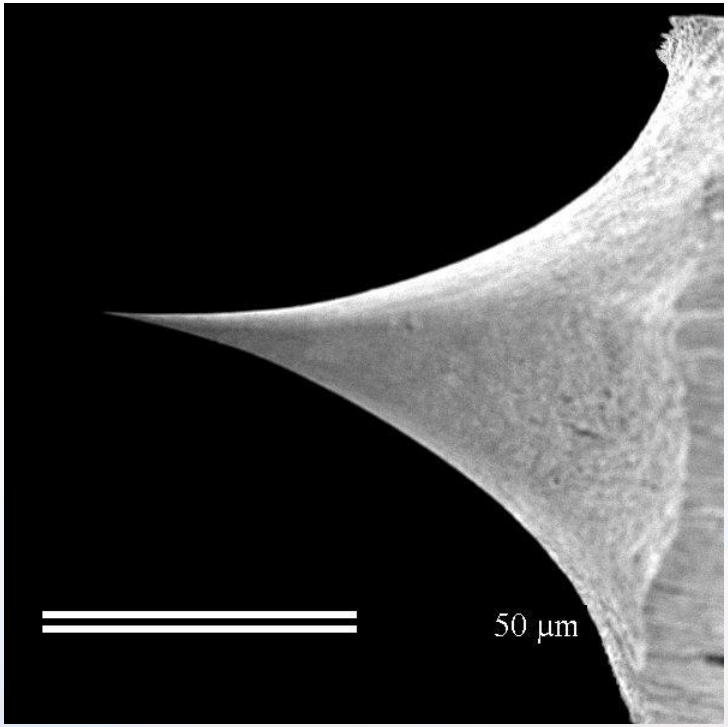
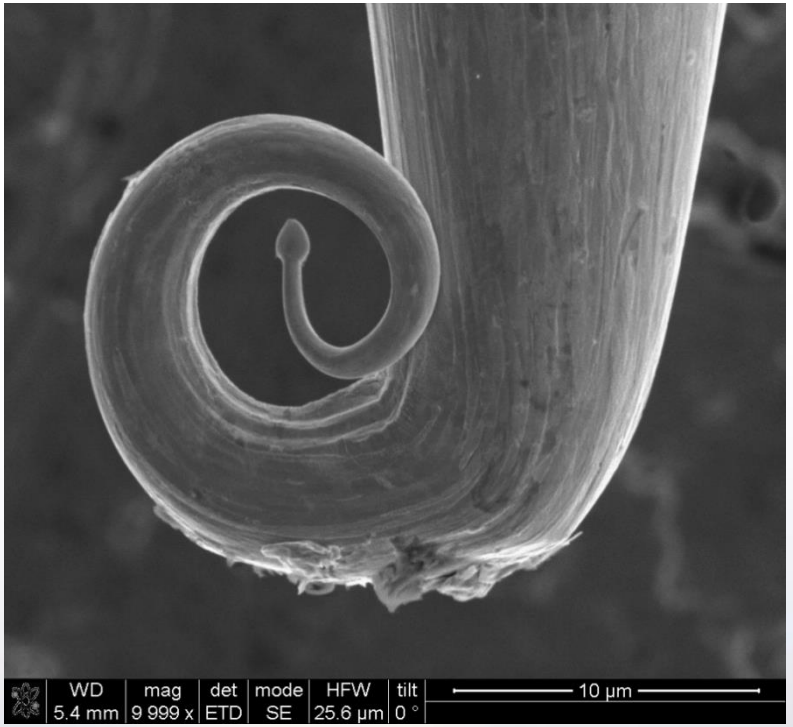
doi: 10.1063/1.4812365

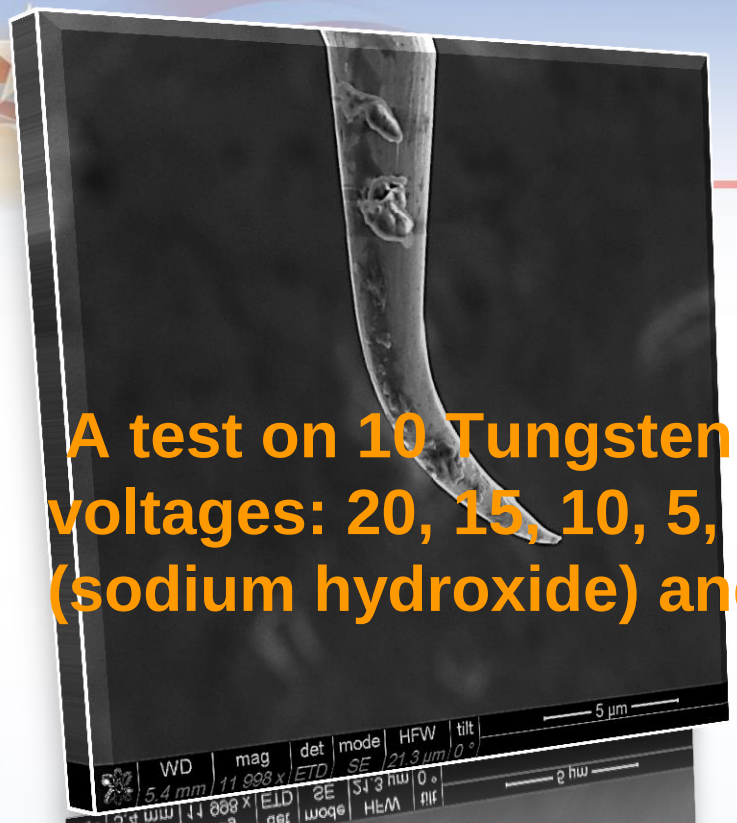


Expectations and Reality



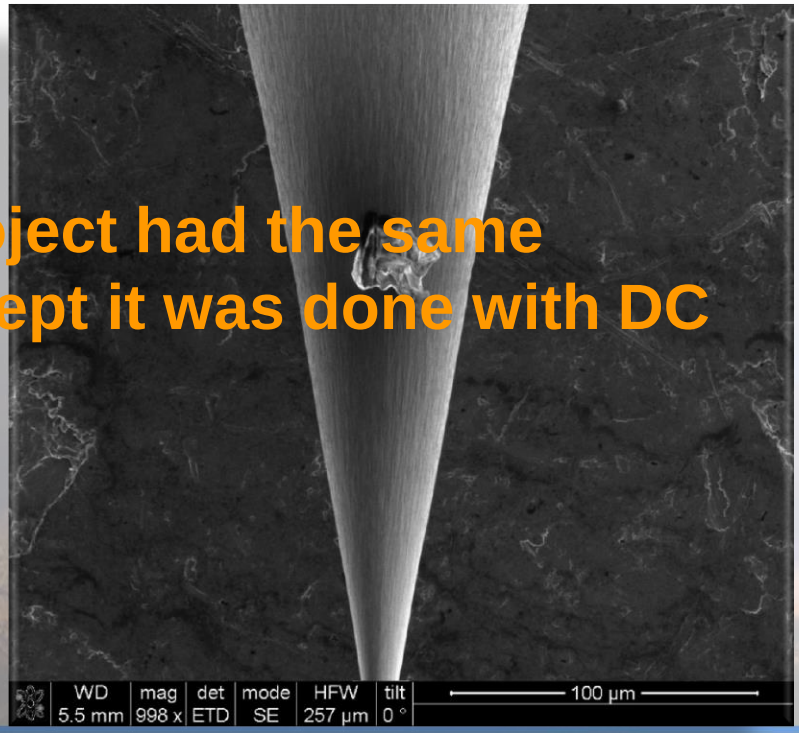


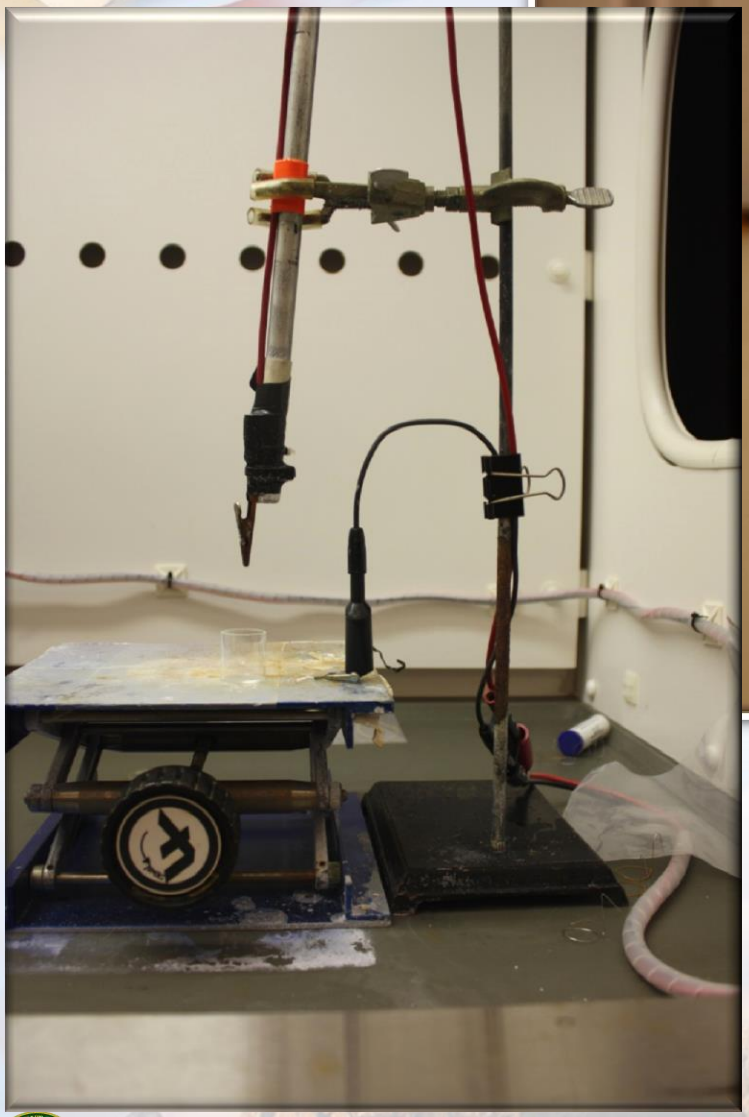
My Project



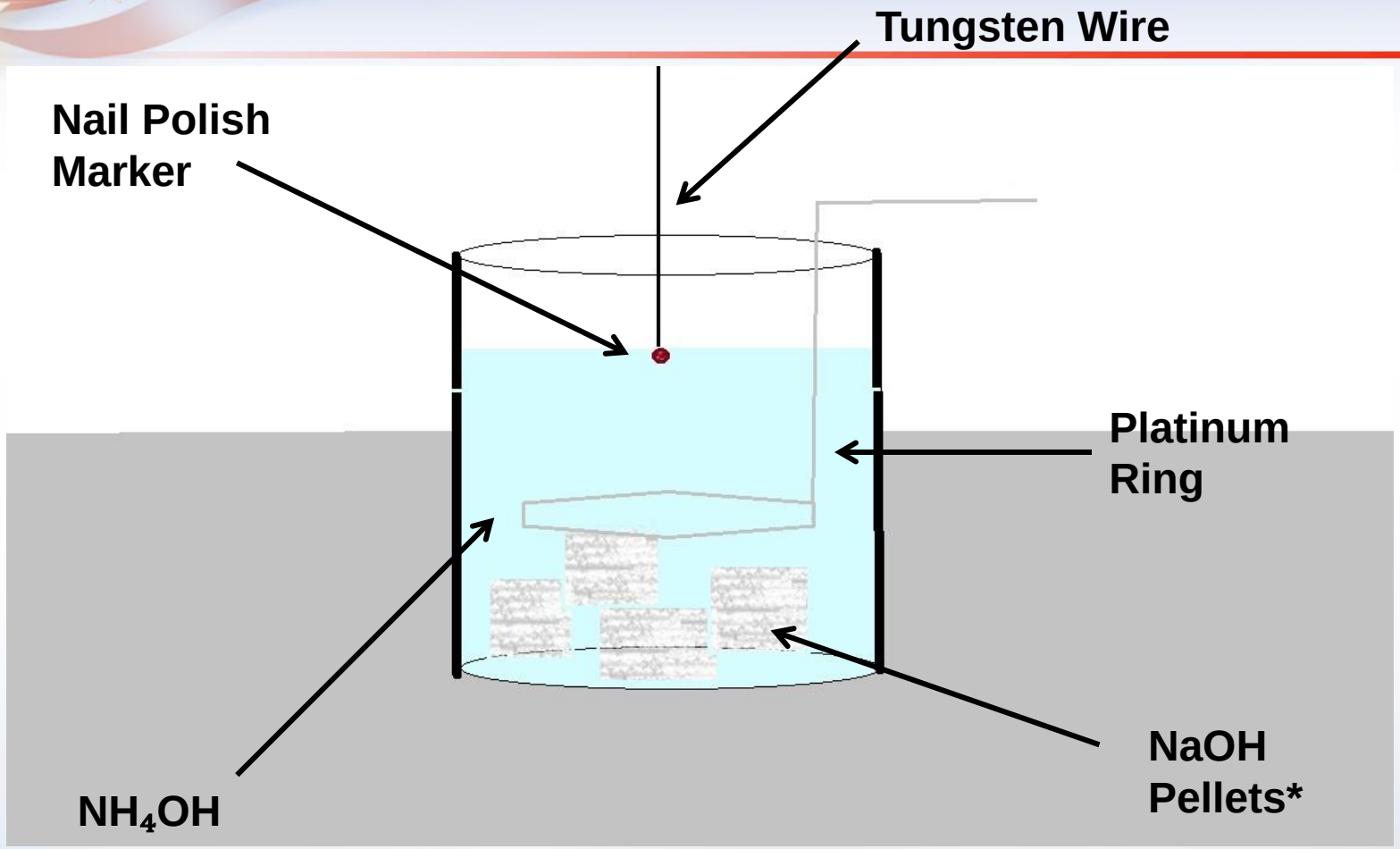
A test on 10 Tungsten probes etched at constant AC voltages: 20, 15, 10, 5, and 2.5 in a solution of NaOH (sodium hydroxide) and NH_4OH (ammonium hydroxide)

The second part of my project had the same conditions as the first except it was done with DC voltage instead of AC





Set up for etching is inside of fume hood to ensure safety during the experiment



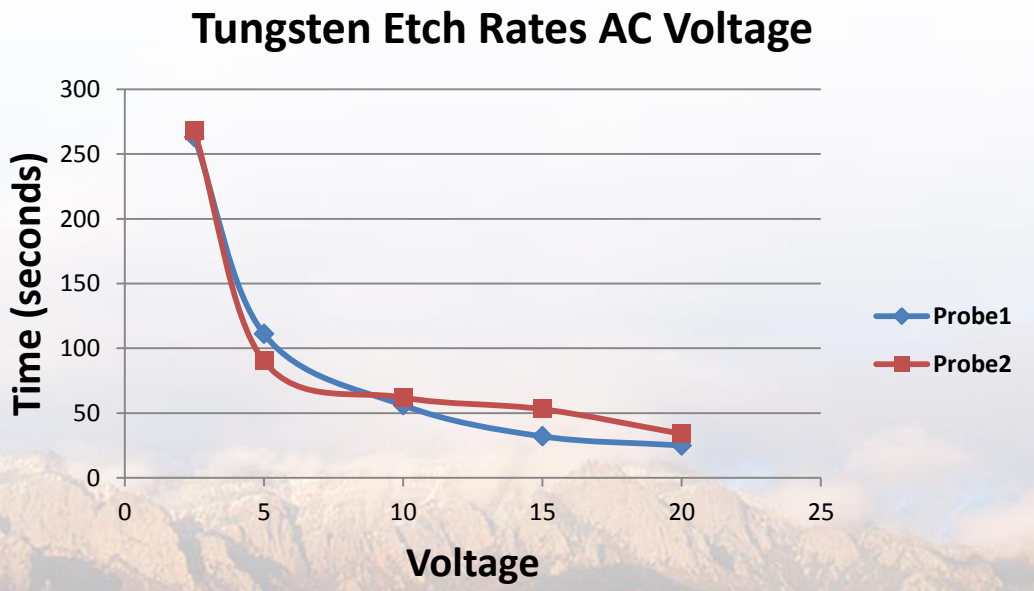
*the pellets should be entirely dissolved in the solution before the Pt ring or W wire are added and the electrolysis process begins

Tungsten Data AC

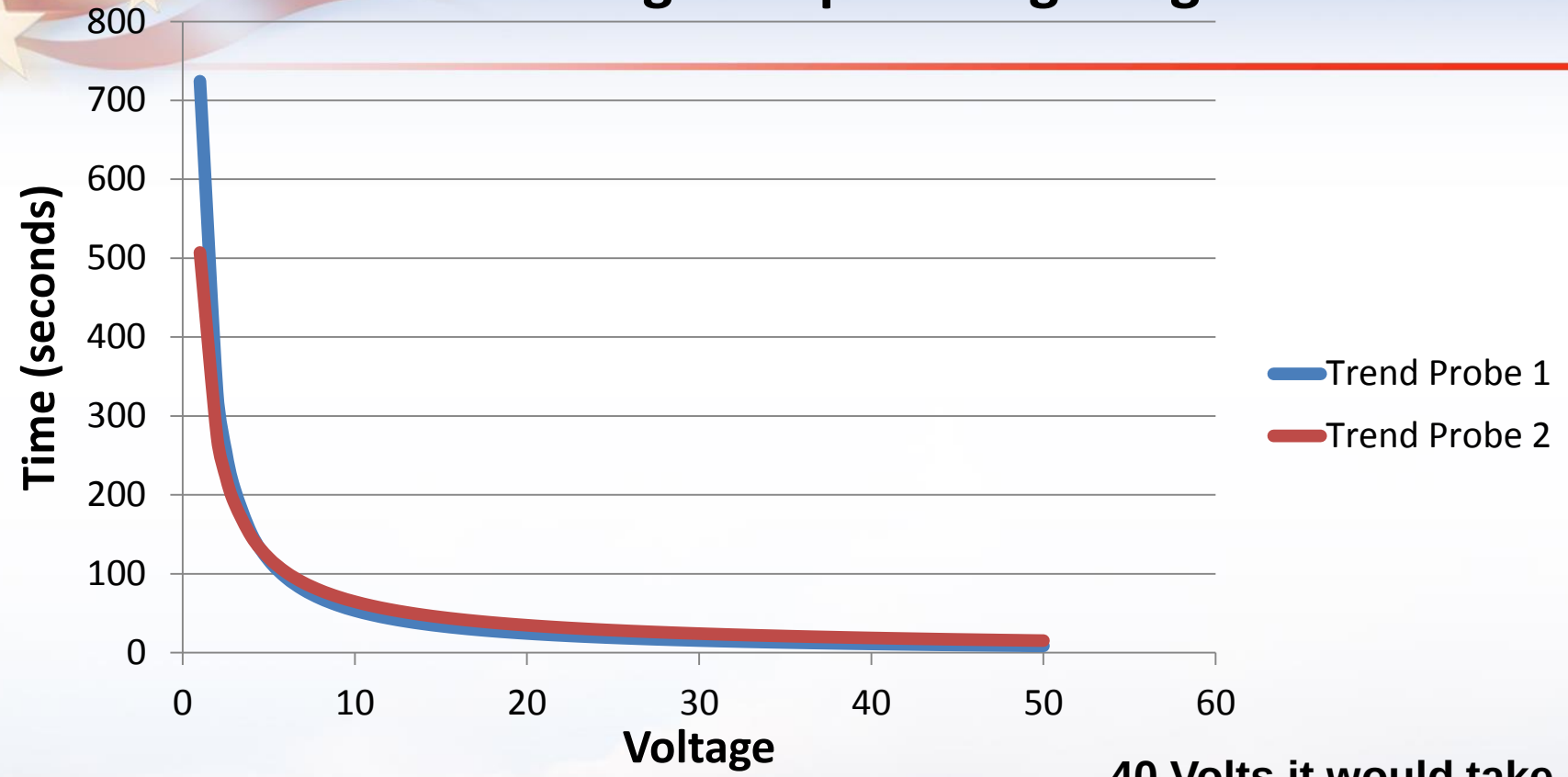
Voltage (Volts)	Time for First Probe (sec)	Time for Second Probe (sec)
20	25	34
15	32	53
10	56	62
5	111	90
2.5	263	268

A trend can bee seen from the data as the amount of time it took the tips to fall of increased as the Voltage was decreased

The graph shows the data starting from the lowest voltage, showing how much longer it took to etch



Estimation For Single Step Etching Tungsten AC



Trend 1: $y = 724.3x^{-1.133}$
$R^2 = 0.9975$

Trend 2: $y = 507.22x^{-0.895}$
$R^2 = 0.9401$

40 Volts it would take approximately 18 seconds, and at 1 Volt it would take around 12 minutes

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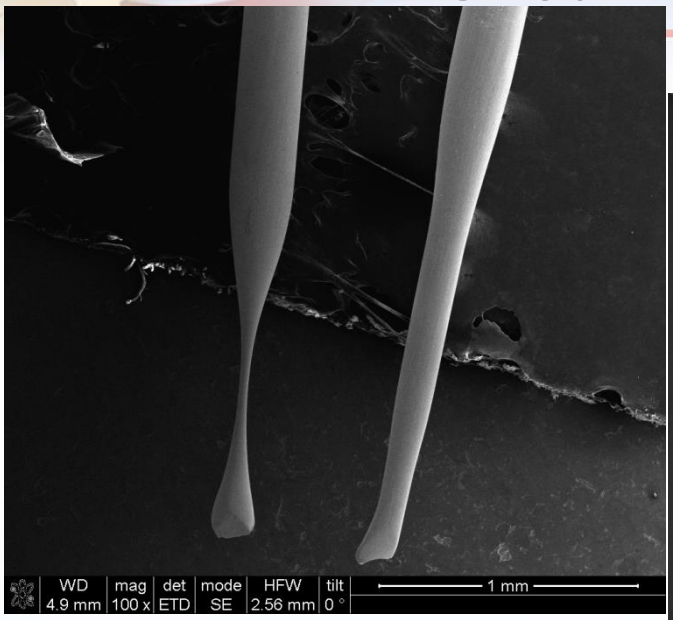
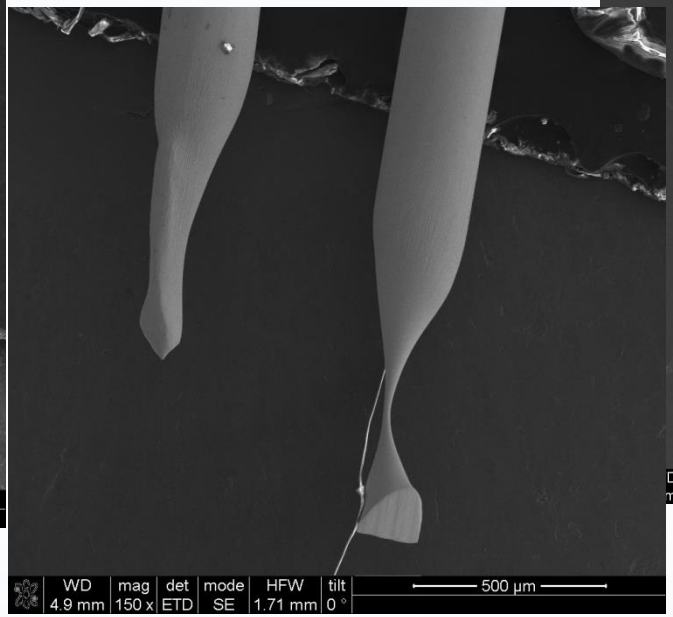
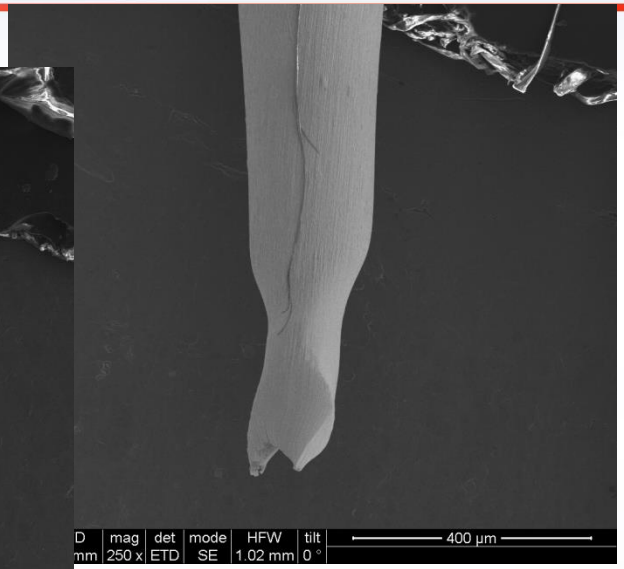
Finished AC Tungsten Probes







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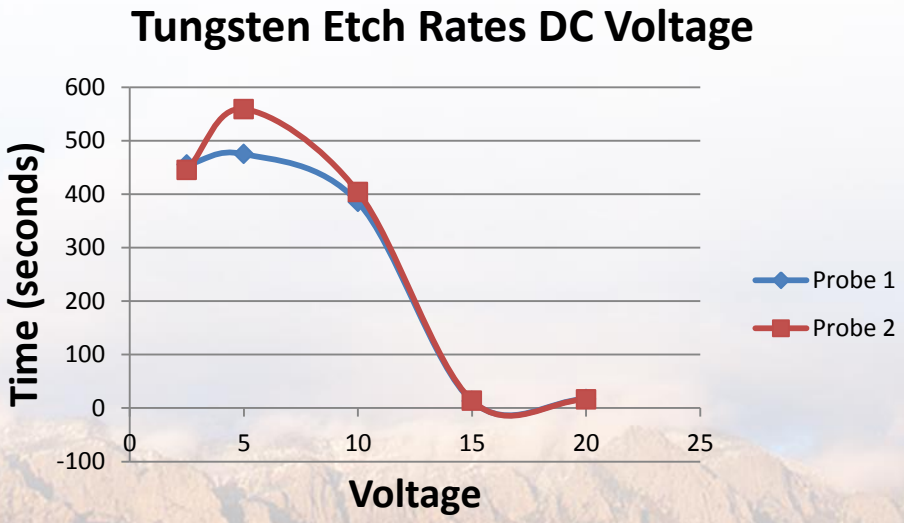
Tungsten Data DC

Voltage (Volts)	Time for First Probe	Time for Second Probe
20	17 sec	16 sec
15	13 sec	14 sec
10	6 min 25 sec	6 min 44 sec
5	7 min 55 sec	9 min 19 sec
2.5	7 min 35 sec	7 min 25 sec

The results from the DC voltage test were greatly varied, ranging from seconds to several minutes, however overall the DC voltage took longer on average than the AC voltage tests

There may have been a few outliers, it was often hard to know when the tip would drop since times were so varied during the experiment



Finished DC Tungsten Probes

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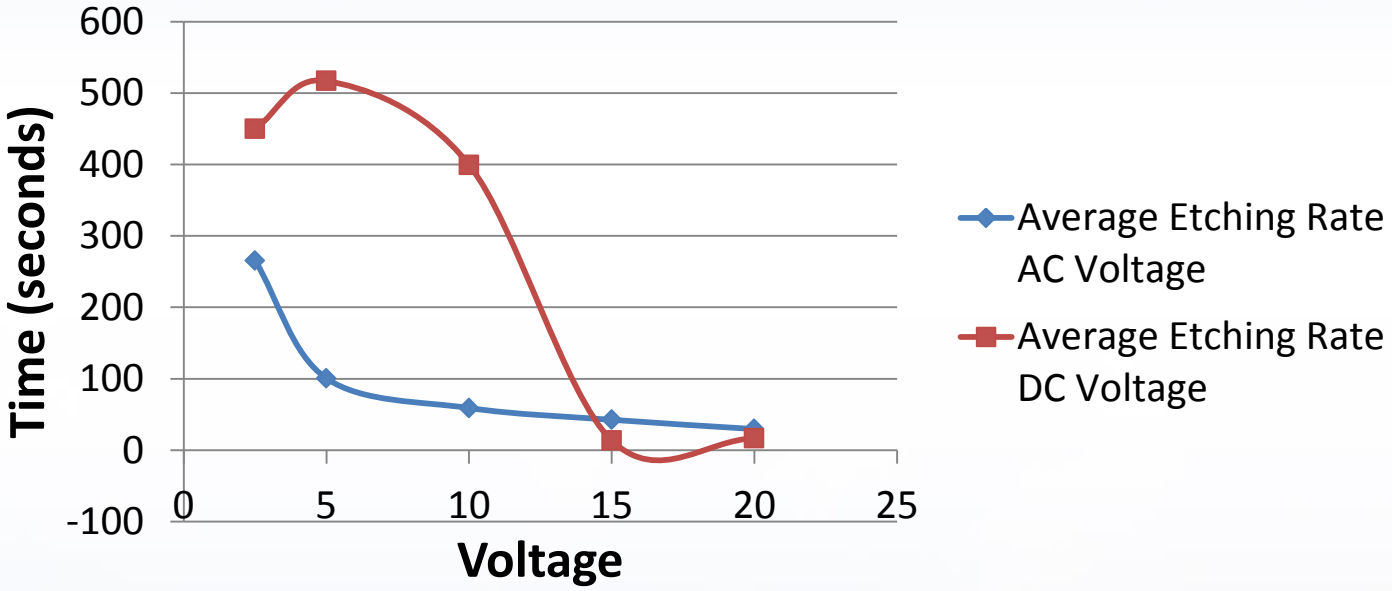
Comparison of Etch Rates for AC and DC Voltage



AC Voltage

DC Voltage

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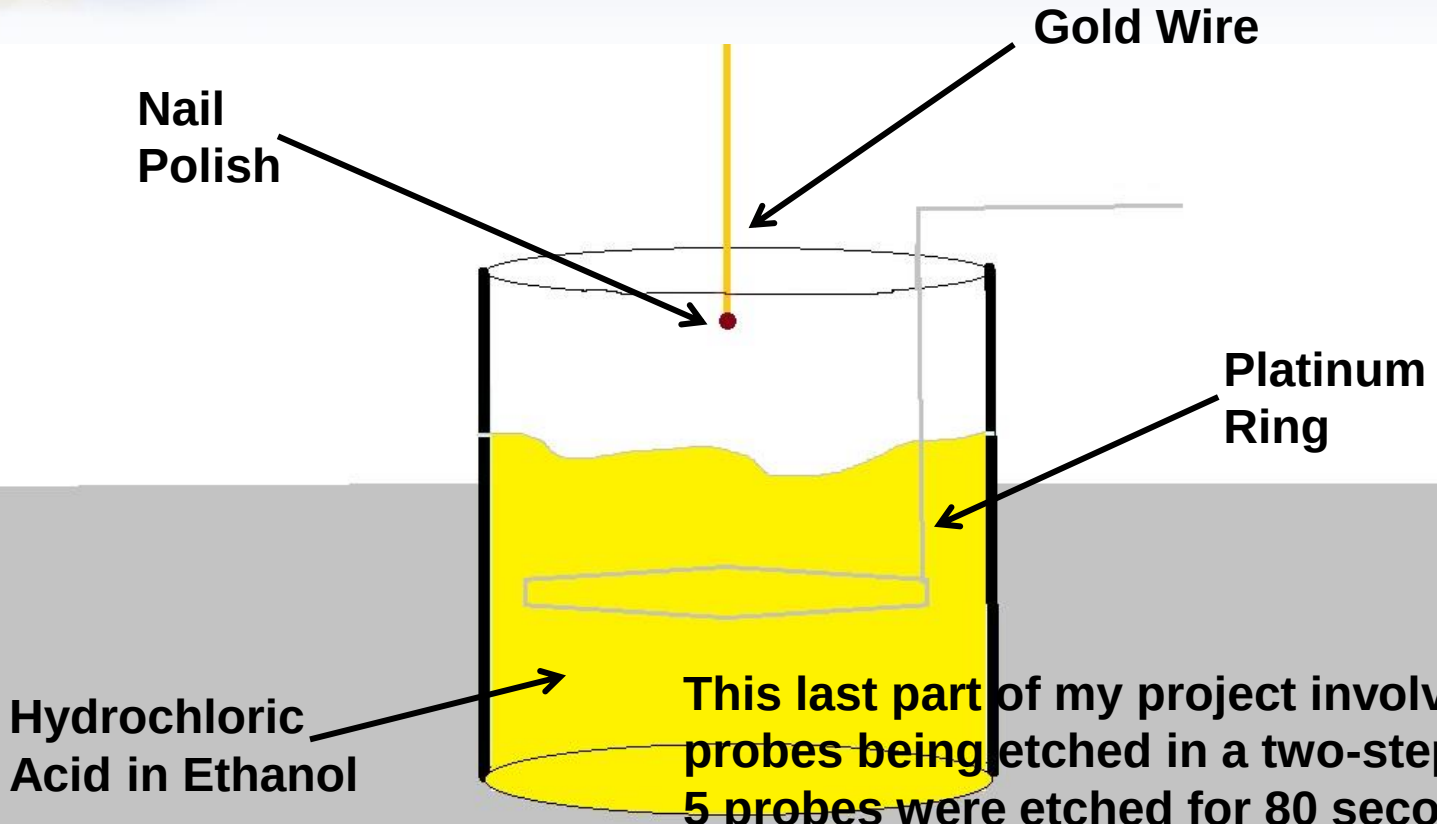
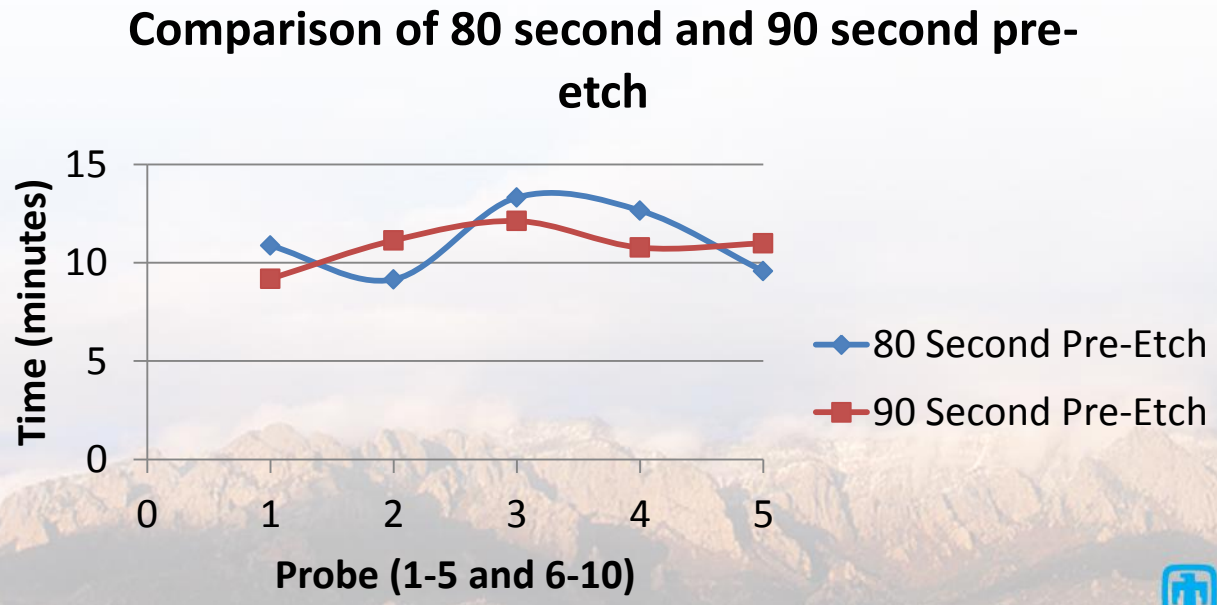
Electrochemical Etching of Gold Probes

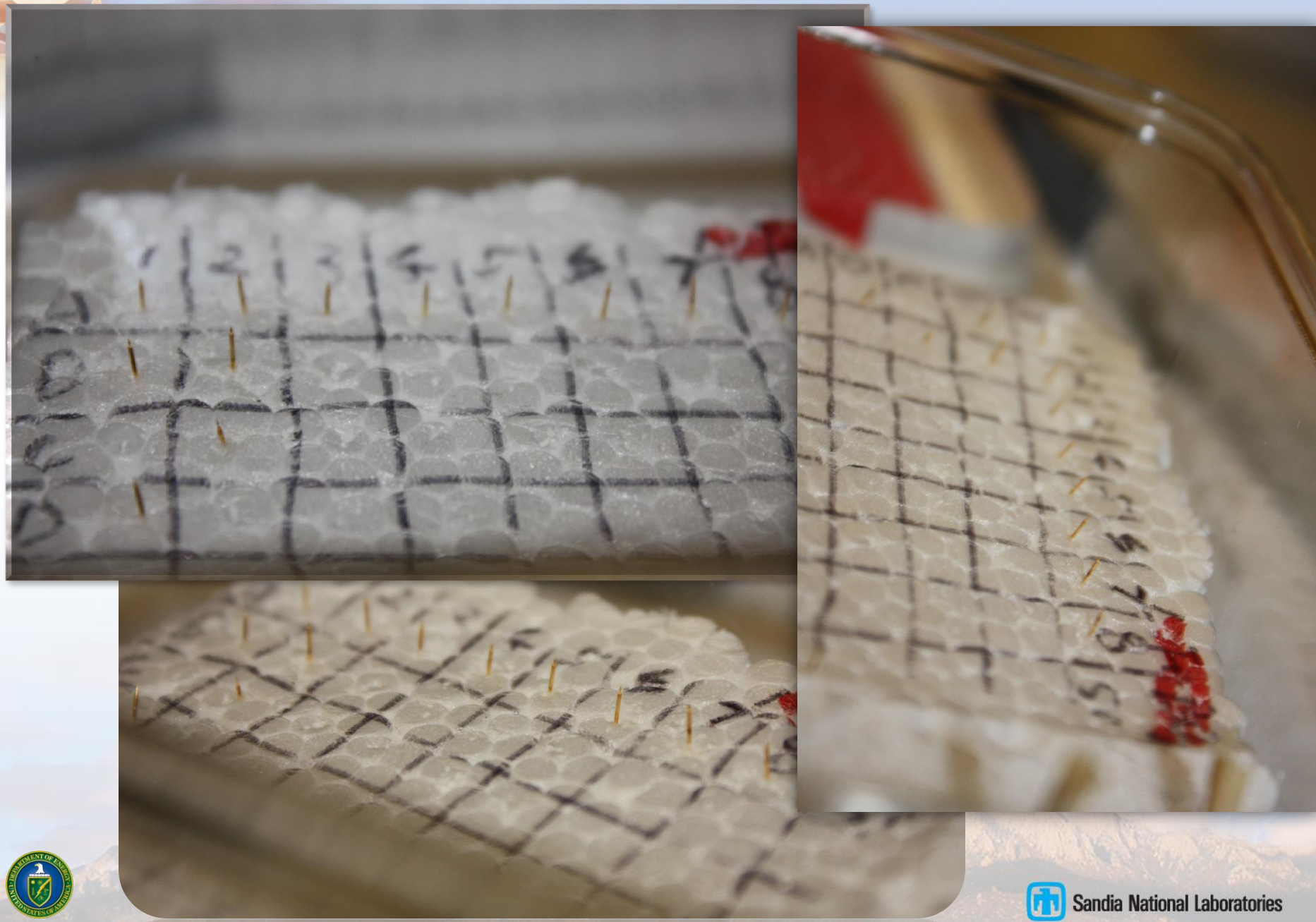


This last part of my project involved Gold probes being etched in a two-step process. 5 probes were etched for 80 seconds at 10 volts then 2.25 volts until the tip fell off and 5 were etched for 90 seconds at 10 volts and then reduced to 2.25 volts until the tip dropped off

Gold Data

Probes Pre-Etched for 80 seconds on 10 V	Probes Pre-Etched for 90 seconds on 10 V
10 min 52 sec	9 min 11 sec
9 min 9 sec	11 min 8 sec
13 min 19 sec	12 min 7 sec
12 min 39 sec	10 min 46 sec
9 min 34 sec	10 min 59 sec





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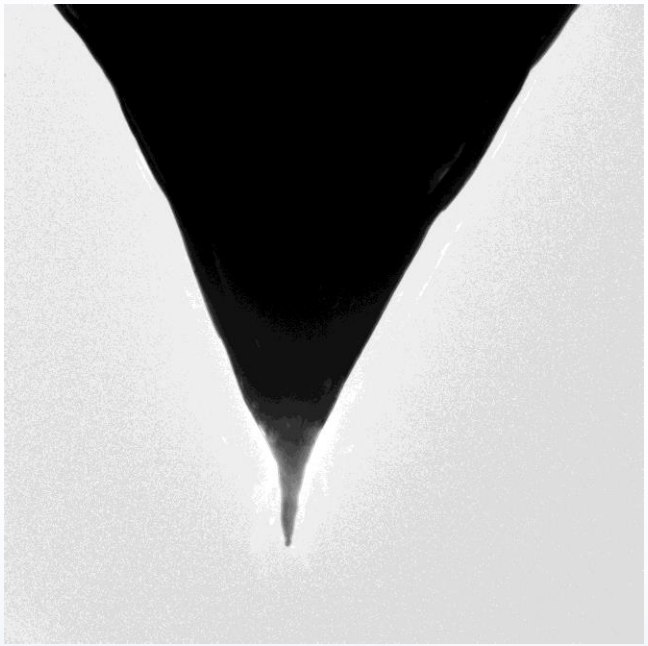
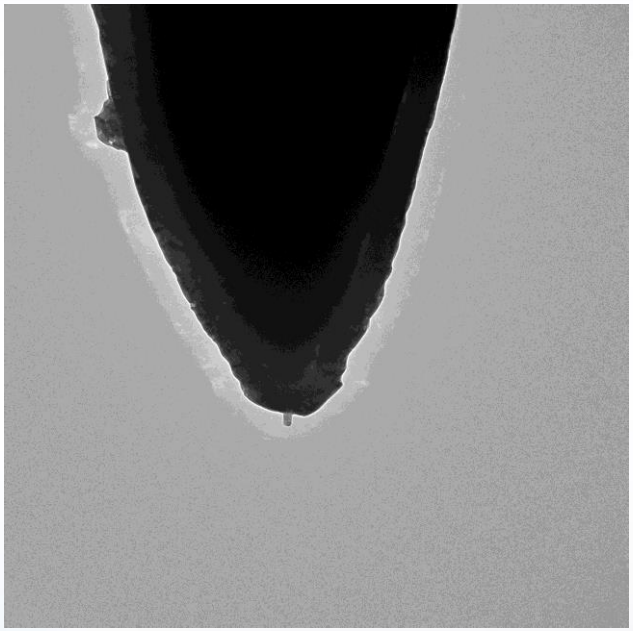
Finished Gold Probes





Gold Probes Etched Using the Same Conditions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