

THE NUCLEUS

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Monthly Meeting

Douglas S. Johnson of Pfizer speaks at Amgen

Summer Scholar Report

Pt-Ir Tip Creation for Use with Thiols and Thioethers on Gold Surfaces

By Sean Dwyer of Stonehill College

Karen Piper

Some NESACS History by the 2012 Arno Heyn Book Prize awardee

Fostering Innovation Workshop

By Katherine Lee

Summer Scholar Report

Pt-Ir Tip Creation for Use with Thiols and Thioethers on Gold Surfaces

By Sean Dwyer, Department of Chemistry, Stonehill College, Easton, MA

Abstract:

Pt-Ir chemical etching techniques using CaCl_2 solutions were studied for creating properly shaped, sharp tips for STM use to study self-assembled monolayers (SAMs) of thioethers and thiols on gold surfaces. Initially, the goal of this research was to study thioethers on gold surfaces using physical tip etching procedures. Physical procedures can be used to create tips for use in STM; however, these procedures have proven to not be as reliable. Therefore, in order to achieve good resolution of images of thiols and thioethers on gold surfaces, chemical etching techniques were adapted. A segment of Pt-Ir wire was held in a solution of CaCl_2 and a voltage was applied, causing the wire to taper, forming a tip. Rough etching and fine etching at varying concentrations, wire length, and voltage caused different reaction rates, and the rate of reaction determined the usefulness of the tips. A rate too fast, and the tip would be too dull, but a rate too slow would cause the tip to bend and have poor shape. By studying the different parameters, the best conditions for tip etching were found.

Introduction:

Scanning Tunneling Microscopy is a key analytic technique in studying surfaces and self-assembled monolayers that grow on them. A SAM is formed when compounds in a solution absorb onto a surface in repeating ordered patterns. Both thiols and thioethers have been shown to form SAMs on gold surfaces, and they have many applications.¹ Thiol monolayers have had a variety of uses including electrochemical processes, adhesion, and modeling membranes.² Another monolayer of particular interest is one created by thioethers. It has been shown that thioethers, when anchored to a surface, can create a tiny motor, similar to flagella on bacteria.³ This means that SAMs of thioethers have great potential for use as a motor for nanoparticles. The SAM surfaces of dioctylsulfide, dodecylsulfide, and octadecanethiol were studied to see how they form on surfaces of gold. This was attempted at different conditions in order to see how these factors affect SAM packing.

An STM is able to generate atomic resolution even at ambient temperature, as long as a properly shaped, sharp tip is created. Figure 1 shows an STM image of a gold surface at ambient temperature. For a tip to be useful, it needs to have the smallest surface area possible at the tip, the best tips consisting of only a single atom protruding from the end of the wire. A tip also must be the proper shape. A tip must not have any bends or be too long or too short. If the tip is very long it can begin to vibrate in the STM, distorting an image, and if it is too short, higher parts of the wire can interact with the surface to decrease the quality of the image. To create consistently useful tips, chemical etching techniques were studied.⁴

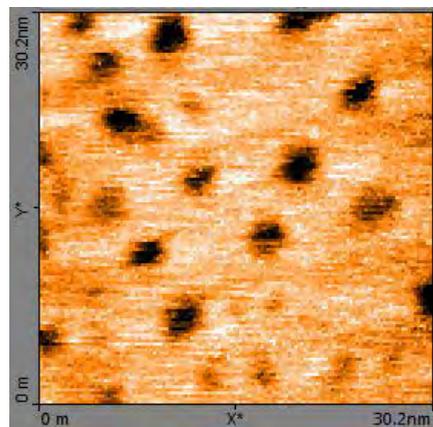


FIGURE 1: STM Image of Octadecanethiol on Gold surface

Chemical etching is a process that uses a voltage applied across a solution and a wire in order to cause that wire to taper and create a tip. There are two methods of chemical etching; rough etching and fine etching. In rough etching, a wire is typically etched in the middle, causing the wire to extrude and break, creating a sharp tip. A fine etching procedure usually begins with a wire that has already been rough etched. Then the etching focuses on the tip to make it sharper or better shaped. In order to see the differences in the different SAMs, effective chemical etching techniques of Pt-Ir wire must be found. Several different techniques were used that each affected the tip differently.

Procedure:

The SAM surfaces were created on an Au(111)/mica substrate provided by Agilent Technologies. They were annealed using a hydrogen flame for several seconds, and then viewed using the Nanosurf Easyscan2 STM system to confirm that the annealing was successful. The annealed gold surfaces were placed in sealed beakers of solutions of the dioctanesulfide or dodecylsulfide, and left to sit overnight. When taken out of the solution, the gold surface was dried by gently blowing nitrogen over it. It was then placed in the STM to be viewed.

The Pt-Ir tip was created in a two-step process of rough then fine etching. Figure 2 shows the arrangement for the procedure. In the rough etching process, between 1 to 5 mm of wire would be placed into a solution of CaCl_2 . The CaCl_2 concentrations used were 0.5, 1.5 and 3.0 M. A voltage was applied across the wire at voltages ranging from 10 to 50 V. Figure 3 shows the placement of the wire into the solution at the start of the reaction. The end point of the process, the moment when the wire was cleaved, was signified by a sudden silence and stopping of bubbles.⁴

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Cover: Pictured at the Malta Conference: *Frontiers of Science: Research and Education in the Middle East held November 10-15, 2013* are (L-R) Marinda Wu (ACS President), Rob Luke (British High Commissioner to Malta), Zafra Lerman (President, Malta Conferences Foundation), George Abela (President, The Republic of Malta) and Gina Abercrombie-Winstanley (U.S. Ambassador to Malta). Photo by Catherine Costello. For a complete article about the conference and more photos see the January Nucleus and the NESACS Website Photo Gallery.

Editorial Deadlines: April 2014 Issue: February 15, 2014

May 2014 Issue: March 15, 2014

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Karen Piper

Some NESACS History by the 2012 Arno Heyn Book Prize awardee

By Karen Piper

I began working for the Section in 1987 when Janice Fineman had resigned as administrative secretary and David Howell had taken over those duties while the Section looked for a replacement. The company for which I was office manager had been sold two years earlier, and I was in the process of starting a bookkeeping/payroll services business. The Section hired me as the administrative secretary, and I recall going to Northeastern to pick up the Section's files from Dave. Dave was a pillar of the Section's Board, having been Editor of the Nucleus, Section Treasurer, and Secretary. He was, however, not notable for being organized, and I recall retrieving files from an office in which one's physical welfare seemed threatened by the piles of paper inside. Fortunately, Janice had been an extremely organized secretary, and the files were in good order.

I had the good fortune to be the

administrative secretary during the chairmanships of Lloyd Taylor, Tom Gilbert, Mike Strem, Joe Billo, Chuck Kolb, and Katie Stygall. At that time, the annual reports to National were all on paper, and during January, my living room was covered with piles of paper for the seven copies that needed to be made. It was gratifying to receive the ACS Large Section Award in 1990 for the chairmanship of Mike Strem. Recalling my work as administrative secretary brings to mind many stories, but the best memories are of the people I was privileged to work with. Phyllis Brauner was my mentor. She had large ideas and usually was able to get them to happen. Ed Atkinson, Dick Handrick, Arno Heyn, Dave Howell, Ted Light, and a host of others, now departed, were people who cared deeply about the Section and were easy to work with.

Janice Fineman had worked closely with Richard Handrick, and she

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kept records for the Trustees for which Dick acted as Treasurer. I took over these records, and then, when Bill Adams at Salem State decided to give up the position of Business/Advertising manager of the Nucleus, those positions passed to Russ McCann and Vince Gale. I inherited the Business Manager's position from Russ McCann in 1991. Later, I took over the circulation manager's responsibilities from Dick Handrick. Dick kept the Nucleus mailing list on 3X5 cards and transferred them to IBM punchcards, which were processed by Wang Laboratories. I was able to put the list into a dBase III file and print the cheshire labels in my office.

I was also administrative secretary during the first years of the Esselen Award, as a result of which I developed a close relationship with members of the Esselen family. I had the privilege of working with Arno Heyn when he became editor of the Nucleus in 1989. When Arno assumed the chairmanship of the Esselen Award Committee in 2003, I had the opportunity to renew that relationship. His graciousness coupled with his attention to detail made that year very rewarding. Receiving the Arno Heyn book award in 2012 in recognition of our relationship was an unexpected pleasure.

In 1992 I left the position of administrative secretary and it eventu-

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Monthly Meeting

The 938th Meeting of the Northeastern Section of the American Chemical Society

Thursday, February 13, 2014

Amgen, Inc.

360 Binney Street, Cambridge, MA 02142
Building 1000 in One Kendall Square Complex

4:30 Board Meeting in Amgen Massachusetts Conference Center

5:30 Social Hour in Amgen Massachusetts Conference Center

6:30 Dinner in Amgen Massachusetts Conference Center

7:30 Welcome, **Dr. Catherine Costello**, NESACS Chair

Evening Lecture: *Application of Chemical Biology to Characterize the Selectivity of Covalent FAAH Inhibitors and the Mechanism of γ -Secretase Modulators*

Dr. Douglas S. Johnson, Associate Research Fellow, Worldwide Medicinal Chemistry, Pfizer Inc

THE PUBLIC IS INVITED

For those would like to join us for dinner, please make a reservation by noon, Thursday, February 6 using PayPal: <http://acssymposium.com/paypal.html>. Select the pay with credit or debit card option and follow the additional instructions on the page. Cost: Members, \$30; Non-members, \$35; Retirees, \$20; Students, \$10. New members or those seeking additional information, contact the NESACS administrative secretary, Anna Singer, at secretary@nesacs.org (preferred) or at (781) 272-1966, 9 AM - 6 PM. Dinner reservations not cancelled at least 24 hours in advance must be paid.

****For those joining us for dinner, reservations must be made no later than noon, Thursday, February 6.****

****If you wish, join us for the evening program only, starting at 7:30 PM. To facilitate check in at Amgen security, RSVP to the NESACS administrative secretary, Anna Singer, at secretary@nesacs.org by noon on Thursday, February 6.****

Directions:

Public Transportation:

Kendall Square is located along the MBTA's Red Line. Amgen-Massachusetts is a five-minute walk from the Kendall/MIT stop.

By car from the west:

Take I-90 (Massachusetts Turnpike) East to Exit 18, "Brighton/Cambridge." After the tollbooth stay right and follow the sign for "Cambridge/Somerville." At the first traffic light, follow the sign for "Cambridge." Continue over the bridge and stay in the right lane. At the end of the bridge (before the Mobil gas station) turn right onto Memorial Drive. The Charles River will be on your right. Continue east on Memorial Drive for about 2 miles. At the fourth traffic light, move to the center lane and continue under the Longfellow Bridge. Do not follow the sign for "Downtown Boston/Government Center. After the bridge, Memorial Drive turns into Edwin H. Land Blvd. Stay left and follow the signs for "Kendall Square. At the fifth traffic light, turn left onto Binney Street. Follow the signs for Cinema Parking to the One Kendall Square Garage.

Public Parking:

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Biography



Doug Johnson obtained his BS in chemistry from the University of Minnesota in 1991 where he did research in the laboratory of Prof. Thomas R. Hoye. Doug obtained his Ph.D. in organic chemistry in 1996 under the guidance of Prof. Dale L. Boger at the Scripps Research Institute in La Jolla, CA, and served as an NIH postdoctoral fellow in the laboratory of Professor David A. Evans at Harvard University.

He joined Pfizer in 1999 and is currently an Associate Research Fellow in the Neuroscience Medicinal Chemistry group, located in Cambridge, MA. He is a co-author on more than 60 publications and patents. He is a coauthor of the book *Contemporary Drug Synthesis* and is an editor of *The Art of Drug Synthesis* and *Modern Drug Synthesis*.

During his tenure at Pfizer, he has played significant roles on teams that have advanced 3 compounds into the clinic – PD 0332991, a CDK4/6 inhibitor in phase III clinical trials for breast cancer; PF-00217830, a D2 partial agonist, which advanced to phase II for schizophrenia; and PF-04457845, a FAAH inhibitor in clinical trials for the potential treatment of CNS disorders.

In addition, his group is interested in applying chemical biology methods to enable drug discovery projects. Most recently, his group has used clickable photoaffinity probes to characterize the targets and the mechanism of action of γ -secretase inhibitors (GSIs) and modulators (GSMs) relevant to Alzheimer's disease. \diamond

The Norris-Richards Undergraduate Summer Research Scholarships

The Northeastern Section of the American Chemical Society established the James Flack Norris and Theodore William Richards Undergraduate Summer Scholarships to honor the memories of Professors Norris and Richards by promoting research interactions between undergraduate students and faculty.

Research awards of \$3500 will be given for the Summer of 2014. The student stipend is \$3000 for a minimum commitment of ten weeks of full-time research work. The remaining \$500 of the award can be spent on supplies, travel, and other items relevant to the student project.

Institutions whose student/faculty team receives a Norris/Richards Undergraduate Summer Research Scholarship are expected to contribute toward the support of the faculty members and to waive any student fees for summer research. Academic credit

may be granted to the students at the discretion of the institutions.

Award winners are required to submit a report (5-7 double-spaced pages including figures, tables, and bibliography) of their summer projects to the NESACS Education Committee by October 24, 2014 for publication in *The Nucleus*. They are also required to participate in the Northeast Student Chemistry Research Conference (NSCRC) in April 2015.

Eligibility:

Applications will be accepted from student/faculty teams at colleges and universities within the Northeastern Section. The undergraduate student must be a chemistry, biochemistry, chemical engineering, or molecular biology major in good standing, and have completed at least two full years of college-level chemistry by Summer 2014.

Criteria for Selection:

- scientific merit - important factors include the originality of the project, the depth of the investigation, the significance of the scientific questions you pose, and the methods you propose to use.
- feasibility - evidence must be provided to demonstrate that the project can be completed by you in the time available and with the facilities at your disposal.
- preparation - your academic record, your ability to handle the project, and the background study you have made on your research problem will be taken into consideration.
- commitment - the depth of your commitment, and that of your department, faculty, and institution to independent research as a vital component of science education will be assessed.

Completed applications are to be submitted, no later than March 28, 2014, to the Chair of the Selection Committee:

Professor Jonathan Rochford
Department of Chemistry
University of Massachusetts Boston
100 Morrissey Boulevard
Boston, MA 02125-3393 ◇

Karen Piper

Continued from page 4

ally passed into the very capable hands of Marilou Cashman and, now, to Anna Singer. I have retained my connections with the Trustees, *The Nucleus*, and the Esselen Committee. The Business Manager also serves as the circulation manager for *The Nucleus*, works closely with the Advertising Manager, Vince Gale, and is responsible for accounting. After 25-plus years of association with the Section, it is satisfying and rewarding to see the continuation of dedicated people carrying on the traditions of the Northeastern Section. ◇

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National Recognition for NESACS Student Chapters

The ACS Committee on Education has selected the following student chapters in the Northeastern Section to receive special recognition for the programs and activities described in their 2012-2013 reports:

Outstanding

- Gordon College, Wenham, MA; Justin Andrews and Sarah McCarron, chapter co-presidents; Prof. Joel Boyd, faculty advisor.
- Northeastern University, Boston, MA; Christine Dunne and Elise Miner, chapter co-presidents; Prof. Kathleen Cameron, faculty advisor.

Commendable Recognition

- Simmons College, Boston, MA; Veronica Nowakowski and Kristin McDonough, chapter co-presidents; Prof. Changqing Chen, faculty advisor.
- Suffolk University, Boston, MA; Gianna Mancuso and Elsy Naveo, chapter co-presidents; Prof. Doris Lewis and Prof. Andrew Dutton, faculty advisors.

Honorable Mention

- Stonehill College, Easton, MA; Samantha Sweeney and Stephanie Murray, chapter co-presidents; Profs. Cheryl Schnitzer and Marilena Hall, faculty advisors.

Student involvement in applying green chemistry principles and practices is essential to the integration of environmentally benign technologies in academia and industry. The ACS Green Chemistry Institute recognizes ACS student chapters that have engaged in at least three green chemistry activities during the academic year. Listed below are the 2012-2013 Green Chemistry

Fostering Innovation

By Katherine Lee



Dr. David Harwell (front far left), *Fostering Innovation* course Facilitator, pictured with many of the thirty attendees
Photo by Katherine Lee

On December 2, Dr. David Harwell, Assistant Director, Career Management and Development at the American Chemical Society led an ACS Leadership Development course on “Fostering Innovation” at Pfizer Inc, in Cambridge, MA. Thirty members of NESACS attended the 4-hour course, which challenged the attendees to change the way they and the people they work with look at an issue and create new ideas, in order to:

- Tap into a systematic method for creating for new approaches
- Eliminate personal and organizational barriers that inhibit one’s ability to think more innovatively
- Have a proven process to generate ideas

Award recipients located within the Northeastern Section.

- Gordon College, Wenham, MA
- Northeastern University, Boston, MA
- Suffolk University, Boston, MA
- University of Massachusetts Boston, Boston, MA

All chapters receiving special recognition will be honored at the 247th ACS National Chemistry Meeting in Dallas, TX, on Sunday, March 16, 2014. ◇

Funding for this course was provided through a grant from the ACS secured for NESACS by Dr. Mukund Chorghade, Chair of Professional Relations, NESACS, while the meeting venue was provided by Dr. Mark Bunnage, VP of BioTherapeutics Chemistry, Worldwide Medicinal Chemistry, Pfizer Inc. The meeting was hosted by Dr. Katherine Lee, Pfizer Inc. We thank Kareem Redmond, ACS; Anna Singer, NESACS; and Jennifer Palmer, Pfizer for administrative support.

Did you know that the ACS Leadership Development System includes scheduled, facilitated courses such as the above course, which NESACS brought to the Boston area, and that Leadership Development courses are also offered at ACS National Meetings? In addition, self-paced online courses are available.

For more information, visit: http://www.acs.org/content/acs/en/careers/pr_ofdev/leadership.html ◇



Summer Scholar

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FIGURE 2: Setup for Etching Pt-Ir Tips

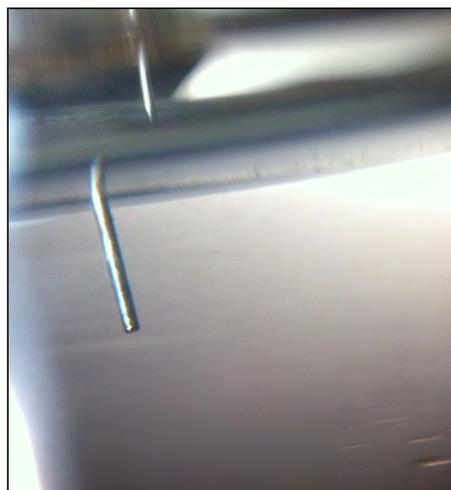


FIGURE 3:
Close Up of
Pt-Ir Etching

Several different fine etching techniques were tried, but only two had consistent success. The tip focused method was to only place the very tip of the wire into the solution, and etch the tip at voltages between 5 and 10 V.⁴ Using an optical microscope, the tip was placed barely touching the solution. The second method was to place 1 mm of the wire into the solution, apply a voltage between 5 and 10 V, and slowly pull the wire out of the solution. This was done between 1 and 10 times for any tip depending on its initial sharpness and shape. The tips were characterized using an optical microscope at 400 and 1000 times magnification.

Results:

The rough etching of tips appeared to be affected by several factors; the voltage applied, concentration of CaCl_2 solution, and length of wire inserted into the solution. It was found that medium voltages (25 - 30 V) produced sharp tips that

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FIGURE 4:
Tip Results from Different Etching Techniques

FIGURE 4A:
Rough Etched Tip
That Has a Bend
Due to a Slow
Reaction Rate
(400x)

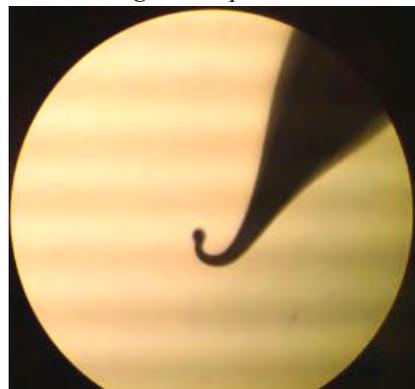


FIGURE 4B:
Rough Etched Tip
That is Too Long
Due to a Slow
Reaction Rate
(1000x)

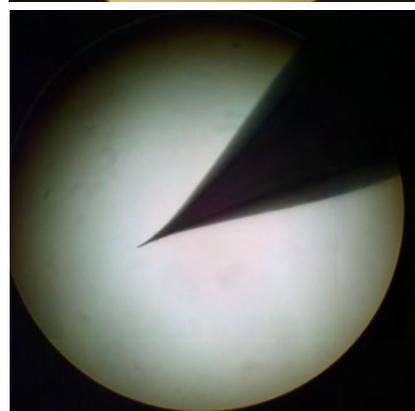


FIGURE 4C:
Rough Etched Tip
That is Too Short
Due to a Slow
Reaction Rate
(1000x)

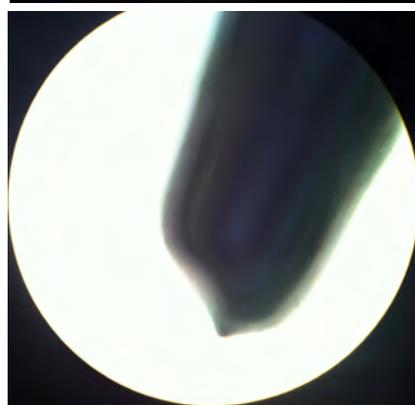
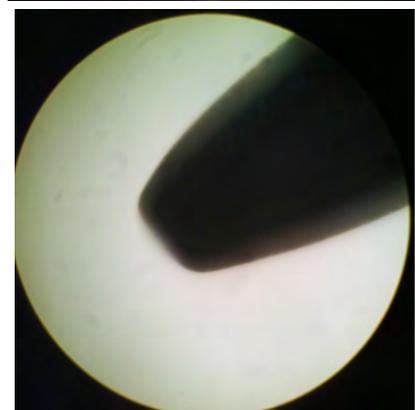


FIGURE 4D:
Rough Etched Tip
That is Dull Due to
a Fast Reaction
Rate (1000x)



Summer Scholar

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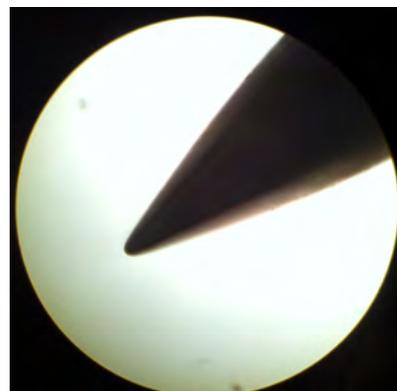
possessed good shape. Voltages higher than this, (40 – 60 V) react too quickly, and do not produce sharp tips. Voltages lower than this (10 – 20 V) react too slowly, and take several hours to complete, and also have a poor shape. Higher voltages increased the rate of etching. The poor shape caused by low voltages is created due to an unclean break during the rough etching process. High voltages caused dull tips because they would continue to react with the wire even after wire broke. Placing more of the wire (3 – 5 mm), into the solution caused the tips to be sharper, however, this often caused the tip to bend and have an unusable shape. This was due to the etching point having too large of a force onto it, causing it to break too soon. Placing less wire, (1 – 2 mm) created tips that were not as sharp, but were usually free from shape defects. Higher concentrated solutions caused the reaction to go faster. This caused the tips to be dull, and without any benefit in shape. Lower concentrations caused the reaction to go slower, and create sharper tips, but did not appear to cause any issues with the shape. It appears that for the rough etching, a medium voltage with a low concentration using a short amount of wire in the solution makes the best tips. This is because these conditions cause the tip to react at the proper rate. At a higher reaction rate, the tip does not become sharp enough, and at a lower rate, bends and curves in the tip become too common. However, in all cases of rough etching, a fine etching procedure was used to either make the tip sharper, or have a better shape. Examples of tips are shown in Figure 4.

Two major fine etching procedures were studied, and they each had different uses.⁴ The tip focused etching process made tips become sharper. However, this process did not improve tip shape significantly. Lower voltages were better for this method, because if the voltage was too high, the tip would begin to dull. The pull-down method was very effective at removing bends from tips, as well as improving length. This method did not improve tip sharpness, and sometimes made tips duller. Depending on the problem with the shape, different voltages were needed. For tips that were bent about 10 V worked best. If the voltage was too high, the bend would just be broken leaving a dull tip. However, if the voltage was too low, the bend would not be corrected. For fixing tips that were too long, a higher voltage is needed, as breaking part of the end of the tip is desirable. For short tips, higher voltage is desirable as well, because it etches the wire more creating a longer tip. Both the pull down and tip focused methods are extremely useful when used together. The pull-down method fixes the shape of the tip, and then the tip focused method can be used to sharpen the tip further. An example of a sharp tip is shown in figure 5. This combination can make almost any tip from the fine etching process into a usable sharp tip.

Conclusion:

Consistently good tips can be created from Pt-Ir wire using these methods. The ideal conditions for rough etching a

FIGURE 5:
*Fine Etched Tip
After Both Methods
(1000x)*



wire are to have 2 mm go into the solution of 0.5 M CaCl₂ and apply 25 V. The wire should turn out to be both rather sharp as well as have a good shape. With this wire, if shape needs to be improved, the pull-down method will work. Then after a good shape is acquired, the tip focused fine etching can be used to sharpen the tip further creating a tip that is suitable for use in the STM to view thiols and thioethers on gold surfaces.

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2. Colin D. Bain, Hans A. Biebuyck, and George M. Whitesides, Comparison of Self-assembled Monolayers on Gold: Coadsorption of Thiols and Disulfides, *Langmuir*, **5** (723), 1989
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Monthly Meeting

Continued from page 5

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Calendar

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Note also the **Chemistry Department** web pages for travel directions and updates.

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<http://www.umassd.edu/cas/chemistry/>

<http://www.uml.edu/Sciences/chemistry/Seminars-and-Colloquia.aspx>

<http://www.unh.edu/chemistry/events>

Feb 3

Prof. Theodor Agapie (Cal Tech)

Boston College, Merkert 130 4:00 pm

Feb 6

Buchi Lectures in Organic Chemistry

Prof. Phil Baran (Scripps)

MIT, Rm 6-120 4:00 pm

Feb 7

Buchi Lectures in Organic Chemistry

Prof. Phil Baran (Scripps)

MIT, Rm 6-120, 4:00 pm

Feb 10

Prof. Danica Fujimori, (UC San Francisco)

Harvard, Pfizer Lecture Hall 4:15pm

Prof. Sunny Zhou (Northeastern U.)

MIT, Rm 4-370, 4:00 pm

Feb 11

Prof. Binghe Wang (Georgia State)

“Targeting Carbohydrate-based Biomarkers for the Development of Diagnostics”

Boston College, Merkert 130 4:00 pm

Prof. Michael Hagan (Brandeis Univ.)

MIT Rm 6-120, 4:15pm

Feb 12

Harvard/MIT Inorganic Chemistry Seminar

Prof. Peter Stang (Univ. of Utah)

MIT Rm 6-120, 4:15pm

Feb 13

Physical Chemistry Seminar

Prof. Michael Hagan (Brandeis Univ.)

Harvard, Pfizer Lecture Hall

4:15pm

Prof. Nicole Eyet (St. Anselm College)

U. New Hampshire, Rm N104 11:10 am

Feb 19

Harvard/MIT Inorganic Chemistry Seminar

Prof. Kenneth Karlin (Johns Hopkins Univ.)

Harvard, Pfizer Lecture Hall

4:15pm

Feb 24

Prof. Andrei Yudin (Univ. of Toronto)

Boston Univ., Metcalf Rm 113

4:00 p.m

Prof. Jason Sello (Brown U.)

MIT, Rm 4-370, 4:00 pm

Prof. Matthew Kanan (Stanford Univ.)

Harvard, Pfizer Lecture Hall

4:15pm

Feb 25

Prof. Wei Min (Columbia Univ.)

MIT, Rm 6-120, 4:00 pm

Feb 27

Prof. Geoff Coates (Cornell)

MIT, Rm 6-120, 4:00 pm

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contact -- Vivian Walworth
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Feb 28

Prof. Paul Ha-Yeon Cheong (Oregon State U.)

MIT, Rm 6-120, 4:00 pm

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