Production of Tungsten Nanoemitters for Ultrafast Electron Diffraction

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The Experiment

We research the arrangement of the atoms in a crystal during structural transformations. To observe the processes, we use ultra-fast electron diffraction to compile "images" of the atoms in transition. These images will help us to learn about how the macroscopic properties of materials arise from their atomic scale properties. These techniques could advance understanding of strongly correlated materials or lead to the development of novel electronic devices.

The Summer Project

The probing electron beam is created by focusing a laser onto a tungsten source to eject electrons. To achieve the resolution we need in our images, the electron beam must be as small as possible. Though the laser can be very finely focused, the electron beam diameter can be reduced even further by limiting the size of the tungsten source. Our etching station makes these nanoemitters: extremely sharp wires with tip diameters on the order of 10 to 100 nanometers.

Station Design

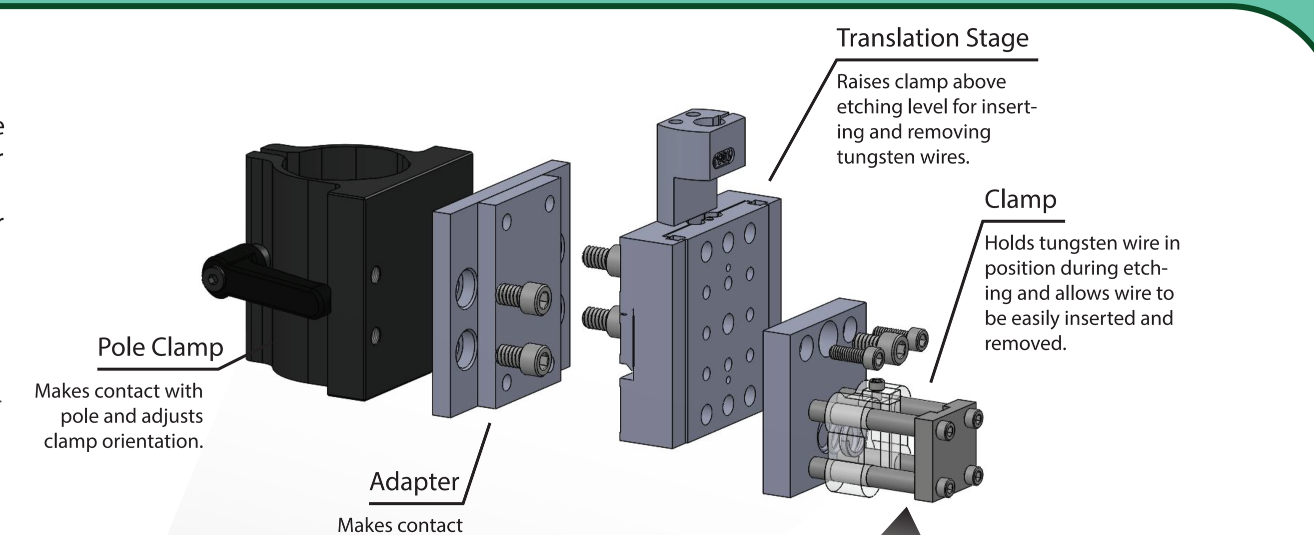
The etching station is designed to accomodate the tungsten etching process with freedom for later adjustments and decisions. The station can accomodate two configurations of wire for different applications.

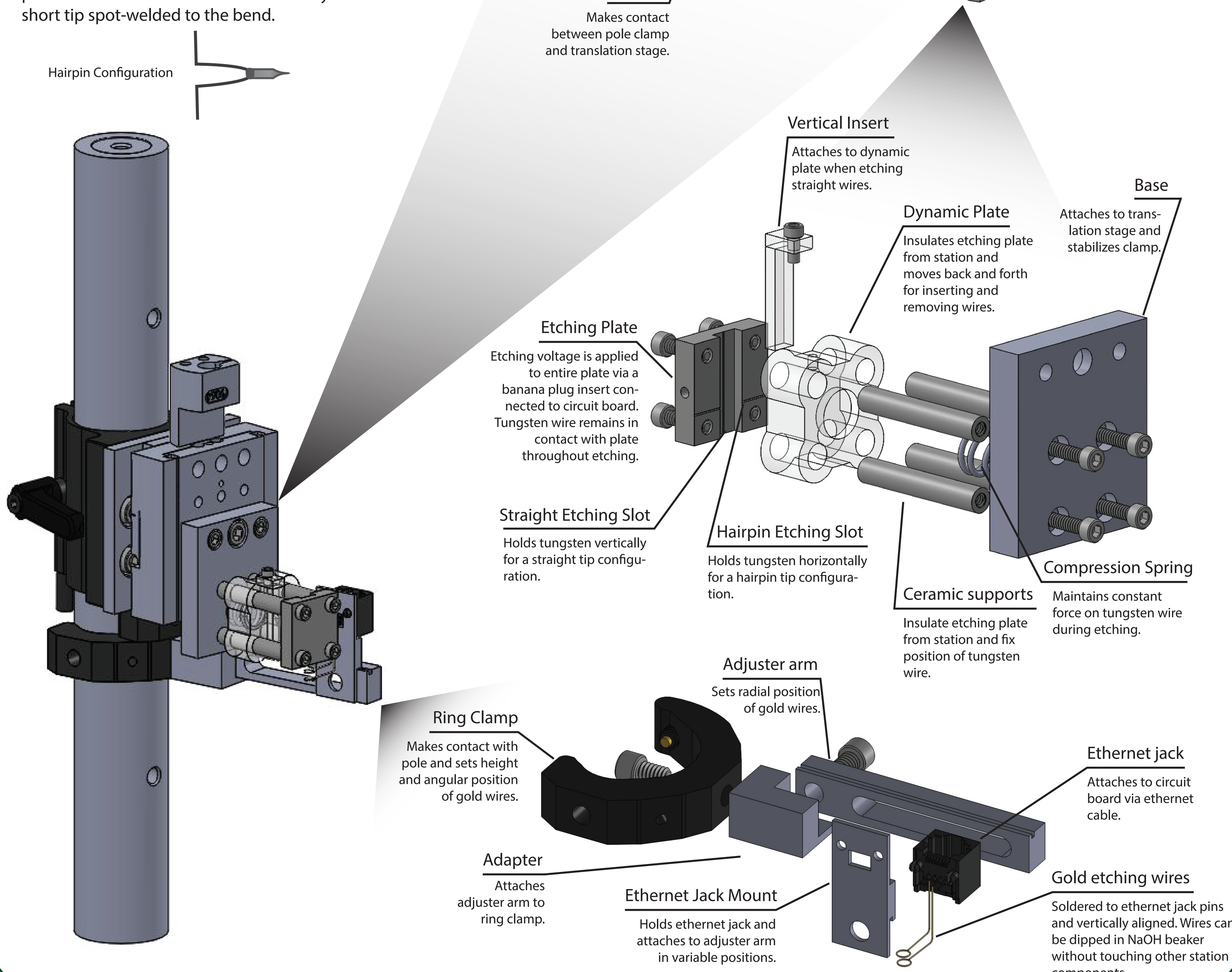
For testing the station and making modifications to produce consistent and high quality tips, a straight wire configuration is sufficient.

Straight Configuration

For experimental applications it is often more practical to mount a bent wire with a very short tip spot-welded to the bend.

Hairpin Configuration





Future Work

The design stage of the project is complete, but there are several more steps before the etching station can be used to produce tips for experimental use.

Station Machining

Many of the station components must be made in the machine shop before assembly. Custom aluminum parts will be made on the Bridgeport and CNC mills, and acrylic pieces will be made on the laser cutter.

Enclosure Machining

We will use a modified aluminum box to hold the circuit board and power source for the station. This box will have machined mounting holes for all of the external circuit components and direction connections to the station.

Circuit Board Assembly

The circuit components must be mapped onto a through-hole protoboard then soldered in place. This includes making attachments for all the components that will need to be accessed after the station is complete, such as adjustment potentiometers.

Station Testing

Once the station is fully assembled, its capabilities must be tested. We will create many etched tips under different adjustable parameters and measure their radii under an electron microscope.

References

D. Kunze, B.S, *Aufbau einer Ätzstation mit automatischer schneller Abschaltung zur Herstellung von Nanoemittern*, Friedrich-Alexander-Universität Erlangen-Nürnberg, July 11, 2016.

P. Hommelhoff, C. Kealhofer, A. Aghajani-Talesh, Y. Sortais, S. Foreman, M. Kasevich, Ultramicroscopy, volume 109, issue 5, pg. 423-429, 2009.

M. Kulawik, M. Nowicki, G. Thielsch, L. Cramer, H.P. Rust, and H.J. Freund, T. P. Pearl and P. S. Weiss, Review of Scientific Instruments, Vol 74, No. 2, 2003.

Schrodinger's Sharpener: User Manual, (Obbligato Objectives, Inc., Toronto, 2001).

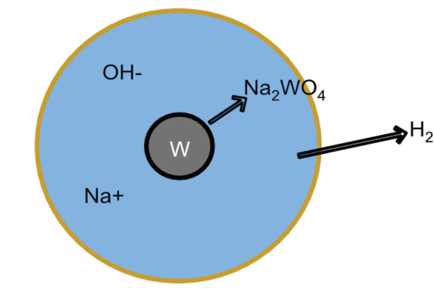
Production of Etched Tips

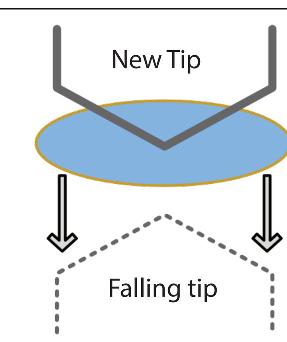
1. The circuit board brings terminal K1 to V_{etch} and supplies 10mA to the tungsten wire.

2. At the etching site, V_{etch} drives the following oxidation-reduction reaction:

$$W + 2H_2O + 2NaOH \rightarrow 3H_2 + Na_2WO_4$$

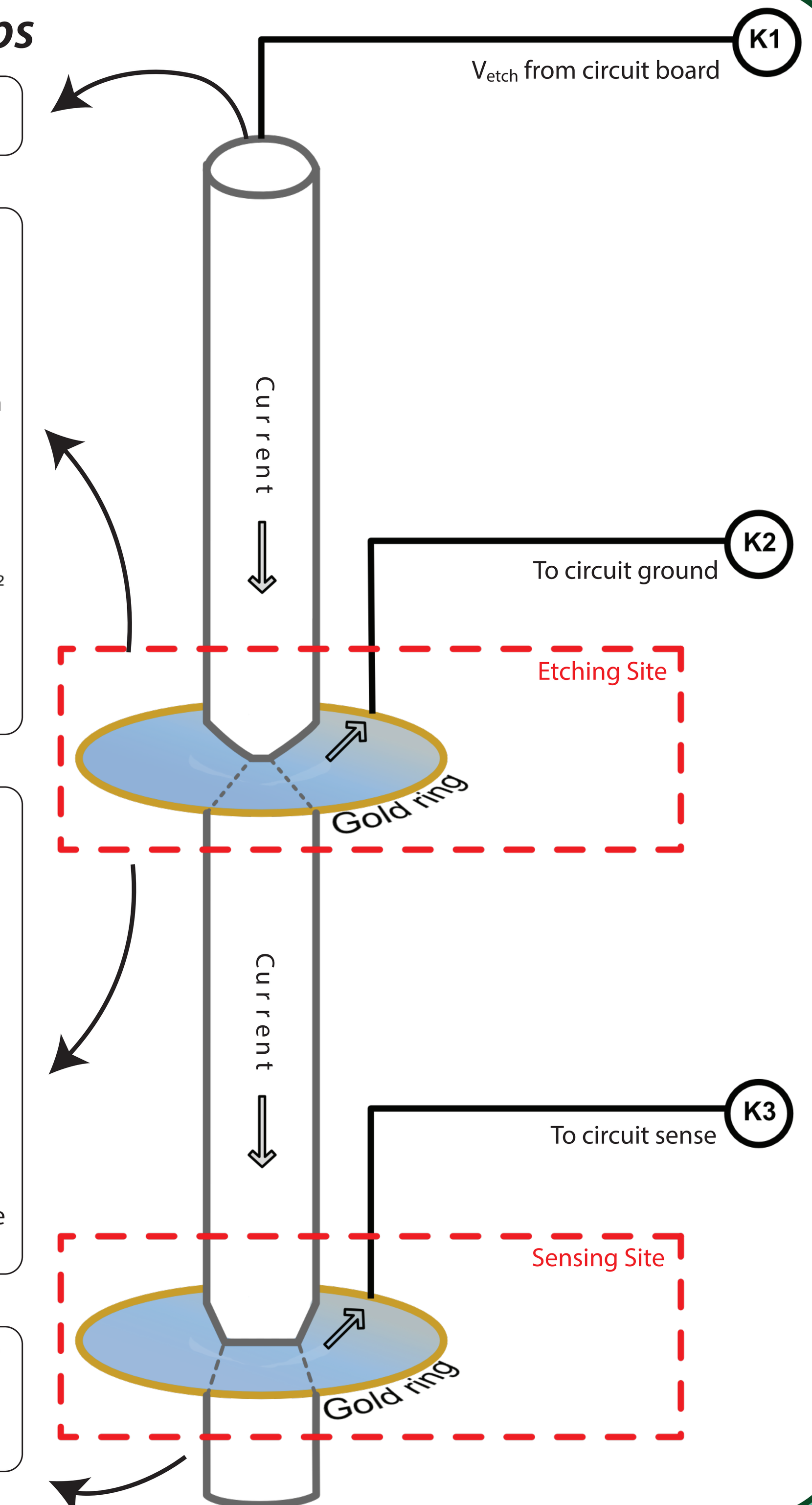
As the W oxidizes to form tungstate ions, the tips becomes sharper. In reality, the reaction has many intermediate steps, but this diagram conveys the essential results.



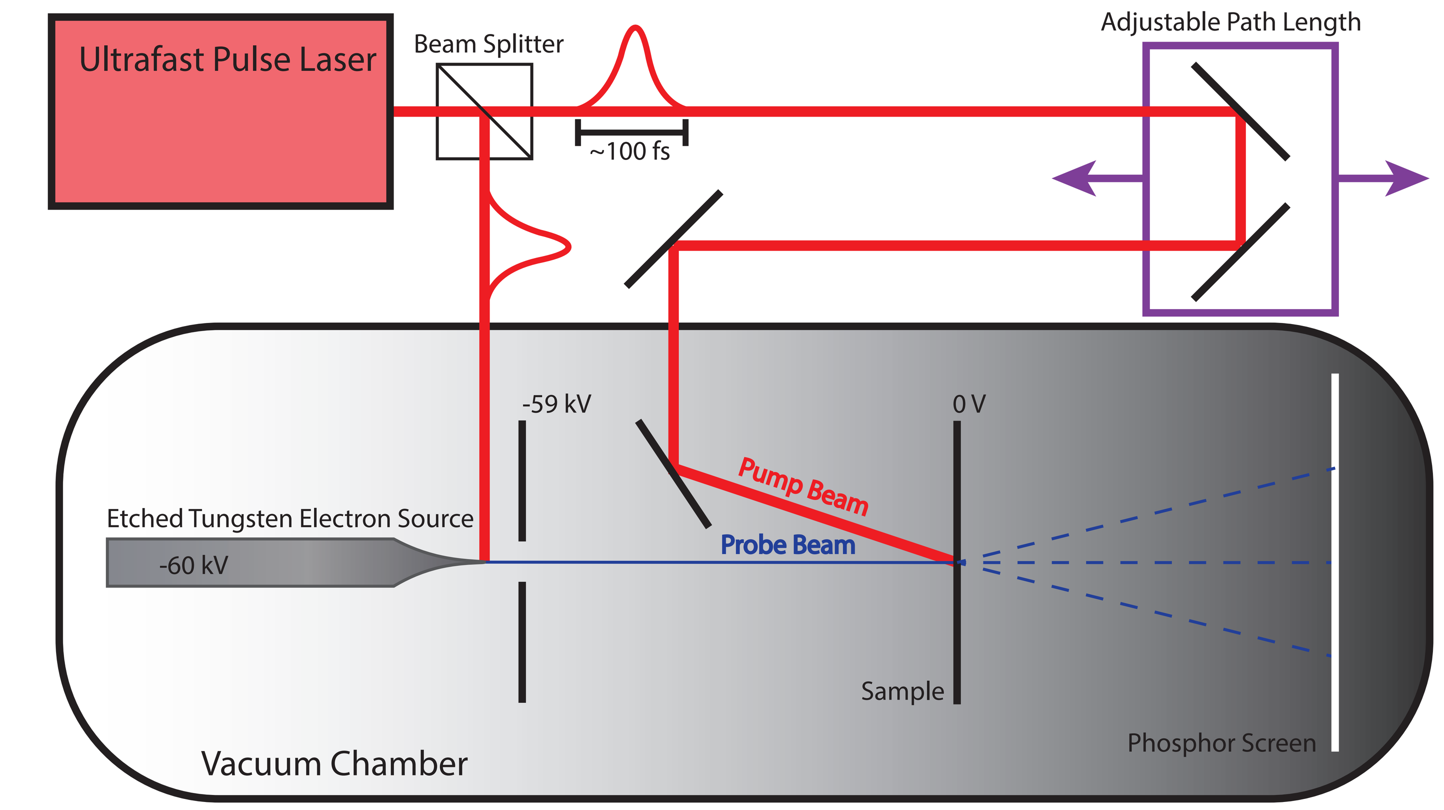


3. The tip is sharpest when the bottom of the wire falls away, but etching continues--dulling the tip-- because it remains in contact with the NaOH at potential V_{etch}

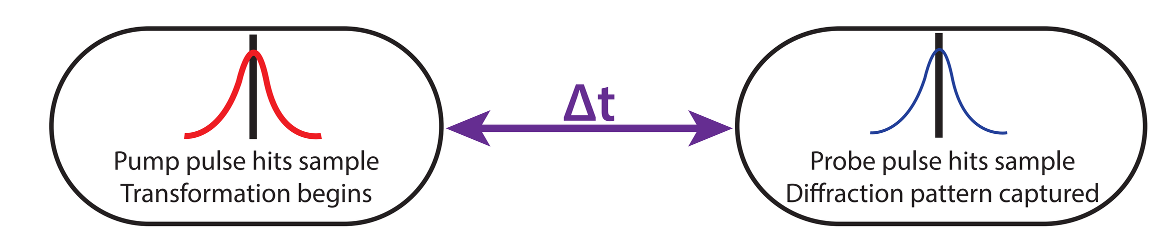
4. The circuitry senses when the bottom of the wire reaches 0V and switches off V_{etch} within 200ns to prevent dulling of the tip at the etching site.



Optical Setup

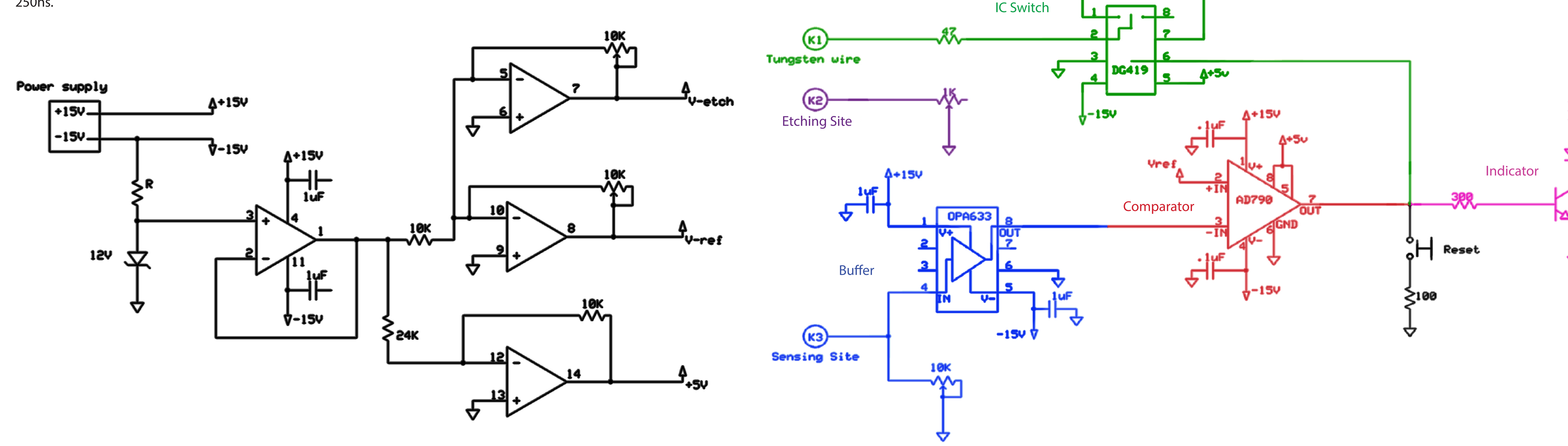


In order to study electron diffraction in rapidly changing crystal samples, we will use a "pump-probe" method. The ultrafast laser emits a light pulse on the order of 100 femtoseconds to "pump" the sample, beginning the transformation. The same pulse is directed by a beam splitter to the etched tip where electrons are excited then directed by a potential gain to "probe" the sample. The resulting diffraction pattern is recorded on a phosphor camera screen. The pumping laser pulse and probing electron pulse are separated by a Δt determined by the relative path lengths and the speed of the accelerated electrons. By adjusting the position of the mirrors that direct the pump beam we can slightly adjust this time and observe the electron diffraction at different points during the crystal's rapid structure transformation.



Circuitry Design

At the moment that the bottom of the tungsten wire drops off, we must immediately stop the etching process to prevent dulling of the tip. The circuit shown below is designed to sense this event and switch off the etching voltage in less than 250ns.



Circuitry Sections

	Buffer	Comparator	IC Switch	Etching Site	Indicator
Function	Isolates circuitry from the etching station. Independent control of current through K3 and current feed into circuitry.	Generate TTL logic signals for the IC switch input.	Turn the etching voltage on and off based on TTL logic input.	Choose the current through the etching site.	Light an LED when etching is completed
Integrated Circuitry	OPA633 — High speed buffer	AD790 — Comparator	DG419 — Analog Switch	Potentiometer	JFET - PNP transistor

Truth Table

	Input Current	Input Voltage	Output Voltage	Input Condition	Output Voltage	Input Condition	Output State	Etching current	Input Voltage	Result
Etching On	≈ V-etch ÷ R	Pin 4 = K3 ≈ Vetch	Pin 4 = K3 ≈ Vetch	Pin 2 > Pin 3, Vref > K3	TTL Low, < 0.8V	TTL Low, < 0.8V	Pins 8 and 2 connected, Vetch power supply connected	≈ V-etch ÷ R	TTL Low, < 0.8V	Transistor in cut-off, no current flow, LED off
Etching Complete	0	0	0	Pin 2 < Pin 3, Vref < K3	TTL High, > 2.8 V	TTL High, > 2.8 V	Pins 1 and 2 connected, open circuit, Vetch = 0	0	TTL High, >2.8 V	Transistor active, current permitted, LED on
						Reset button depressed, input voltage brought to ground	Pins 8 and 2 connected, Vetch power supply connected			
Transition times Etching On / Complete			5ns		45ns		150ns	0ns		N / A
									Total Time	200ns