Exploring the Nanoworld Using an STM

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PHY-312

I.) Introduction

Investigating the world beyond what the human eye can resolve has always been of great importance to science. Telescopes are utilized to explore phenomena at great distances while microscopes help researchers peer at the very small. Of the latter, optical microscopes are the most common type. However, by the very nature of light, microscopes of this sort can resolve objects only down to the micrometer range. In 1986, Gerd Binnig and Heinrich Rohrer won the Nobel Prize for the development of the scanning tunneling microscope, or STM for short. This new technology pushed the resolution possible down to the range of angstroms.

For our PHY-312 project, we sought to design and build our very own STM. Here, we present some background on quantum mechanical tunneling and STM's, discuss our design, and then examine the results of the project.

II.) Background and Theory

Before discussing the operation of a STM, it is prudent to first discuss the phenomenon of the quantum mechanical tunneling. To begin, imagine a free electron (that is, not in a potential field) traveling along with some fixed energy. Now, say this travelling electron was to encounter a sudden and large potential barrier.

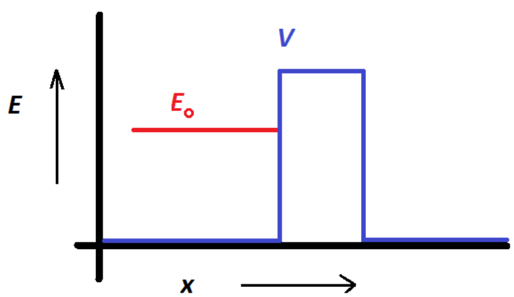


Figure 1: A traveling electron of energy E_0 encountering a potential barrier of height V.

Under the classical understanding of physics, the electron in this hypothetical scenario would be unable to pass through the potential barrier and would be reflected. However when quantum mechanics are taken into consideration, a very different picture is painted.

From Schrödinger's equation, we know that:

$$\frac{-\hbar}{2m}\frac{\partial^2}{\partial x^2}\Psi(x)+V(x)\Psi(x)=E_0\Psi(x)$$

Using our hypothetical potential for *V(x)*, the solutions are given by:

$\Psi_1(x)=Ae^{ikx}+Be^{-ikx}$, before the potential barrier

$\Psi_2(x)=Ce^{jx}+De^{-jk}$, in the forbidden region

$\Psi_3(x) = Fe^{ikx}$, after the potential barrier

where $k = \sqrt{\frac{2mE_0}{\hbar^2}}$ and $j = \sqrt{\frac{2m(V - E_0)}{\hbar^2}}$

What these equations mean is that the electron's wavefunction acts as a travelling wave before and after the barrier, but like an exponential under the potential barrier. The coefficient A is the amplitude of the incoming wavefunction, B is the amplitude of the reflected portion of the wavefunction, and F is the part of the wavefunction that is transmitted through the barrier.

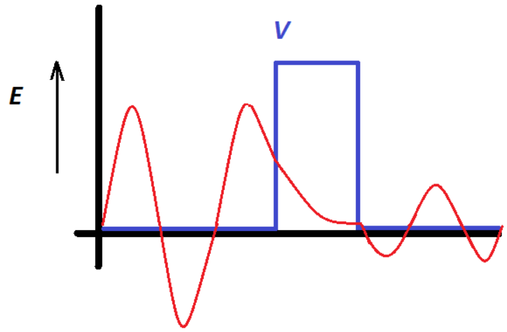


Figure 2: The electron interacting with the potential barrier.

The transmission of the wavefunction, *T*, (the amount of the wavefunction that makes it through the potential barrier) is given by the expression:

$$T = \frac{|F|^2}{|A|^2}$$

Physically, this means that for an electron approaching the potential barrier, there is a finite probability that the electron will be found on the other side. Instead of "climbing over" the potential barrier, the particle "tunnels" through. When the proper boundary conditions are applied, it can be shown that the transmittance varies exponentially with the width of the potential barrier. So a small increase in the width of the potential barrier leads to a large decrease in the transmittance through the barrier.

The STM makes use of the concept of quantum mechanical tunneling in a very inventive way. The sample to be scanned is placed on a surface that is held at a constant voltage. An atomically sharp tip is then lowered to be very close (closer than a micron) to the sample. The gap separating the sample from the tip serves as the potential barrier. From quantum mechanics, we know there is a finite probability that the electrons in the sample will tunnel through to the tip. This movement of electrons between the sample and tip creates a current. We also know that the probability of tunneling is related to the width of the barrier. So, this means that as the tip moves farther away the current will diminish and conversely that the current will increase as the tip gets closer to the sample. By examining the current, information about the distance between the tip and sample can be determined.

The STM operates by maintaining a constant tunneling current between the tip and the sample as the tip is scanned across the surface of the sample. This is achieved by using feedback. The tunneling current coming from the tip is amplified and then used as feedback to move the tip up or down. The feedback is recorded along with the x and y position of the tip. From this data, a topographic map of the sample can be created.

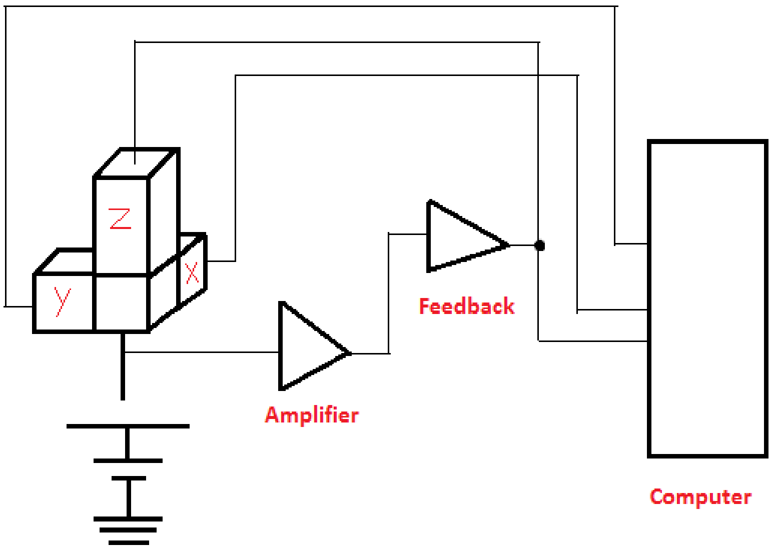


Figure 3: Block diagram of a STM

III.) STM Design and Construction

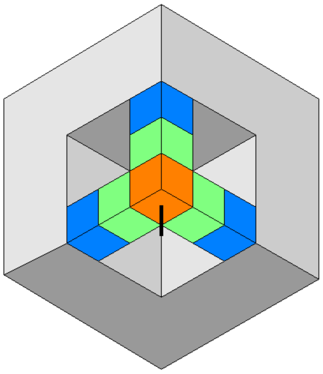
The construction of our STM consisted of three major parts: the physical body of the STM, the scanning tip, and the electronics. Due to the complexity of the project, each project member was assigned to direct a particular part of the project: Tom was in charge of the main STM construction; Nathan was in charge of constructing the STM tips; Alex was in charge of building the electronics.

III, a.) STM Body

The main body of the scanning tunneling microscope seemed like the simplest part of the construction yet probably the most frustrating. After much research on the body of the microscope, there seems to be many different body types but each design idea branches out from three basic designs and each had its own advantages and disadvantages. The three basic types would include the cylinder drum design, the tip on top design, and the tip on bottom design. (Klaus) The reason for so many different types of designs is because the main goal in these designs is to eliminate as much noise as possible. After learning about the multiple design types, the one that seems to work the best and is easier to build is building the tip at the top design. This design seemed to be the best because there was a lack of supplies to build the drum design. As for the tip at the bottom design, even though gravity would help to hold the tip in place, it is much more difficult to build. The cylinder design is basically a steel drum with the tip hanging from the top. The sample would be placed at the bottom of the steel drum and the piezos would move the tip. (Klaus) This type of design removes the use of the micrometer screws. The tip at the bottom design is when the tip is pointing up at the sample and the sample would move around where the tip adjustment would be controlled by the piezos.

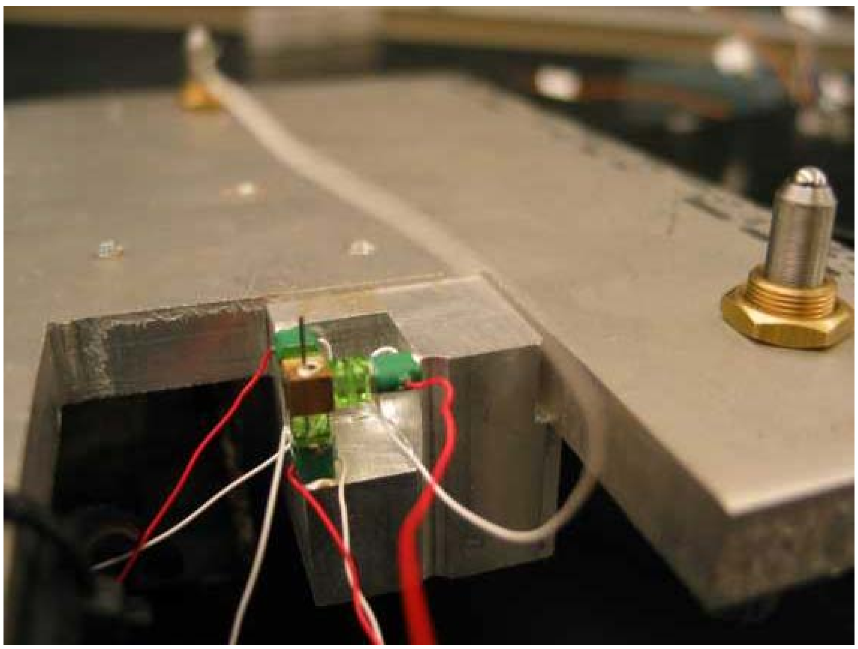
To build the body of the scanning tunneling microscope, we cut two equally sized pieces of metal which would act as the top and bottom of our microscope. To install the micrometer screws we drilled three holes into the piece of metal that would be acting as the top of the microscope. We placed the micrometer screws such that two holes were placed in the front in each corner an inch from each side of the metal and one in the middle back. After drilling these holes in the top of the microscope, the metal was then realigned with the bottom part and we drilled little divots which act as holders for the micrometer screws.

To build the arm, however, was much more difficult. The requirements that we needed to make the microscope work are the ability to conduct electricity and something that would not expand and contract with the piezo controllers. (Lars) In the end, we decided to go with a design such that the center point of this piece, which we call the tripod, is made of brass and the limbs of the tripod out of crystal beads. If we build the tripod piece like this, then we will meet both of the requirements that we needed to build the STM. We decided to us a brass bead which has a hole that can be used to put the holder for the tip. The brass bead was too big so we trimmed it down to a 4mm cube. We used crystal beads because they do not expand and contract as much as other things. We glued the crystal beads to the brass cube using very strong non-conductive glue. Then we glued the piezo controllers to the crystal beads using silver epoxy which is unnecessary.



STM scanning head schematic. Brass cube in orange; crystal beads in green; piezo controllers in blue; aluminum block in gray.

We also needed to make the arm so that it would stay stable on the top part of the microscope. To make the arm, we used a block of metal that is about 2” X 1” X 1” and then we cut a 13mm cube out of one of the corners to which we glued the tripod piece and piezo controllers to the empty space we cut out on the arm. We then used the silver epoxy to glue it to the arm. (It would have been better to use regular epoxy which is not conductive, but the silver epoxy worked just fine.) The glue used to put the brass and the crystal beads together would work just fine. Then we used a 24 guage stainless steel hypodermic tube to glue to the hole in the brass bead which will hold the tungsten tip.



The STM scanning head.

The plan is to put the etched tip into this tube and put a slight bend in the tip to keep gravity from pulling the tip out of the tube. We fastened the arm to the body with a single screw by creating threads in the top piece of metal. At the bottom portion of the microscope, We applied a layer of plastic followed by a layer of aluminum tape which we glued to the bottom using a glue stick. This is to prevent a current from going to the sample through the micrometer screws directly to the tip. This will also help force quantum tunneling to occur.

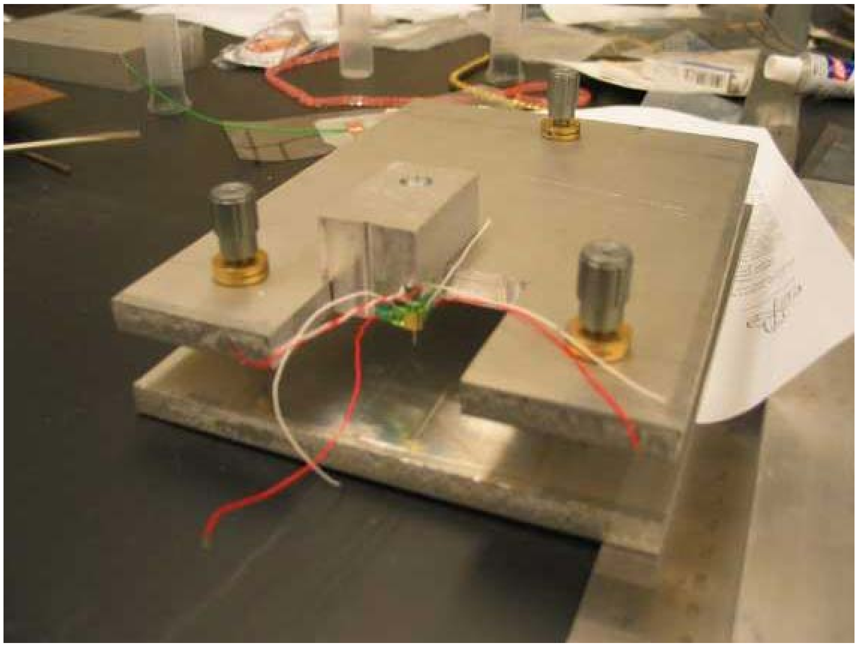


Figure 4: The STM body.

The piezoelectric devices move the scanning tip in three dimensions by expanding when a voltage is applied. The micrometer screws allow the scanning tip to be slowly and carefully lowered toward the sample.

III, b.) Scanning Tip Production

The most essential part of an STM is the tip. A bad tip will produce lousy images. Because of that the tip is usually the culprit of bad images. In a perfect tip, it would end in a single atom (Lucier), but that is practically impossible. Good tips are on the order of 10-50 nanometers in width.

There are many different ways to produce a tip. It is possible to produce one by just taking a knife and shaving off a wire, but that usually ends with a tip that is too wide and not nearly straight enough to produce good images. Another way is just by slowly pulling at both ends of the wire until it breaks, but that could also end up too wide as well as have a curved loop at the end from the recoil of the break. The general consensus among researchers is to electrochemically etch the wires (Lucier). Electrochemical etching is a very reproducible technique to produce reliable STM tips.

The substance used for our tips was tungsten (W). Tungsten is commonly used because it is quite simple to create extreme sharp tips by only doing a single etching process, and uses fairly mild chemicals. A drawback is that tungsten is vulnerable to oxidation (Lucier). The chemicals that are most commonly used are potassium hydroxide (KOH) or sodium hydroxide (NaOH). The chemical reaction that occurs is as follows:

$$\text{Cathode: } 6H_2O+6e^- \rightarrow 3H_2(g) +6OH^-$$

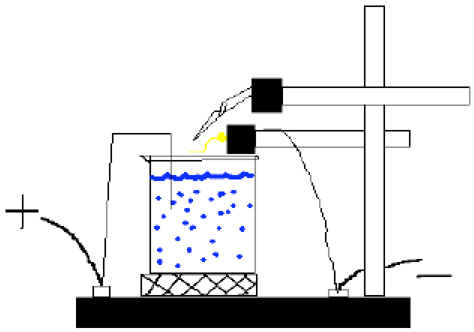
$$\text{Anode: } W(s) + 8OH^- \rightarrow WO^{2-}_4 + 4H_2O + 6e^-$$

$$\text{Total Reaction: } W(s) + 2OH^- +2H_2O \rightarrow WO^{2-}_4 +3H_2(g)$$

The anode is the tungsten wire, where as the cathode is the KOH or NaOH solution (Jones). The total reaction is the cathode and anode added together. The etching takes place at the meniscus of the contact between the tungsten wire and the KOH or NaOH solution. It occurs at the meniscus because of the greater energy at the curved surface than anywhere else and the greater energy causes the etching to begin (Teague). Once the etching begins it will continue at that same spot and not change unless the meniscus moves. As the reaction continues the wire near the meniscus will get thinner and thinner until the strength of the thin wire is overcome by the weight and the bottom portion of the wire falls off known as the “drop off” (Lucier). For the best results the power should be shut off immediately when the drop off occurs, since the chemical reaction does not stop when the drop off happens. If the reaction keeps going after drop off occurs duller tips will result.

Two different setups were tested to see which obtained better results. Both setups used roughly 2 Molar KOH. About 6.7 grams of solid KOH is added to 30 mL of deionized water and mixed until fully dissolved. Deionized water was then added until the total volume of the solution was 60 mL. Both setups also used about 11 V across the tungsten/KOH point of contact.

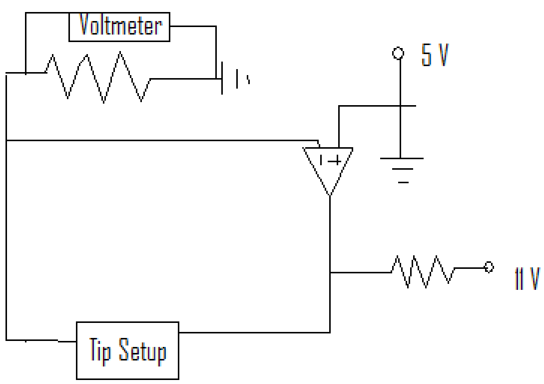
The first setup tried was submerging a gold-plated copper wire in the KOH with the tungsten wire above it in about the center of the loop. The output of the power supply was connected to the tungsten wire. The circuit was completely attaching a wire to the gold-plated wire through a resistor and then to ground (Lucier). To know that the reaction is occurring tiny bubbles must be forming on the gold-plated loop (the bubbles are the hydrogen gas). A volt-meter was connected to either side of the resistor because when the break off occurs the current drops drastically, so the current through the resistor also drops. According to Ohm's law, V=IR, since the resistor value stays the same and the current drops therefore the voltage would also drop making it possible to attempt to create an electronic shutoff. (The drop in current occurs in both setups.)



Side Veiw

The second setup is a little more complicated. First, a solution of 9 % concentrated saltwater was made by simply using 18 mL of salt and adding deionized water until the total volume was brought to 200 mL. A gold-plated copper wire loop is set a few millimeters above the saltwater and the tungsten wire is suspended through the gold-plated loop and making contact with the saltwater. The gold-plated wire is then connected to the same resistor used in the first setup and then to ground with the voltmeter attached to the same place. The saltwater solution is moved off to the side and a solution of KOH is put around the gold-plated loop and removed to create a bubble or film of KOH inside the loop. After the bubble is created the saltwater is replaced making sure that the saltwater solution never comes in contact with the loop. A copper wire is then inserted into the saltwater solution making sure that it does not touch the loop or tungsten wire. The wire is connected to the 11 V power supply causing the etching process to begin, as with the first setup there must be tiny bubbles forming on the loop. If the bubble pops before drop off occurs a new bubble needs to be placed to continue the etching process. Caution needs to be used to not bump the gold-plated loop or tungsten wire after the initial time it is placed above the saltwater solution (Jones).

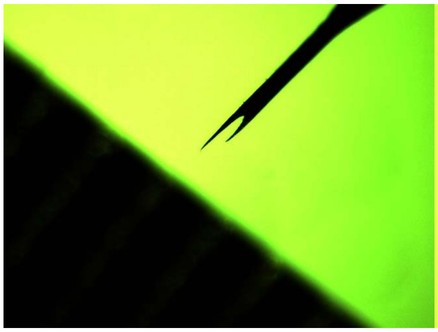
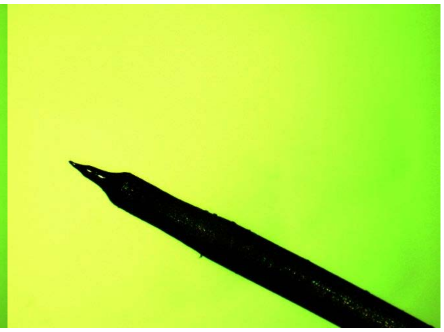
The drop off for both setup occurs at between .400 and 1 V reading on the voltmeter. The voltage drops off instantly to between 50 and 100 millivolts. An electronic shutoff was attempted to be made. The 11 volts went through a resistor and then to the tip setup, then through a resistor to ground. Between the tip setup and the resistor the circuit went to the negative input of a comparator op amp. Into the positive 5 volt that went through a pot so the voltage could be adjusted to slightly below the drop off voltage.



Shut off circuit

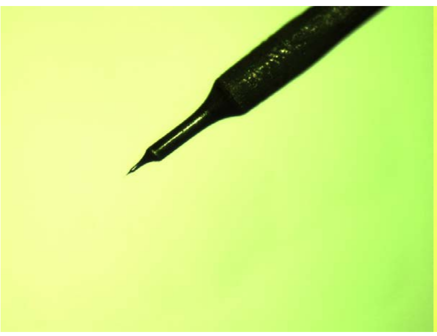
The second setup had much better results than the first. A big source of error in the first was that when bubbles came off of the loop and came to the surface some would then get stuck to the tungsten wire changing the meniscus. The meniscus is also changed because when the bubbles popped they sent a ripple through the KOH that also changed the placement of the meniscus. In changing the meniscus the resulting tips became multi-staged which produces poor images (Jones). A bifurcated tip can also result if the tungsten wire is cut from the length using wire snips. Even though tungsten is a hard metal using snips could result in the end splitting which would in turn cause the etched tip to be split. A split tip will also result in bad images so a hand held grinder should be used to cut the tungsten (Jones).

Images of both setups for making the tips (all images are at 100 times magnification):



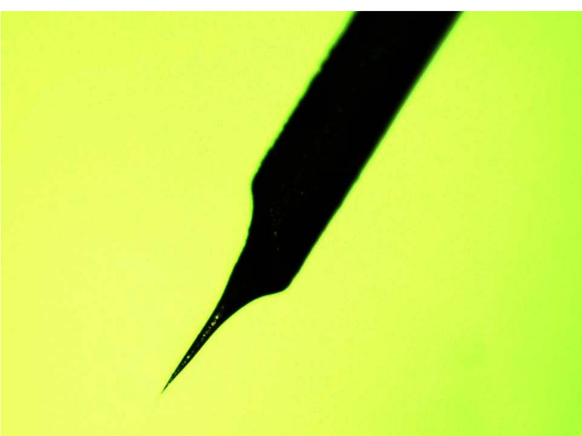
Left Image: Bifurcated tip caused by using the snips to cut the original wire.

Right Image: Tip made with the first setup



Left Image: Example of a multistage tip caused by shifting of the meniscus.

Right Image: The “drop off” of the best tip that was made.



Above: Best tip that was made.

III, c.) Electronics

The first electronic component designed and built was the pre-amplifier. The role of this part was to take the tiny tunneling current (on the nA scale) and amplify that into a readable voltage without too much noise, which would effectively destroy the small signal. The first attempt at this was a simple current-to-voltage converter using the AD823 op-amp. Unfortunately, there were several problems with this design. Since the input bias current for the AD823 is in the 100 pA range, it was necessary to include another resistor and use the transimpedance configuration for a proper ground.

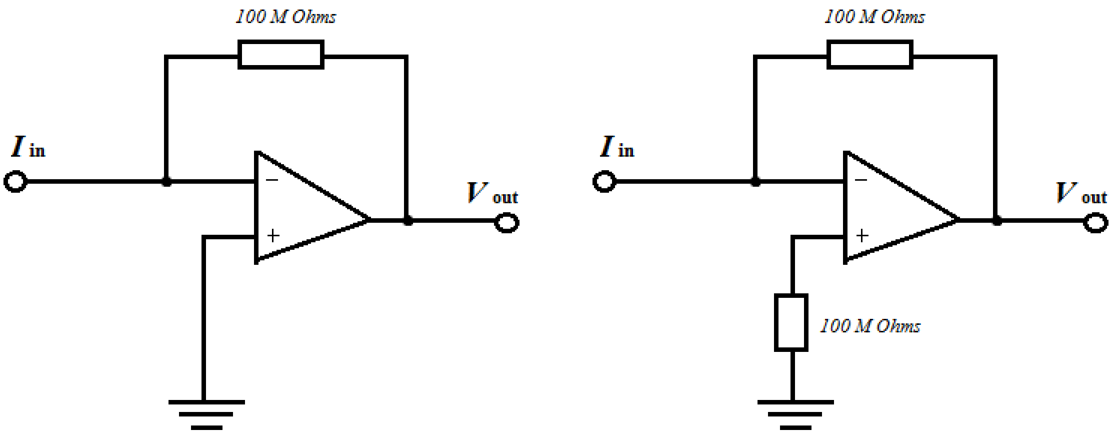


Figure 6: *Simple I-V converter pre-amp (left) and modified design (right). Offset circuitry not shown.*

Testing the pre-amplifier proved to be the most difficult part in the process. This difficulty arose from a combination of reasons. Most troubling was equipment malfunction. Originally, we intended to test the pre-amplifier with a variable current source and the Physics Department's Keithley electrometer. Sadly, the electrometer was not working properly and only provided order of magnitude measurements. To further complicate matters, it was extremely difficult to shield the pre-amp from noise while testing. At currents on this small of a scale, any movement around the circuit could change the capacitive coupling and alter the current greatly.

To circumvent these testing issues, a precise current source was built. The current source put out a constant current which was determined by a specific resistor in the circuit. This was then fed through an ammeter and finally to the pre-amp. The current input to the pre-amp and voltage output from the pre-amp could both be read and compared. This was done for several currents from the mA range down to the 0.1 μA range. From here, the current could no longer be reliably read by the ammeter. However, different resistors were used in the current source to create still smaller currents and the behavior of the pre-amp output continued to change as expected although the actual input current could not be read.

Once the pre-amp was fully tested and its behavior seemed satisfactory, the final circuit was built. Instead of the AD823 op-amp, the AD549 was used in the final circuit. This was because the AD549 offered the advantages of a lower input bias current (in the fA range), its casing could be grounded to eliminate some noise, and it had very long leads so that the input signal could be fed directly into the op-amp. An offset was added to the final circuit so that the pre-amp could be easily tuned. The entire final pre-amp assembly was shielded by a copper box and the input from the scanning tip was fed into the pre-amp by a shielded coaxial cable. All of these features allowed for the clean amplification of a signal with very little noise getting involved.

Once the pre-amp had been completed, the other circuitry could be dealt with. The next components to assemble were the supplies for the x- and y- piezos. These devices take a voltage from the computer and supply a smooth, symmetric power supply to the piezoelectric devices. This was accomplished by a series of inverting amplifiers.

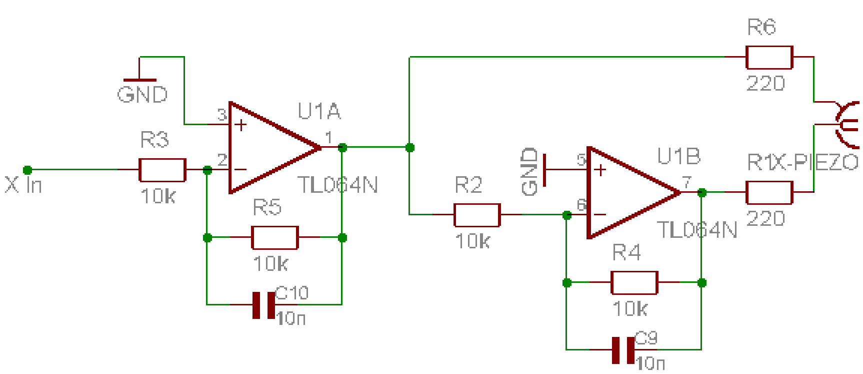


Figure 7: *Supply for x-piezo. Supply for y-piezo is identical.*

More difficult than the supplies for the x- and y- piezoelectric devices was the supply for the z-piezo. This was more difficult because it includes feedback to maintain the constant tunneling current as well as various control loops to maintain the desired parameters.

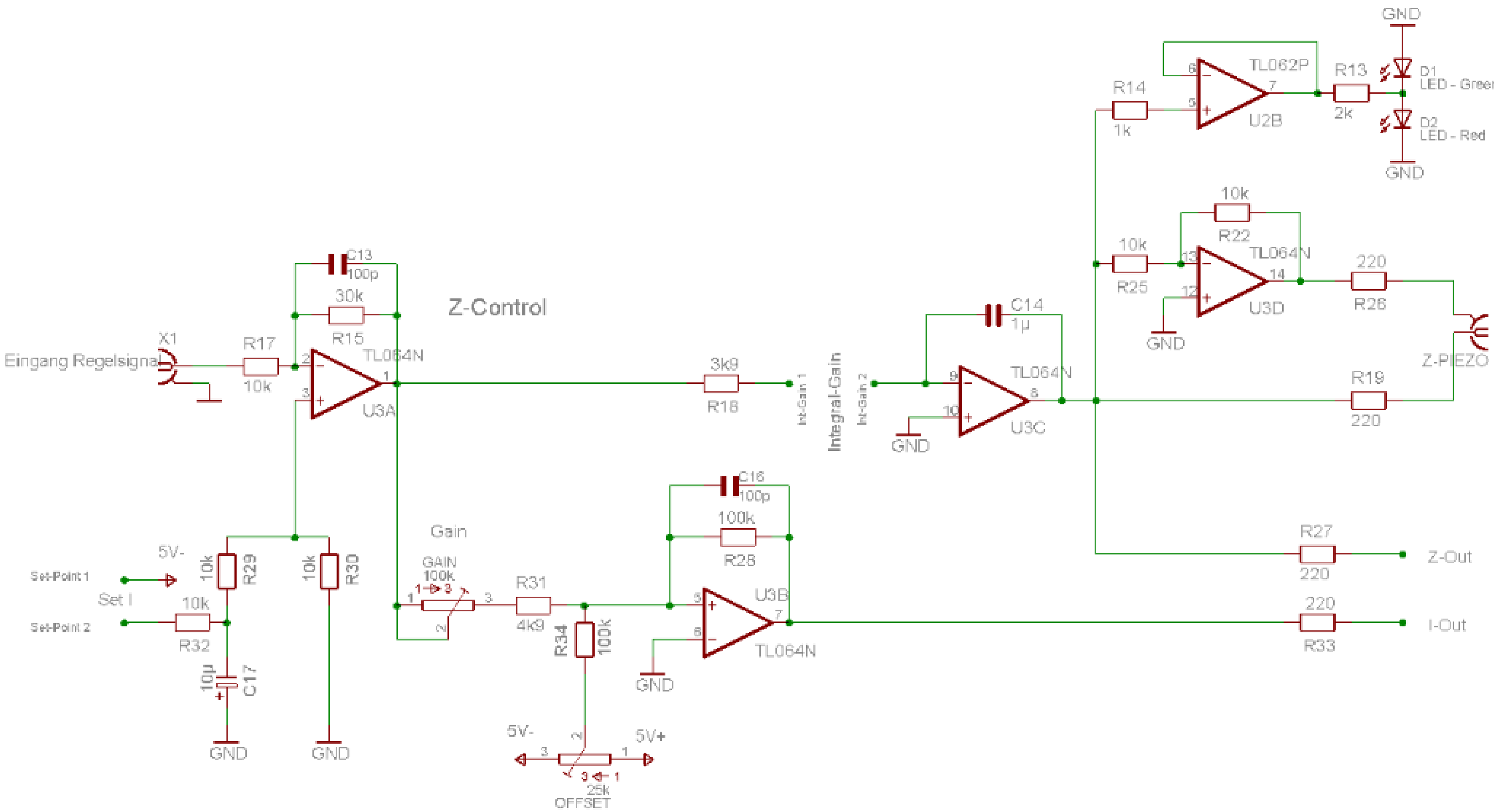


Figure 8: *Supply and control for z-piezo.*

The circuit takes the amplified signal from the pre-amp and compares that with the "set-point" voltage, which determines the constant tunneling current. The difference between these two signals is then integrated. The gain on the integrating amplifier is controlled via a potentiometer. Changing this potentiometer changes the rate at which the feedback reacts. The integrated signal is then sent to another op-amp that creates a symmetric power supply for the z-piezo, in a similar fashion as the x- and y- piezoelectric supplies. The power going to the z-piezo is output to the computer to provide the information needed to image the sample, as well as an additional channel measuring the actual tunneling current.

IV.) Results and Future Work

Unfortunately due to time constraints, the construction of our STM was not fully completed. Each individual component was constructed and met the desired specifications, but the parts have yet to be connected into a cohesive whole. This, however, is not really the big obstacle remaining. The computer control and interface portion of the STM has yet to be written. This program would output voltages to the x- and y-piezoelectric devices in a grid pattern to scan the sample, collect the z-output data from the z-piezoelectric supply, and assemble all of the collected data into an image. This was to be done using LabView. LabView offers the advantage of having built-in virtual instruments (or VI's) that would make the imaging portion of the programming a snap.

V.) Conclusions

We were able to get all 3 parts made and working, but unfortunately ran into issues when putting the whole system together. Further things that need to be worked on is obtaining a good shutoff circuit for creating the tips, as well as being able to put all the pieces together to make a complete STM instead of just have all three separate parts.

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