PARALLEL PLASMA FIELD DIRECTED SPUTTER SHARPENING OF FIELD EMITTERS

BY

ERIC DERWEN LEE

B.S., University of Illinois at Urbana-Champaign, 2007

THESIS

Submitted in partial fulfillment of the requirements
for the degree of Master of Science in Electrical and Computer Engineering
in the Graduate College of the
University of Illinois at Urbana-Champaign, 2009

Urbana, Illinois

Adviser:

Professor Joseph Lyding

ABSTRACT

Many of the current methods to sharpen field emitters are time consuming and can only process one field emitter at a time. In this thesis, a method is proposed to parallel process multiple field emitters using a DC glow discharge plasma as the ion source in conjunction with field directed sputter sharpening methodology. With field directed sputter sharpening, a positive voltage bias is applied to the field emitter and impinging ions are repelled away from the apex of the tip to the shank of the tip. This produces a very sharp tip with radius of curvature of approximately 1 nm. A plasma system was designed and built, and tungsten tips for use in scanning tunneling microscopy (STM) applications were sputtered with argon ions. The result is that as the tip voltage bias to plasma voltage bias ratio is increased above approximately 0.1, the cone angle of the tungsten tip decreases. The radius of curvature of the tip also decreases, but the result is not always consistent and does not reach the same order of magnitude as the result achieved by field directed sputter sharpening with an ion gun. This result shows much promise and further work is necessary to refine the process and achieve consistent results.

ACKNOWLEDGMENTS

I would like to acknowledge the many contributors to this thesis. I would like to thank Professor Joseph Lyding for allowing me to join his research group and to be able to write this thesis. Much of this work would not have been possible without the contributions from Scott Schmucker, who constantly gave feedback and ideas for implementing the experiment. I would also like to thank the rest of the STM group, who will not be named individually but whose suggestions proved vital to the experiment. In addition, none of the characterization of the tungsten tips would have been possible without the use of the TEM in the Beckman Microscopy Suite, with special regards to Scott Robinson.

TABLE OF CONTENTS

1. INTRODUCTION

1.1. Motivation

There are many techniques used currently to image small features and to characterize various surfaces. A large number of these microscopy techniques require the use of a sharpened metallic field emitter to image, such as atomic force microscopy (AFM), field ion microscopy (FIM), and scanning tunneling microscopy (STM). In order to obtain a high resolution image, however, it is necessary to have a sharp field emitter or tip. In addition, there are many applications in field emitter arrays, such as in the use of flat panel displays. The sharper the field emitters are in the array, the more effective the field emission and performance of the display [1]. As a result, many methods have been developed to sharpen and to prepare these field emitters. These methods include annealing the tips to remove oxides and sputtering the tips with an ion gun in order to sharpen the apex [2]. Of these methods, a technique known as Field Directed Sputter Sharpening is very promising as it can produce tips that have a radius of curvature at the apex of the tip of approximately one nanometer [3]. This is extremely sharp and the technique is very reproducible. However, this method can only prepare one tip at a time, and each method can also be very time consuming. In addition, if the method is performed ex situ there is a very good chance that the tip could be damaged during transport or during characterization of the tip, such as in a transmission electron microscope. If this were to occur, the entire process would have to be repeated and therefore it would be very inefficient.

1.2. Thesis Statement

This thesis proposes a method of sharpening and preparing multiple tips at the same time using field directed sputter sharpening by using a plasma as an ion source. Unlike an ion gun which only produces ions in a concentrated beam, the plasma would be able to produce many ions in a larger spread beam, suitable for sputtering multiple targets. These ions would then be accelerated towards the targets, in this case the tips, and would ideally sputter-sharpen the tips. The advantage over traditional sputter sharpening would be that multiple tips could be sputtered at the same time as opposed to only one tip at a time. Another possible advantage would be the reduction of the cone angle at the apex of the tips due to ions having less directionality in a plasma as opposed to an ion gun. This high aspect ratio resulting from a small cone angle is advantageous in the imaging applications of the tips. Before the experiment can be set up, however, it is necessary to provide some background information in order to have some insight into the plasma and the mechanisms of the sputtering process.

2. PLASMA SPUTTERING THEORY

2.1. Plasma Generation

In all sputtering processes, an ion source is needed to produce the ions that will strike and erode the target. These ions are usually of a heavy noble gas, such as argon, neon, or xenon, although nitrogen can also be used. In this experiment argon was chosen due to the fact that its properties in sputtering have been well characterized and that it is readily available [4]. All subsequent discussion will use argon as the plasma gas. In typical sputtering applications, either an ion gun or a plasma is used as the ion source. What is unique in this thesis is that while it is very common to use a plasma to sputter thin films of a material, it is much less common to use a plasma to sputter tips. It is therefore necessary to describe the plasma generation in a little more detail to achieve a better understanding.

In order to achieve the bulk sputtering of tips, a plasma is needed to generate the ions. There are two main types of configurations used to produce a plasma for use in sputtering. These are the glow discharge and RF plasmas [4], [5]. In both cases, there is an anode and a cathode, with the target to be sputtered at the cathode and in cases of thin film deposition the item to be coated at the anode. Otherwise the anode is simply an electrode. A glow discharge plasma is a DC plasma while the RF plasma utilizes a 13.56 MHz switching voltage. The advantage of the RF plasma is that it is a more efficient plasma generator and also that it is not required to have a metallic cathode. When using a DC plasma with an insulating cathode, charge accumulates on the cathode and eventually extinguishes the plasma altogether. With the switching voltage of the RF plasma, this charge problem is alleviated. However, a RF plasma is also very complicated to implement, with matching networks needed to optimize the power to the plasma. In this experiment, an insulating cathode was not used and the energy requirements were secondary to the ease of implementation, so therefore a DC glow discharge plasma was chosen as the plasma generating configuration.

When a voltage bias is applied across the cathode and anode of the glow discharge, an electric field is formed and the structure shown in Figure 2.1 is produced [5]. In a glow discharge plasma, the gas atoms have a certain probability of colliding with each other and with the walls of the chamber, and under certain conditions can ionize due to electron impact ionization, ion impact ionization, photo ionization, or thermal ionization [4], [5]. Ionization produces a free electron and an ion, or an ion pair. In order to sustain a plasma, this ion pair production needs to be above a certain threshold. When a high enough voltage is applied across the two electrodes, the free electrons gain energy due to the high electric field and collide with the neutral gas atoms, causing additional ionization. In some cases excitation occurs instead of ionization, and upon the relaxation of the atom a photon is released. This relaxation and release of photons gives the characteristic glow that is defining of the DC glow discharge plasma. Depending on the gas type, there are different energies required to produce ionization or excitation. For argon, it is necessary for the impacting particles to have 11.56 eV for excitation and 15.76 eV for ionization [4].

There are many operating modes for a glow discharge plasma, and most need to be avoided in order to obtain a stable plasma. The equation for the ion production rate per unit volume of plasma per unit time is shown in Equation (1.1) below [4].

$$2.09\times10^{8}(p)(a)(n_e)\int_{\frac{eV_i}{kT_e}}^{\infty}(E-eV_i)^{b}(E^{\frac{1}{2}})(\frac{E}{kT_e})^{\frac{1}{2}}\exp(-\frac{E}{kT_e})d(\frac{E}{kT_e}) \qquad (1.1)$$

For argon, a = 0.125, b =1.077, eV_i = 15.76, p is pressure and n_e is plasma density.

From Equation (1.1) it can be seen that the ion pair production rate directly depends on the pressure of the system and also on the energy which is dependent on the input voltage. Depending on the gas species, there is a minimum voltage that is required in order to create a stable plasma. The maximum voltage, however, is limited by the pressure in the system. When the pressure is too low in the system for a given voltage bias, the current conduction is low and the plasma is not visible and ions are not being produced at the rate required. However, if the pressure of the system is too high, the ion pair production increases and the current conduction is high and the plasma reaches the abnormal glow and eventually arcs, causing possible damage not only to the sample but also to the power supplies and the system itself. Therefore, it is necessary to maintain the pressure so that for a given voltage it does not arc and is also not extinguished.

2.2. Sputtering Yields

With the basic understanding of the glow discharge plasma as a source of ions, the process of sputtering can now be investigated. When an ion is accelerated towards the target, there are a few things that can occur. When the ion impacts the surface, a target atom or ion can be knocked out of place and ejected from the bulk. The ion can also be reflected off the target, and because it is an inelastic collision the ion will lose a large amount of energy to the target and essentially be neutralized.

The ion can also be implanted into the target, and thereby alter the target. Finally, a secondary electron can be ejected as a consequence of the impact. For sputter sharpening, the process of interest is sputter etching, in which the desired outcome of the impact of the ion is to eject an atom of the target. The sputtering rate of a material is dependent on the gas species of the ion and also on the angle of incidence, as well as on the pressure of the system. In general, the sputtering yield is given by equations (1.2), (1.3), (1.4) [4]. Equation (1.2) is only valid for energies below 1 keV, with Equation (1.3) being valid for energies above 1 keV.

$$S = \frac{3\alpha}{4\pi^2} \frac{4 m_i m_t}{(m_i + m_t)^2} \frac{E}{U_O} \tag{1.2}$$

$$S = 3.56\alpha \frac{Z_i Z_t}{(Z_i^{2/3} + Z_t^{2/3})} \frac{m_i}{(m_i + m_t)} \frac{s_n(E)}{U_O} \tag{1.3}$$

$$s(E) = \frac{m_i m_t}{(m_i + m_t)^2} E \times const \tag{1.4}$$

where m_i and m_t are colliding masses, E is energy, α is a monotonic increasing function of $\frac{m_t}{m_i}$.

From these equations it can be seen that a heavy atom is ideal, and in this experiment argon was chosen because it is a heavy noble gas. Obviously, the energy input to the system across the plasma electrodes has perhaps the most important contribution to the sputtering rate as the energy of the ions is directly dependent on the electric field energy input. Also, the pressure of the system has an effect on sputtering yield, as a lower pressure allows for a higher sputter rate due to fewer atoms in the chamber and a longer mean free path, resulting in fewer backscattering collisions that would cause the sputtered element to redeposit itself on the target [4].

In this experiment, the target to be sputtered is a tungsten tip, and the effect of the ion energy on the sputtering yield of argon on tungsten is shown in Figure 2.2 [6]. The angle of incidence of the ion on the target also affects the sputtering yield. Depending on the target composition, atoms in some orientations are more easily removed, and this is shown in Figure 2.3 [4], [7]. This also intuitively makes sense because at 0˚ a 180˚ reversal of momentum is unlikely for sputter ejection and at 90˚ the ions are not even hitting the target.

Sputter sharpening is typically performed using an ion gun in a vacuum chamber, where all of the above can be controlled easily. The energy of the ion, the angle of incidence, and the pressure of the system are all user-controlled parameters. Since a plasma is used in this experiment, there are many differences that need to be taken into account. A plasma inherently produces ions that have some overall direction but when striking the target are very random. The electric field accelerates the ions in the general direction of the target, but the angle of incidence of the impact is very wide. As a result, it is very possible that the target would be made blunter by the process instead of sharper. In addition, the energy of the ions is also not very well controlled. In an ion gun system, the energy of the ions is well controlled with very little deviation from the energy input. However, in a plasma the energy is very random due to the collisions that take place. This leads to a very wide distribution of ion energies, and therefore the sputtering rate cannot be predicted accurately. A comparison of the distribution of the ion energies for a plasma and an ion gun is shown in Figure 2.4 [5]. The energy applied to the plasma system determines only the maximum amount of energy that an ion can have. This can be partially controlled by increasing the voltage input. The only parameter that could be truly controlled in this experiment was the pressure of the system and the gas species used. These issues, while they cannot to be eliminated completely, can be alleviated by some modifications to the system. These modifications are detailed in subsequent chapters.

2.3. Figures

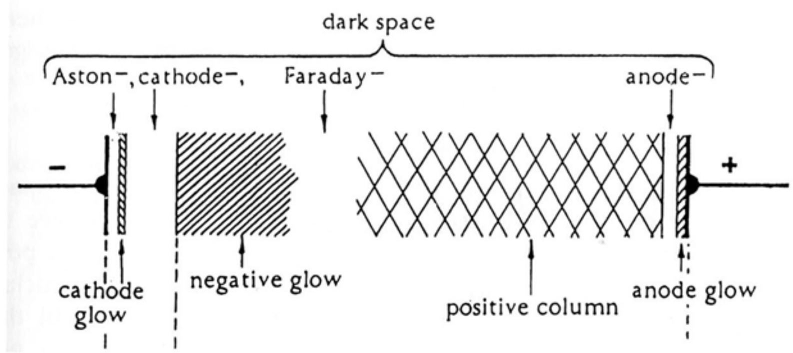


Figure 2.1. Glow discharge plasma

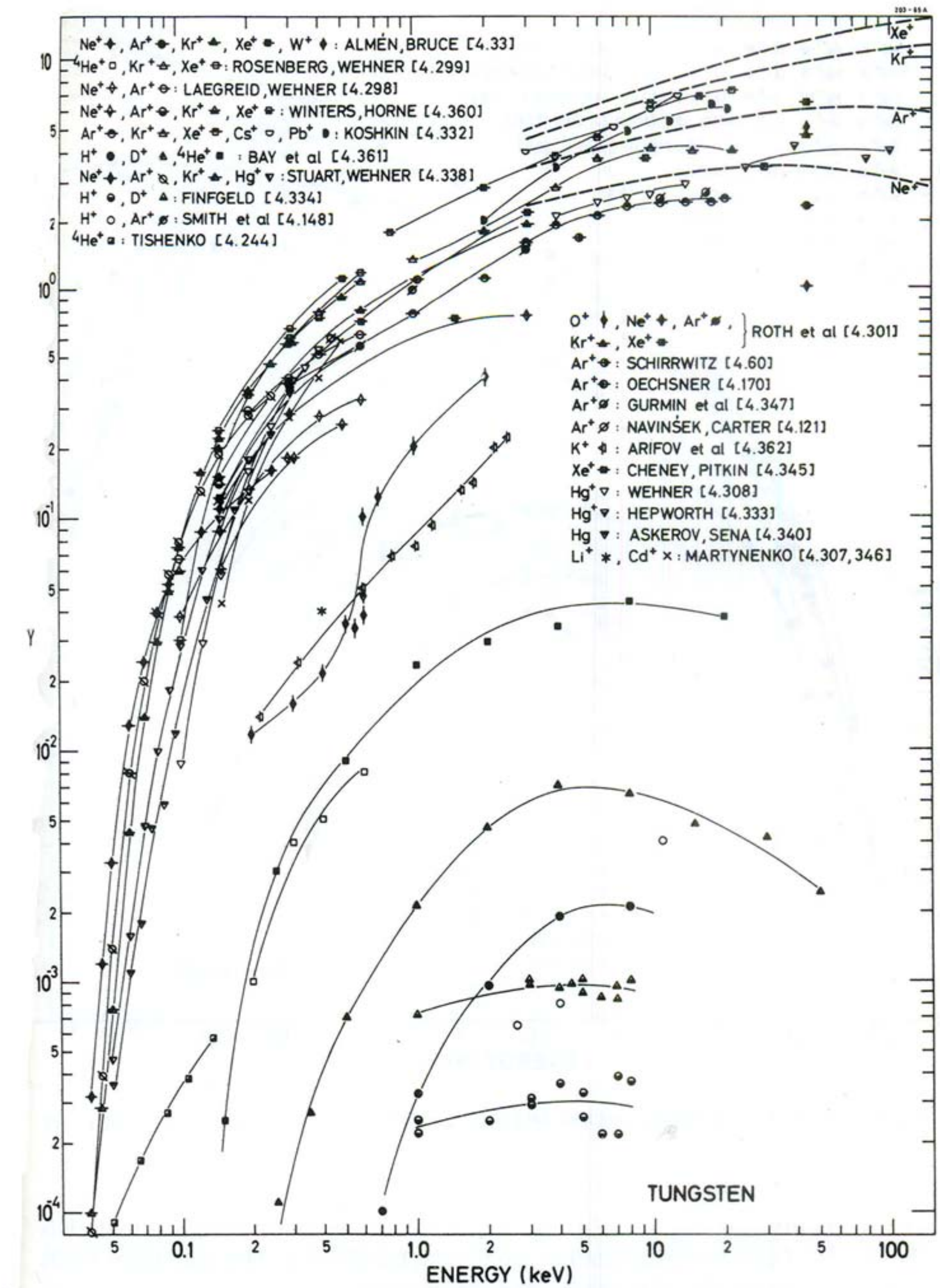


Figure 2.2. Sputtering rate of Ar on W

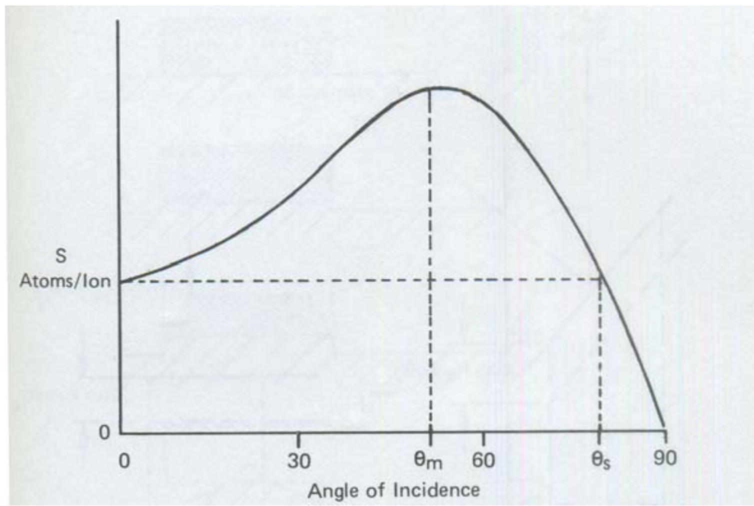


Figure 2.3. Dependence of angle of incidence on sputtering yield

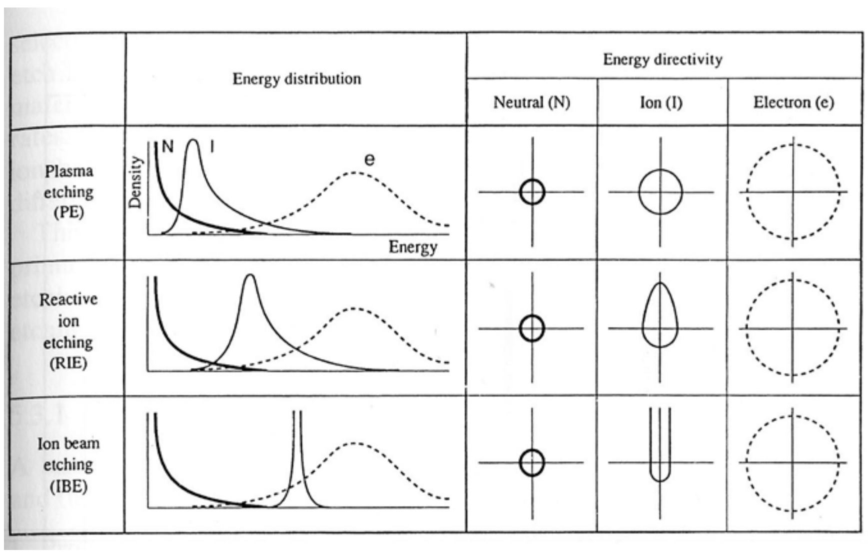


Figure 2.4. Ion energy distribution for the ion gun and a plasma

3. FIELD DIRECTED SPUTTER SHARPENING

3.1. Conventional Sputter Sharpening

Sputter sharpening is not a new technique and has been used extensively to condition and sharpen tips. However, the effectiveness of conventional methods have been limited and leave much to be desired. In conventional sputter sharpening, an ion gun is used to shoot high-energy ions in order to sputter the tips. This occurs at low pressures with ion energies in the thousands of electron volts. The angle of incidence on the tip can also be easily controlled and even altered as the tip is sputtered. It is a very reproducible technique that can produce tips that are not only free of oxide but have relatively sharp tips, with radius of curvature of approximately 20 nm. However, in the past few years a new technique has been introduced that has been able to sharpen tips to a radius of curvature of approximately 1 nm, much sharper and almost at the limit of the imaging systems used to characterize these tips. This technique is known as field directed sputter sharpening, or FDSS [3].

3.2. Field Directed Sputter Sharpening

Field directed sputter sharpening builds upon the conventional sputter sharpening by adding an additional voltage bias to the tip being sputtered. This voltage bias is a positive bias that creates an electric field that emanates from the tip. From general physics, it can be determined that the electric field is highest at sharp points, and therefore the tip and any small protrusions along the tip have the highest electric field. What this does to the sputtering is that ions are repelled from these sharp points, creating an ion path shown in Figure 3.1. Locations that have a smaller electric field are therefore sputtered away, but the apex is preserved and results in a very sharp tip. This greatly reduces the radius of curvature from the conventional technique, and any ion gun sputtering system can be easily modified to accommodate a tip voltage bias. An example of the results of this sputtering is shown in Figure 3.2 [3]. This can be applied very easily to the plasma sputtering case, and should result in tips that are much sharper than could be attained with conventional sputter sharpening techniques.

3.3. Figures

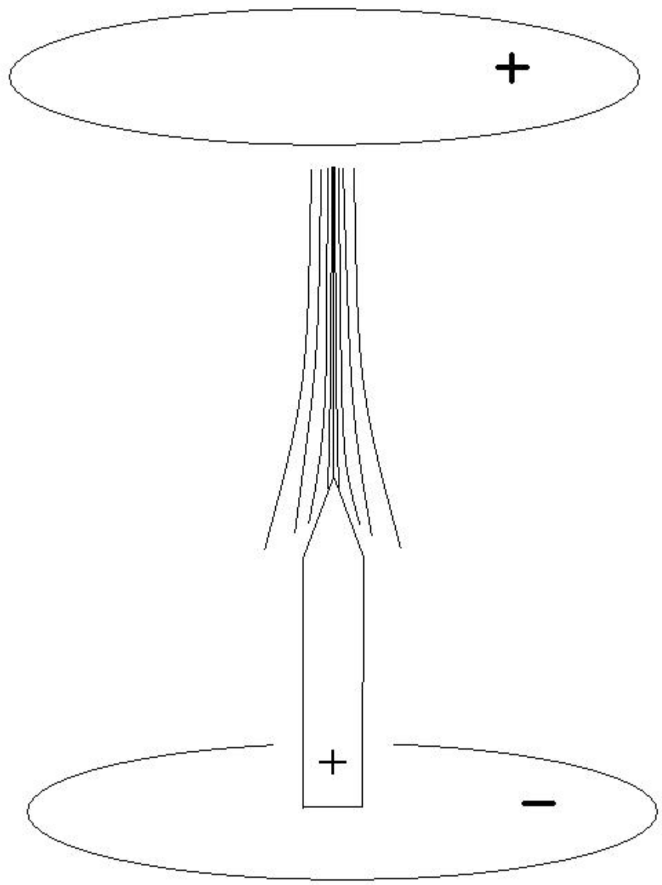
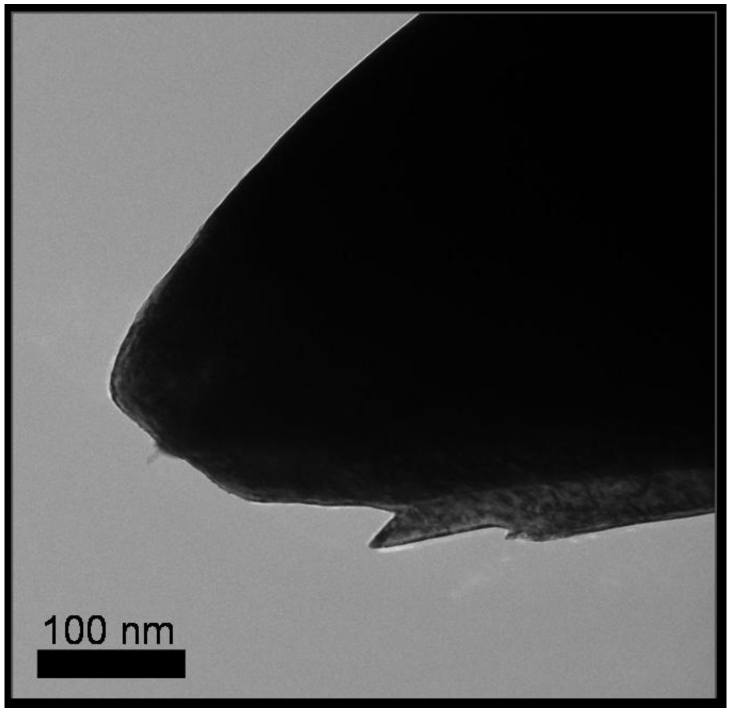


Figure 3.1. Ion path in FDSS

Before



After

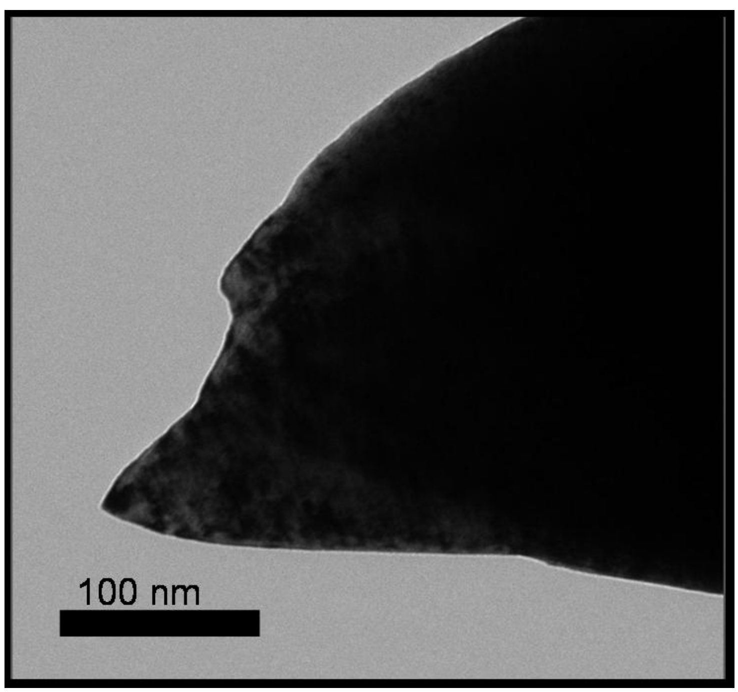


Figure 3.2. Example of FDSS

4. EXPERIMENTAL SETUP

4.1. System Setup

With the proper background information, the experiment could now be set up. This experimental setup is a continuation of previous work performed by Scott Schmucker and Daniel Lukman. Their work involved the construction of the glass cylinder and the initial tip holder design. These are described in further detail below. The chamber was constructed with the purpose of allowing easy access to its interior in order to remove the tip holder and to exchange tips without having to unscrew or remove any large part of the system. The system comprised four main parts that were attached to an existing metal vacuum chamber.

In order to sustain a plasma, a vacuum system needed to be attached to the system in order to pump the system down and to control the pressure of the system. This was satisfied by a standard configuration of a roughing pump and a turbovacuum pump. At the pressures needed to generate a plasma for sputtering ~1E-2 torr [4], only the roughing pump needed to be turned on. The turbovacuum pump was only turned on when the tips were left in the chamber overnight in order to prevent oxide growth and other contamination. The chamber was also fitted with a gate valve and a leak valve. The gate valve was installed between the vacuum pumps and the chamber, and could be adjusted to maintain the proper pressure. The leak valve was installed to introduce the plasma gas species into the system, which as mentioned in the previous section was chosen to be argon due to its inherent properties as a heavy noble gas. The pressure in the chamber was monitored by a convectron gauge in addition to an ion gauge.

A glass cylinder chamber was previously constructed in order to allow the high voltage feedthrough that would bias the anode of the plasma system and also to contain the plasma. The glass construction also allowed the plasma to be visible so that the plasma generation could be verified. The anode itself was a copper ring attached to a collet that was attached to the high voltage feedthrough. The chamber was fitted with a load-lock door so that when the system was vented the door could be opened and the tip holder could be removed. In order to be able to move the tip holder from within the glass chamber to the load-lock door, a linear manipulator was installed opposite the glass plasma chamber. The full range of the linear manipulator was 11 inches, and it was constructed with a welded bellows and a 12 inch aluminum rod. At the end of the aluminum rod was attached a small macor block that was secured by two screws. The macor block was also drilled with holes and threaded to accommodate two long screws that would be screwed in parallel to the aluminum rod. These two screws were dethreaded at the ends in order to allow an easier and more solid connection to collets, and they were also attached to two wires that would provide independent biasing for each screw. The purpose of these two screws was to attach to the removable tip holder and also to provide bias to the cathode and also to the tips in the case of field directed sputter sharpening. Two screws are necessary so that the tip holder is securely attached and does not move around. On the other end of the welded bellows was attached a metal construct that allowed for three electrical feedthroughs. Two of these were used to provide feedthroughs for the ground and the tips. The completed system can be seen in Figure 4.1.

4.2. Tip Holder Design

The tip holder was designed so that multiple tips could be mounted and sputtered simultaneously. The material used for the tip holder was copper, with 10 tip holders soldered into the copper. In the original design, there were two copper plates that sandwiched a macor block. One of the plates, which will be designated the back plate, contained the tip holders, with the macor block and the other plate, designated the front plate, drilled to allow the tip holders to be exposed. These were secured by a 0-80 screw and an 8-32 screw attached to two copper collets. The collets would be able to attach to the two screws mentioned in the previous section that were attached to the macor block on the linear manipulator. The 0-80 screw was secured to the front plate and surrounded by an alumina tubing to allow for an independent biasing of the front plate. The 8-32 screw was attached directly to the back plate and allowed for the back plate to be biased and therefore provide for the tip bias. The reason for this design was that initially the front plate was meant to act as the cathode for the plasma. Subsequent design removed the front plate and the macor block as a separate cathode was implemented with a stainless steel mesh. The tip holder design can be seen in Figure 4.2 and the construction in Figure 4.3.

4.3. Power Supply

In order to generate the plasma and also to provide bias to the tips, power supplies were needed that were able to generate high and stable DC voltages. There were multiple power supplies that were used in the setup of the system. Initially, the Systron Donner M104 was used to generate the plasma. This power supply was able to provide a very stable DC voltage output up to 1000 V but was limited to its current output of 5 mA at that high voltage. While it was able to generate a plasma, it was not a stable plasma and had an issue where the plasma flickered as the current limit of the power supply reduced the voltage output. The Systron Donner was later used to bias the tips in field directed sputter sharpening. This led to the use of Acopian power supplies connected in series to generate the plasma. Each power supply was able to generate a very stable 150 V with a 1 A output. However, due to issues with the proximity of the high voltage lines in the power supply to the chassis, the manufacturer warning was to connect a maximum of two power supplies in series. Therefore, the total voltage that could be applied between the anode and the cathode was 600 V. Two power supplies would provide a positive 300 V while another two would provide a negative 300 V with a common ground between them. While this was able to produce a stable plasma, the energy of the ions was not enough to produce a decent sputtering rate and the setup proved to be complex with many wires required. Finally, an ion gun power supply was acquired that was able to produce 3000 V, with a 50 mA current limit that was sufficient to produce a stable plasma with highly energetic ions.

4.4. Plasma Generation Configurations

In order to generate a stable plasma, there were three different configurations that were experimented with until the final setup. The first iteration had simply the anode with the high voltage feedthrough and the initial tip holder design. With this design and the Systron Donner power supply, a suitable plasma could not be generated as the front plate cathode of the tip holder was at the same potential as the rest of the chamber. Therefore, a plasma was generated but was not confined to the glass chamber. In order to confine the plasma, an alternative configuration was investigated that involved the introduction of a stainless steel mesh [8].

In the second configuration of the plasma system, a stainless steel mesh was placed right behind the tip holder and attached to the aluminum rod. It was biased at the same voltage as the front plate of the tip holder. With the initial Systron Donner power supply, the current limit was reached very quickly and the voltage was limited to sub-500 V. This caused a flickering, unstable plasma which led to the construction and use of the Acopian power supplies. With this mesh configuration and the Acopian power supplies, a stable plasma was generated that was confined to the glass chamber. However, due to the nature of the plasma, this configuration was not suitable to sputter tips as the directionality of the ions in the plasma was random, so the tips were being sputtered on all sides resulting in much blunter tips. In addition, there was a leak that caused nitrogen from the atmosphere to leak into the chamber. While this leak was small, it was enough to change the color of the plasma to a bright purple, which indicated the nature of the gas entering into the system. However, it was later discovered that nitrogen mixed with argon would not have a detrimental effect on the sputtering, and that the adsorption of nitrogen on tungsten would sometimes aid in the sharpening [9], [10]. This issue was still a cause for concern, and was somewhat mitigated in the next configuration by removing a vent valve.

The third and final configuration placed the stainless steel mesh between the anode and the tip holder. The mesh was essentially sized to fit the circumference of the glass tube and was placed 4 inches from the anode. With this configuration, not only would the plasma be confined to the glass chamber, but directionality of the ions would be somewhat filtered to allow only ions traveling perpendicular to the mesh to reach the tip holder. This configuration seemed to produce the best results, and also allowed for the tip holder to be changed so that the macor block and the front plate could be removed. Tips could therefore be more easily placed in the tip holder without having to worry about scraping and ruining the tips on the front plate and macor block. Also, tips of lengths shorter than width of the front plate and macor block could be placed in the tip holder. The three different configurations can be seen in Figures 4.4-4.6.

4.5. Sample Preparation

The samples that were used in this experiment were 15 mil tungsten wires. This size was used instead of a 9 mil tungsten wire in order to facilitate the insertion of the tips into the tip holder. The tips were fabricated using the conventional drop-off technique [11]. Essentially, the wire was placed through a gold loop that had a film of 3M NaOH in the loop. The gold loop acted as a counter-electrode to the positively biased tungsten wire. By applying a voltage bias of 3-5 V, the tungsten wire would be electrochemically etched by the NaOH. After approximately 20 min, the wire would have been sufficiently etched so that the part hanging underneath the gold wire would fall into a small container. Once the etching was complete, the tip was rinsed with acetone, isopropyl alcohol, and deionized water, and then dried with nitrogen. If the tip was to be subsequently sputtered in the plasma sputtering system, the tip would be loaded into the tip holder and then placed in the system under vacuum. All characterization of the samples was performed in the TEM in the Microscopy Suite of the Beckman Institute.

4.6. Figures

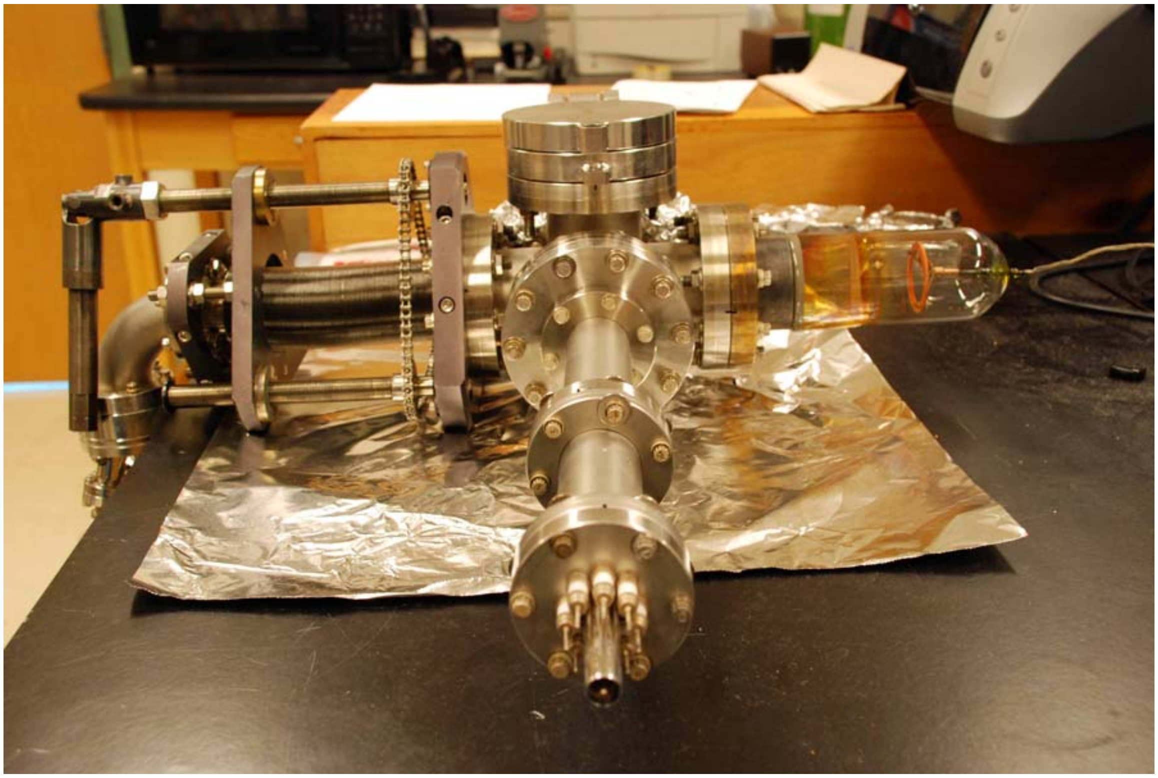


Figure 4.1. System setup

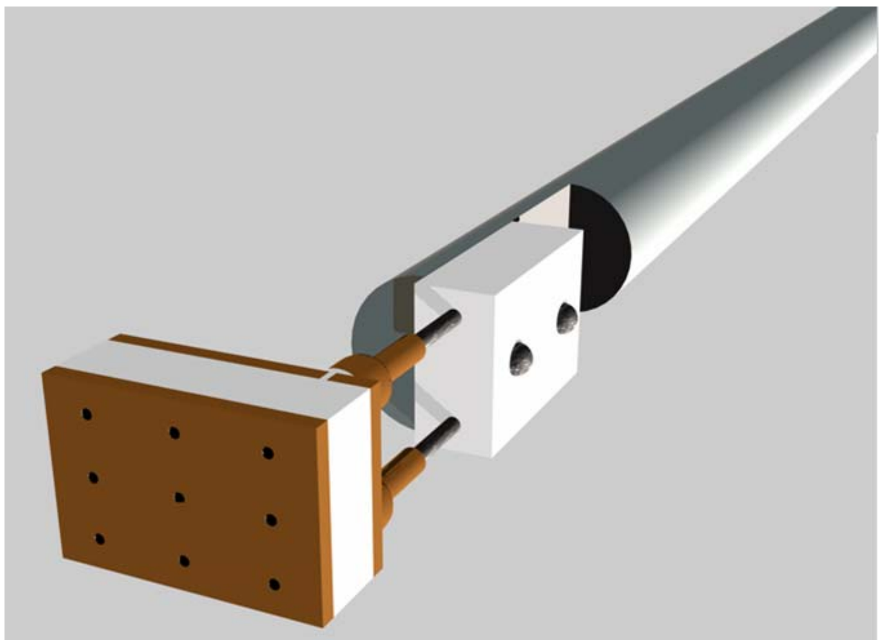


Figure 4.2. Original tip block design

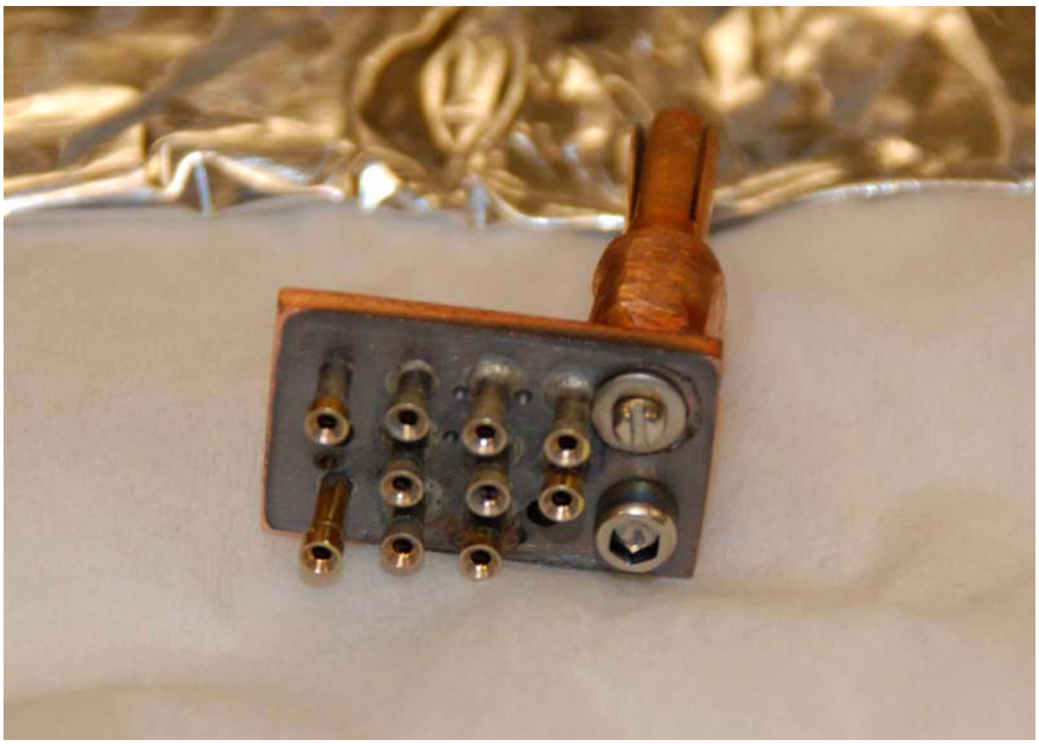


Figure 4.3. Tip block construction

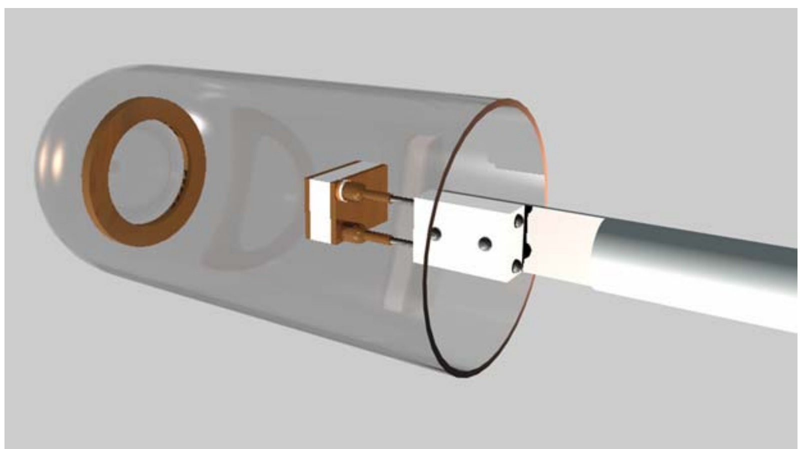


Figure 4.4. Initial configuration

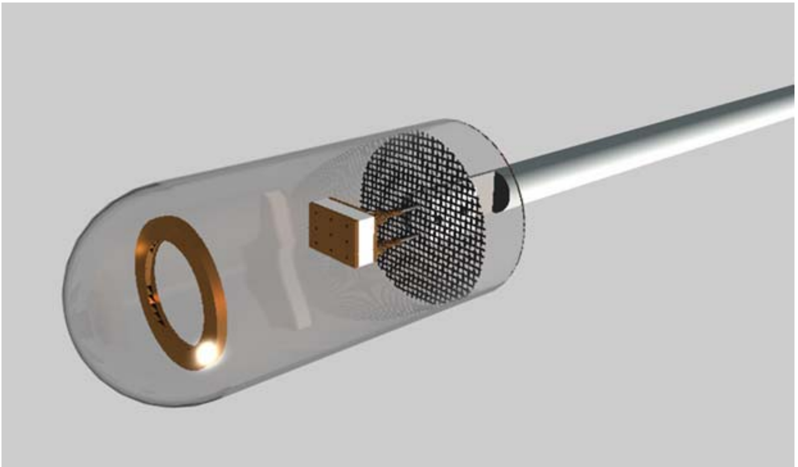


Figure 4.5. First mesh configuration

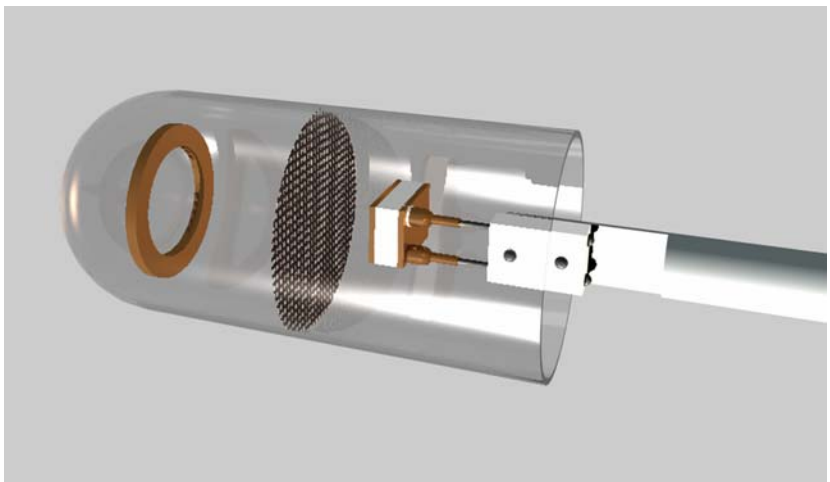


Figure 4.6. Second mesh configuration

5. EXPERIMENTAL RESULTS

5.1. Test Configurations

The test configurations were split into two different sets based on the system configurations from the previous chapter. These are data for the two mesh configurations. The configuration before the introduction of the mesh did not yield any results as no tips were sputtered in the system. The data from the each step led to the development of the subsequent configurations, and the test parameters are consistent with the change in test equipment. In all cases the experiments were conducted at room temperature, with two tips being sputtered simultaneously. In each test case, the important parameters are the voltage of the plasma, the pressure of the system, time spent sputtering, and the voltage bias on the tip. Whenever a voltage bias was applied on the tip, the voltage across the plasma was also altered to maintain the same ion energy.

In the first test configuration, no tips were placed in the system for sputtering as much of the work was to set the basis for creating a stable plasma. The element that was crucial in this initial trial was that the plasma was not isolated to the glass chamber, and therefore it became necessary to introduce the steel meshes.

In the second test configuration, the voltage of the plasma was limited to 600 V due to the limitation of the power supply. The pressure of the system was kept at a constant 0.1 torr. The voltage bias was 0 V and 300 V. Because the mesh was behind the tip holder in the system, there was no directionality of the ions. In all cases, the original tip shape was not conserved, and the radius of curvature decreased dramatically. After this initial change in tip shape, however, there was some limited sharpening and reduction of a cone angle, but nothing near the desired range of values. A full set of data is presented in Figures 5.1-5.4. In Figure 5.1, an example of the sputtering with no tip bias is presented. The tip was sputtered for 60 min, and it can be seen that the oxide was effectively removed from the tungsten tip. There was also some limited sharpening, as the radius of curvature decreased from ~500 nm to 150 nm. There was no evidence of decreased cone angle, and the radius of curvature was still much too high to be usable. Subsequent sputtering yielded no change in sharpness. In Figure 5.2, the tip was sputtered for 30 min with a 50 V tip bias. The radius of curvature decreased from 5000 nm to 2500 nm. Additional sputtering was performed for an additional 30 min, but resulted in the same radius of curvature. Figure 5.3 presents a tungsten tip sputtered for 60 min with a 150 V tip bias. The tip was bent in the process, but an interesting result is the reduction in cone angle. The cone angle changed from 23.6˚ to 15.8˚ for a total change of 7.8˚, or 33 percent. In Figure 5.4, the tungsten tip was sputtered for 60 min with a 300 V tip bias. The radius of curvature decreased from 5000 nm to 4000 nm, and the cone angle also decreased from 34.3˚ to 10.6˚ for a total change of 23.7˚, or 69 percent. While technically these results were positive, the range of values for the radius of curvature prevented the usability of any of these tips.

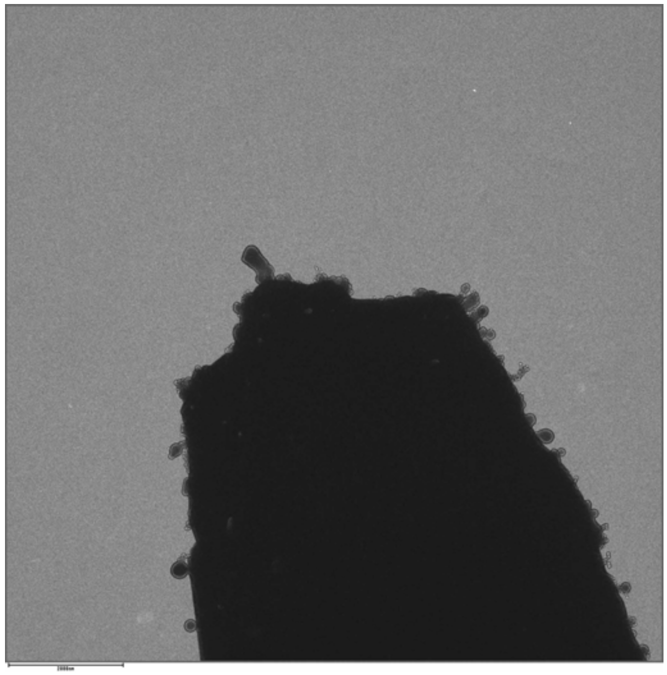
In the third test configuration, the voltage of the plasma was limited to a maximum of 3000 V, but in practice was set in a range from 1000 V to 1850 V. The pressure in the system was set at 0.03 torr for plasma voltages of 1000 V and 1250 V, and down to 0.02 torr for plasma voltages of 1500 V. These were experimentally obtained values such that the plasma did not arc. The voltage bias on the tips was varied from 50 V to 100 V and 200 V. This voltage bias was limited by a phenomenon where the tip block and the mesh created a secondary plasma in the opposite direction of the original plasma. Due to the current limitation of the Systron Donner, this led to an arcing and breakdown of the plasma exactly the same as in the first test configuration. However, besides these limitations many promising results were obtained with this configuration. In all cases the tip shape and radius of curvature were maintained. When there was no tip bias, the results were limited, and many tips appeared to have no visible change besides the removal of oxide on the tip. There were also no visible changes in a tip bias of 50 V. However, in cases of voltage biases of 150 V and 200 V, there were many instances where there was visible sharpening and cone angle reduction. A full set of data is presented in Figures 5.5-5.8. The base plasma voltage for this set is 1.5 kV. The voltage is adjusted so that the energy of the ions remains at a maximum of 1.5 kV, so the bias voltage is added to the base voltage. Another set of data with a base voltage of 1.25 kV is available in Appendix A. In Figure 5.5, the tip was sputtered with no tip bias for 30 min. The radius of curvature was maintained at ~30 nm, and there was no visible change in the cone angle. The oxide on the surface of the tip was removed, however, and the tip appears to be much cleaner. In Figure 5.6, the tip was sputtered with a 50 V bias for 30 min. The radius of curvature was maintained at ~250 nm, and there was a negligible change in cone angle. In Figure 5.7, a tungsten tip was sputtered with a 150 V tip bias. The radius of curvature was maintained at ~200 nm, with the oxide on the tip being removed. There was no visible change in cone angle. In Figure 5.8, the tip was sputtered for 30 min with a 200 V tip bias. In this case, there was reduction in radius of curvature from ~100 nm to ~30 nm. The radius of curvature also decreased from 38.6˚ to 25.1˚ for a change of 13.5˚, or 34.9 percent. These results were much more promising than the previous configuration, with usable tip radii of curvature and decreased cone angles.

5.2. Sharpening and Cone Angle Reduction Evidence

It can be seen from the data that there were many instances of sharpening and cone angle reduction. Results were more prominent with an increased tip bias voltage, especially with regards to the plasma voltage. In particular, as the ratio of the bias voltage to plasma voltage increases, the cone angle decreases. In the first data set the ratio changes from 0 to 0.083 to 0.25 to 0.5. There were no visible changes at the lower ratios, but at the ratios of 0.25 and 0.5 there were 33˚ and 69˚ changes, respectively. In the second set of data, the ratio changes from 0 to 0.03 to 0.09 to 0.11. The cone angle change was only prominent at a ratio of 0.11 where the change was 34.9 percent. This was consistent with another data point taken at 1500 V with a 150 V bias, where the ratio is 0.1. This result is shown in Figure 5.9. In this case the radius of curvature decreased from ~30 nm to ~10 nm, while the cone angle decreased from 21.2˚ to 15.4˚ for a change of 5.8˚, or 27.3 percent. Sharpening was not as consistent, but in most cases of cone angle reduction there was also a reduction in radius of curvature.

5.3. Figures

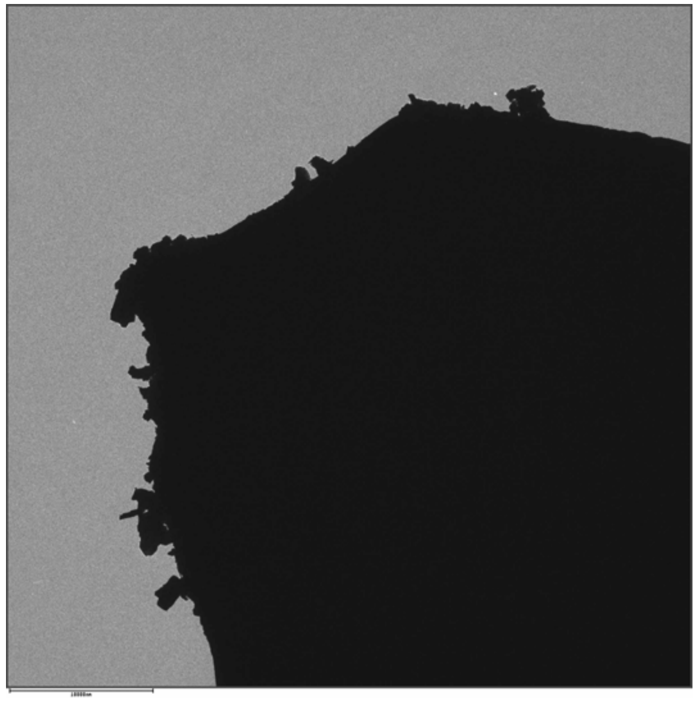
Before



After

Figure 5.1. Second configuration, no bias sputter

Before



After

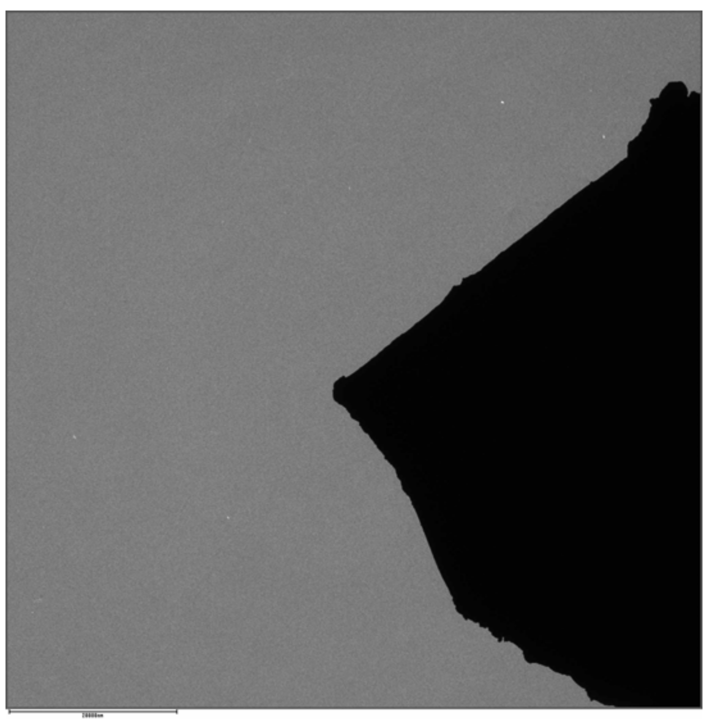
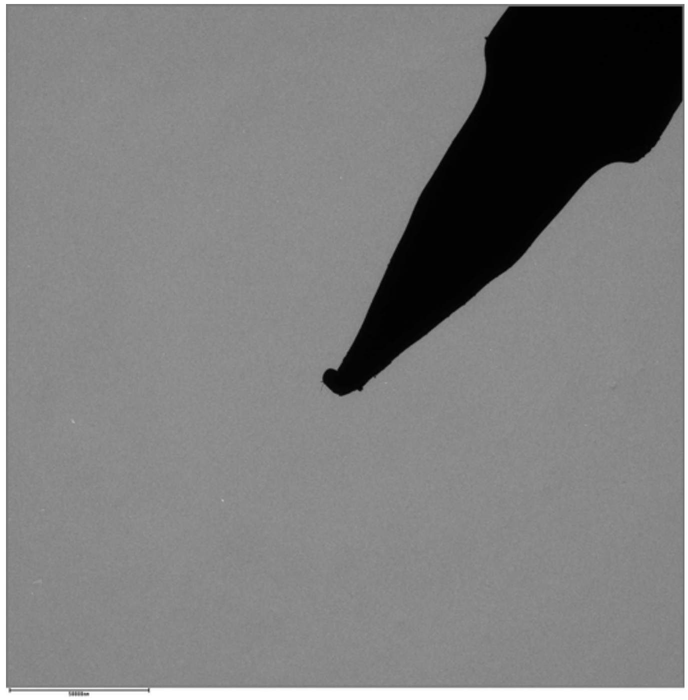


Figure 5.2. Second configuration, 50 V sputter

Before



After

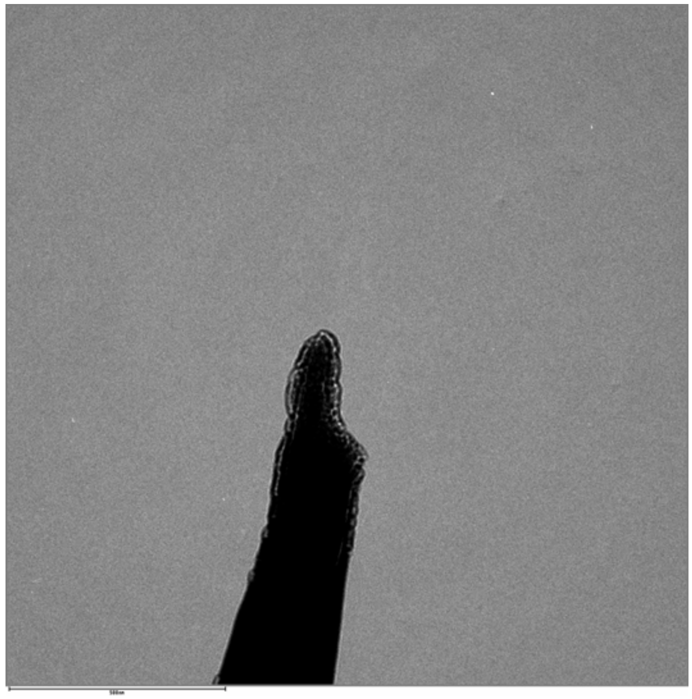
Figure 5.3. Second configuration, 150 V sputter

Before

After

Figure 5.4. Second configuration, 300 V sputter

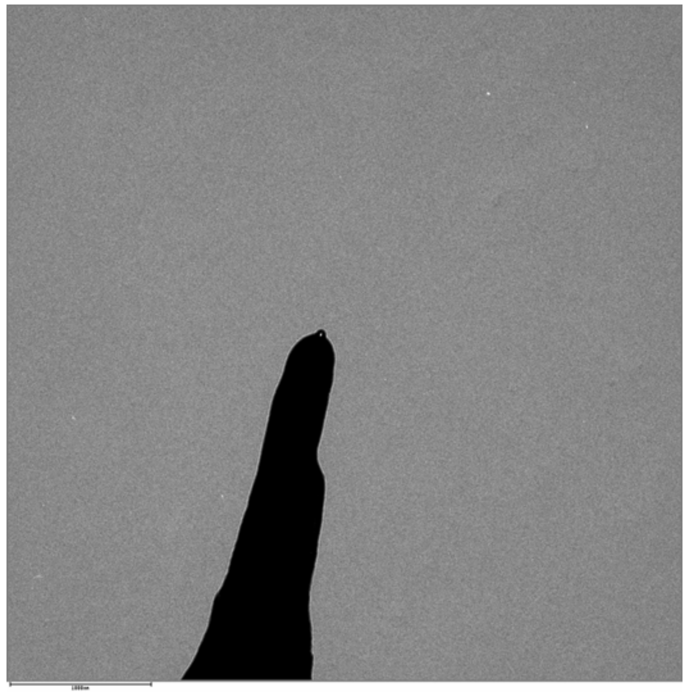
Before



After

Figure 5.5. Third configuration, no bias sputter

Before



After

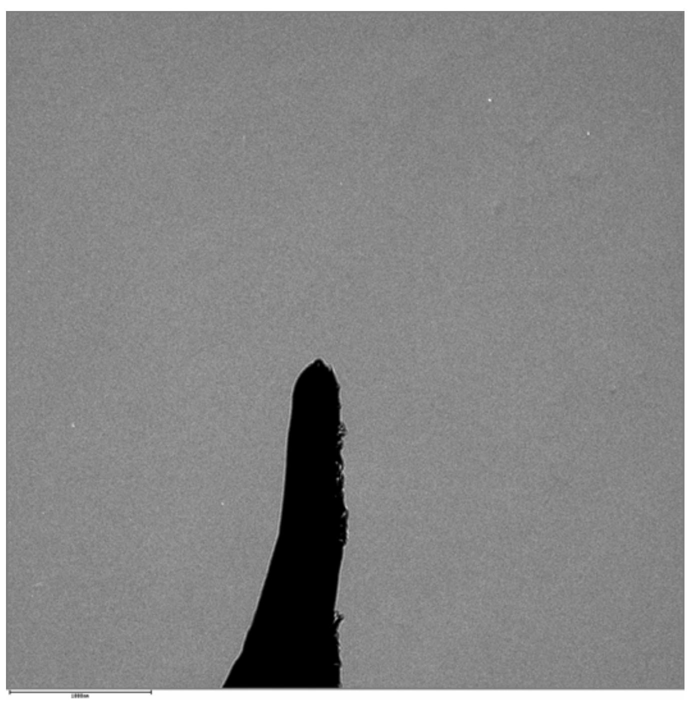


Figure 5.6. Third configuration, 50 V sputter

Before

After

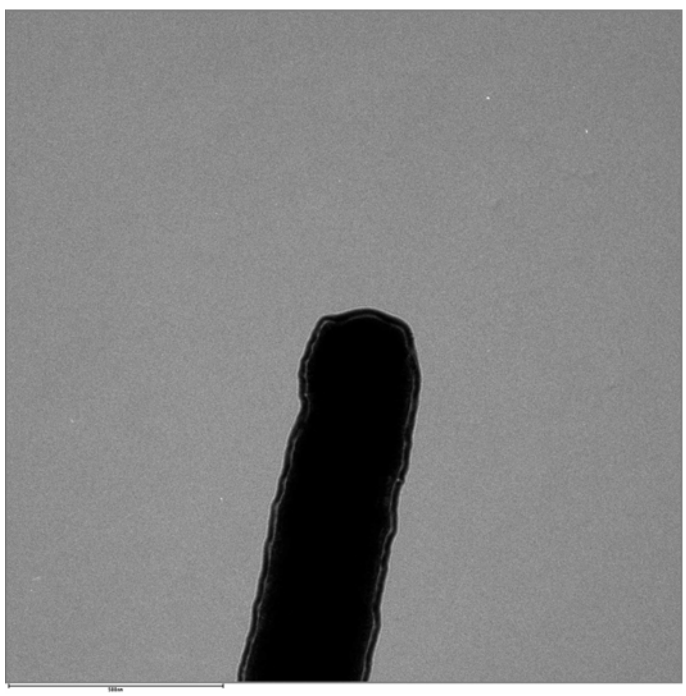
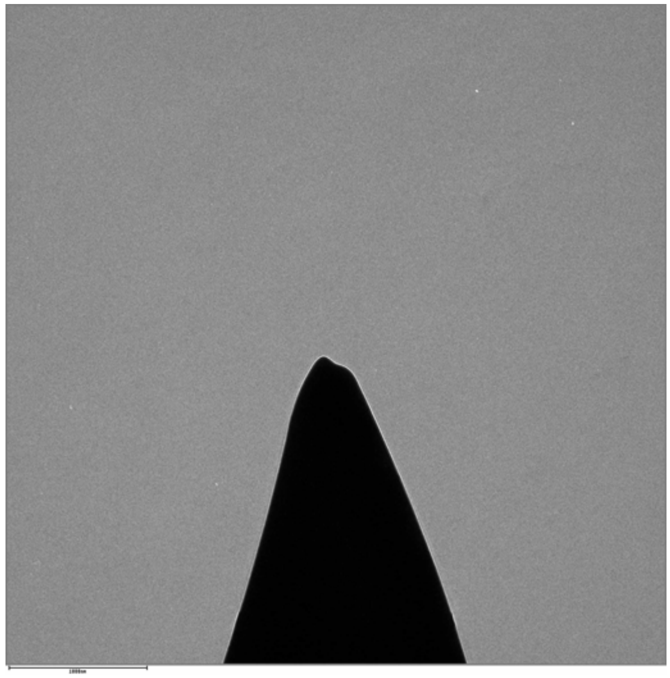


Figure 5.7. Third configuration, 150 V sputter

Before



After

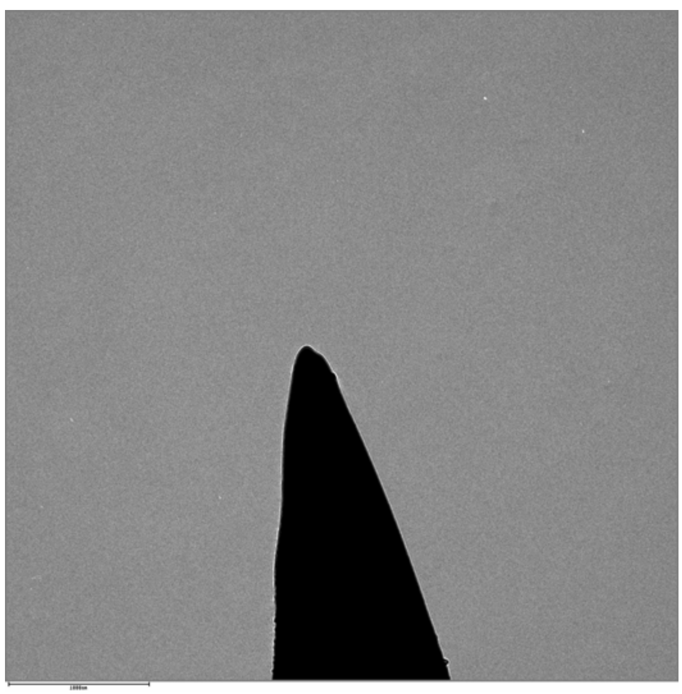
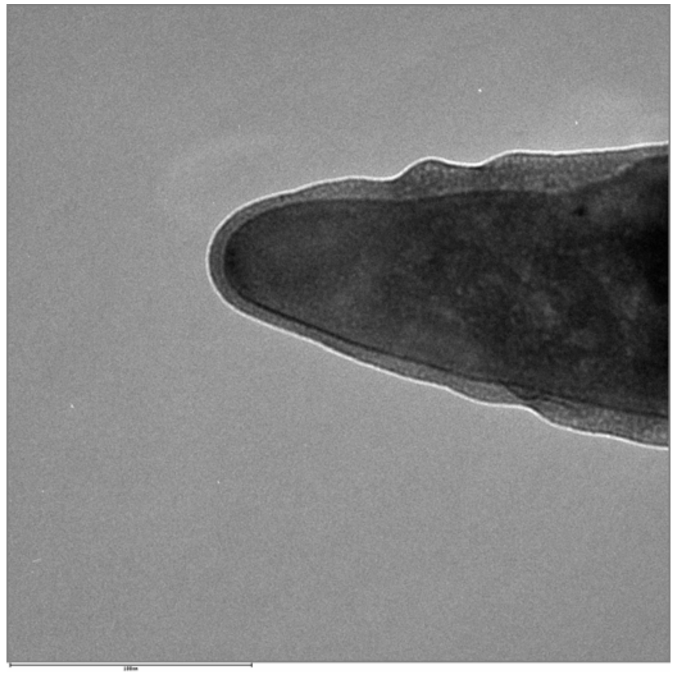


Figure 5.8. Third configuration, 300 V sputter

Before



After

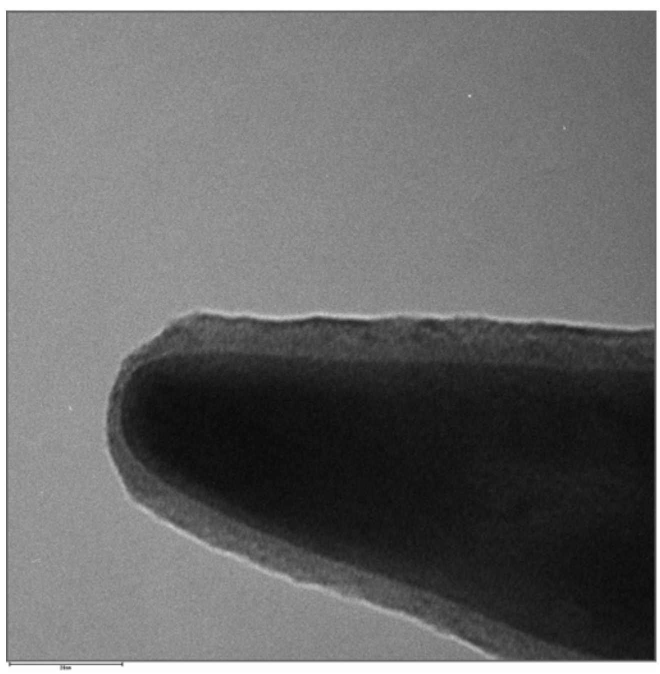


Figure 5.9. 1500 V plasma, 150 V tip bias

6. CONCLUSIONS AND FUTURE WORK

6.1. Thesis Summary

In conclusion, it has been shown that this plasma field directed sputtering system is effective in parallel processing field emitters to a certain degree. As of the writing of this thesis, a case has not been produced where the radius of curvature reached the same level as that obtained with an ion gun. However, the result that is most prominent is the reduction of the cone angle of the tips with increasing tip bias to plasma voltage ratios. This result is logically understandable, as with increased tip bias the electric field at the tip is increased and the impinging ions are further deflected from the apex to sputter the shank of the tip. This maintains the apex and results in a decreased cone angle. More experimentation is necessary in order to achieve a consistent radius of curvature reduction. Plans to achieve this result are outlined below.

6.2. Future Work

With the results from this thesis, there are many opportunities for future work in this experiment. With the present construction of the plasma sputtering system, the energy of the ions is limited to the power supply voltage. In addition, the maximum voltage applied across the plasma is limited to the pressure inside the system due to the plasma gas. Because of the present method of leaking gas into the chamber, only the roughing pump is utilized in order to prevent strain on the turbo pump due to the rapid increase in pressure inside the chamber. This limits the range of the pressure to ~10^-3 torr, and also limits the maximum applied voltage to ~2 kV. In future setups, instead of a leak valve a mass flow controller can be used to leak the gas into the chamber. This would allow more precise control over the gas pressure in the system, and therefore could be used in conjunction with the turbo pump to achieve lower pressures and thereby higher power supply voltages.

Besides changing the gas leakage system, it would be necessary to further isolate the tips from the plasma source. Because of the current configuration, the proximity of the tip block holder to the cathode of the plasma source causes a secondary plasma in the opposite direction to be created when field directed sputter sharpening is utilized. This effect is more predominant as the tip bias is increased, and it results in a current limitation of the tip biasing power supply. Rather than simply substituting a higher current power supply, isolation would also prevent detrimental effects from the secondary plasma such as increased scattering of the ions resulting in blunter tips.

Another future project would be to sputter tips that are composed of different materials, such as iridium or platinum iridium. These tips do not oxidize, and are naturally sharper after the electrochemical etching. In addition, tips of smaller sizes could be sputtered, such as the 9 mil tungsten wires. These tips in general are sharper than the 15 mil wire tips after the drop-off technique, and therefore would be quicker to sputter inside the plasma system. All it would take to be able to sputter these smaller tips would be to modify the tip holder block by crimping the individual tip holders or to place a tip into the holder with another shorter piece of wire in order to hold it in place.

In addition, meshes with different size openings could be utilized in order to see the effects of the mesh hole size on the ion current density at the tips. Changing the mesh could also change the angle of the impinging ions, and perhaps a certain mesh size would produce better results than others. A mesh with larger holes would allow more ions to reach the tip block, but it would also allow ions with wider angles of incidence to be able to reach the tip block. A mesh with smaller holes would reduce the number of ions that reach the tip block, but would filter out ions that had angles of incidence not exactly perpendicular to the mesh.

Finally, in order to improve the ionization efficiency of the plasma, a magnetron configuration could be implemented in the plasma chamber [5], [12]. By placing a magnet behind the tip block, ions would be attracted and confined to the area around the tips. Another method using magnets would be to wrap a coil of wire around the glass chamber. With a current applied, the wire acts as an inductor and produces a magnetic field. This magnetic field could accelerate the ions toward the tips and would provide more efficient sputtering. This setup can be seen in Figure 6.1.

Clearly there is much work that can be done in order to improve the results of the plasma sputtering technique.

6.3. Figures

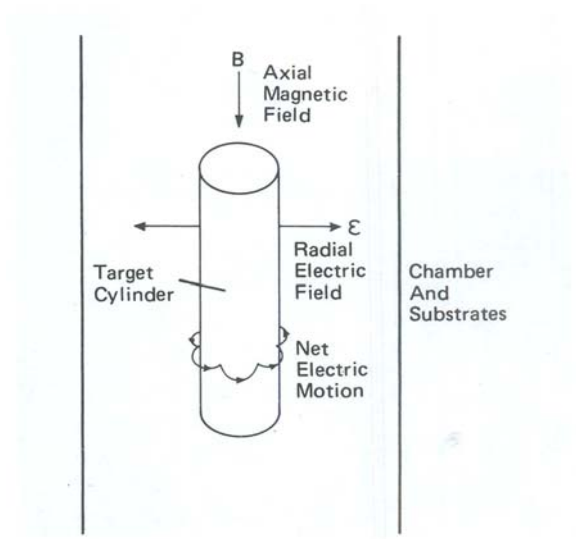


Figure 6.1. Magnetron DC sputtering setup

REFERENCES

[1] O. Auciello, L.Yadon, D. Temple, J.E. Mancusi, G.E. McGuire, E. Hirsch, H.F. Gray, and C.M. Tang, "Ion bombardment sharpening of field emitter arrays," in *1995 International Vacuum Microelectronics Conference*, 30 July-3 Aug 1995, pp.192-196.

[2] P. Janssen and J. P. Jones, "The sharpening of field emitter tips by ion sputtering," *J. Phys. D: Appl, Phys*, vol. 4, pp. 118-23, 1971.

[3] S. Schmucker (private communication), 2009.

[4] N. Chapman, *Glow Discharge Processes Sputtering and Plasma Etching*. New York: Wiley, 1980.

[5] M. Sugawara, *Plasma Etching Fundamentals and Applications*. New York: Oxford UP, 1998.

[6] R. Behrisch, *Sputtering by Particle Bombardment*. Berlin: Springer-Verlag, 1981.

[7] K. B. Cheney and E. T. Pitkin, "Sputtering at acute incidence," *J. Appl. Phys.*, vol. 36, pp. 3542-3544, 1965.

[8] J. Schultz, "Design and characterization of a plasma source for ion bombardment," M.S. thesis, University of Illinois Urbana-Champaign, Urbana, IL, 1997.

[9] H. F. Winters and E. Kay, "Influence of surface absorption characteristics on reactively sputtered films grown in the biased and unbiased modes," *J. Appl. Phys.*, vol. 43, pp. 794-99, 1972.

[10] M. Rezeq, J. Pitters, and R. Wolkow, "Tungsten nanotip fabrication by spatially controlled field-assisted reaction with nitrogen," *J. Chem. Phys.*, vol. 124, pp. 204716, 2006.

[11] P.J. Bryant, H.S. Kim, Y.C. Zheng and R. Yang, "Technique for shaping tunneling microscope tips," *Rev. Sci. Instrum.*, vol. 58, p. 1115, 1987.

[12] K. Wasa and S. Hayakawa, "Low pressure sputtering system of the magnetron type," *Rev. Sci. Instrum.*, vol. 40, pp. 693-697, 1969.

APPENDIX

This appendix contains images for a data set taken with a base plasma voltage of 1.25 kV. The images are taken with no tip bias, and then tip biases of 50 V, 150 V, and 200 V. They are shown in Figures A.1-A.4.

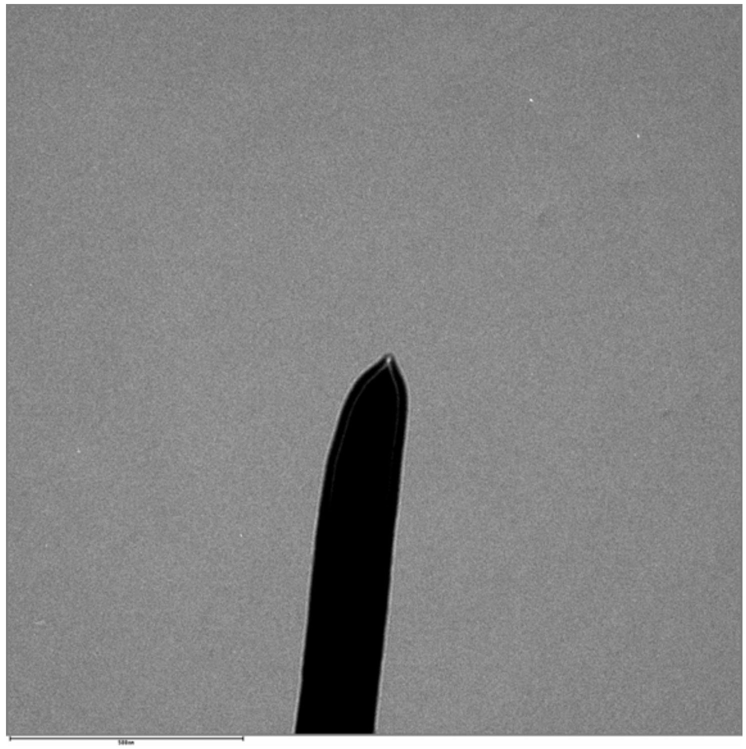
In Figure A.1, the tip was sputtered for 30 min with no tip bias. The only visible change is the removal of oxide at the very apex of the tip. There is no noticeable sharpening change as the radius of curvature is maintained at ~20 nm, and the cone angle remains the same.

In Figure A.2, the tip was sputtered for 30 min with a tip bias of 50 V. The radius of curvature is maintained at ~80 nm and the cone angle also remains the same.

In Figure A.3, the tip was sputtered for 30 min with a tip bias of 150 V. The tip gets noticeably blunter, with the radius of curvature increasing from ~25 nm to ~50 nm. However, the cone angle decreases from 79.5˚ to 52.7˚ for a total change of 26.8˚. This large change in cone angle is probably due to the initial wide shape of the tungsten tip, which also caused the radius of curvature to increase.

In Figure A.4, the tip was sputtered for 30 min with a tip bias of 200 V. The radius of curvature decreases slightly from ~50 nm to ~40 nm, while the cone angle decreases from 72.1˚ to 57.1˚ for a total change of 15˚.

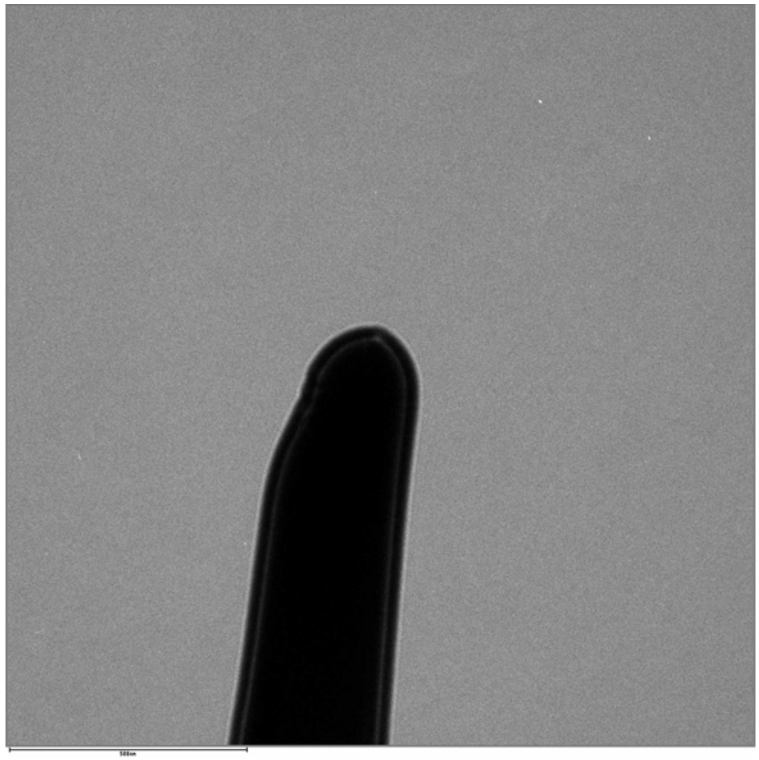
Before



After

Figure A.1. 1250 V plasma, no tip bias

Before



After

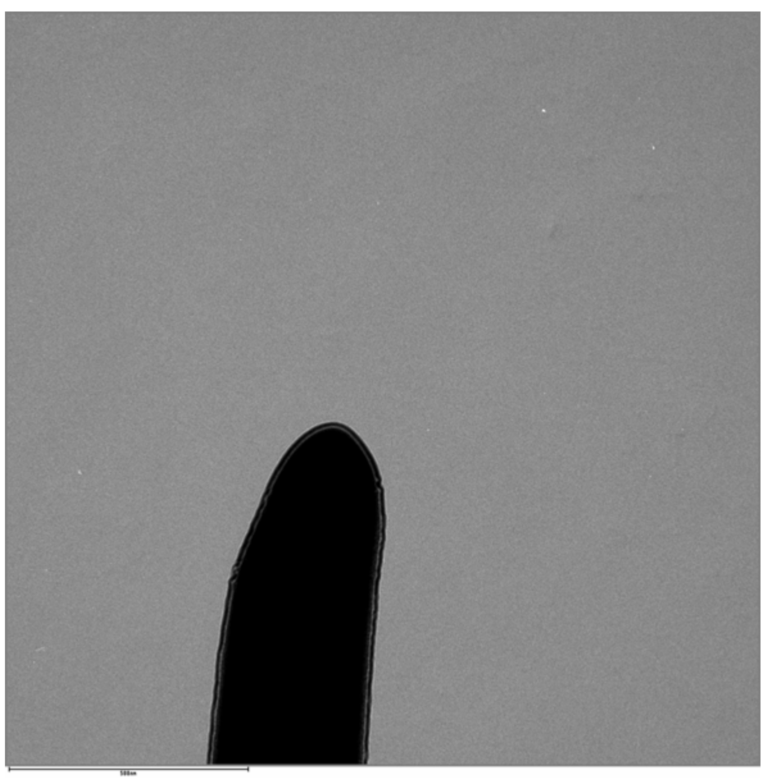
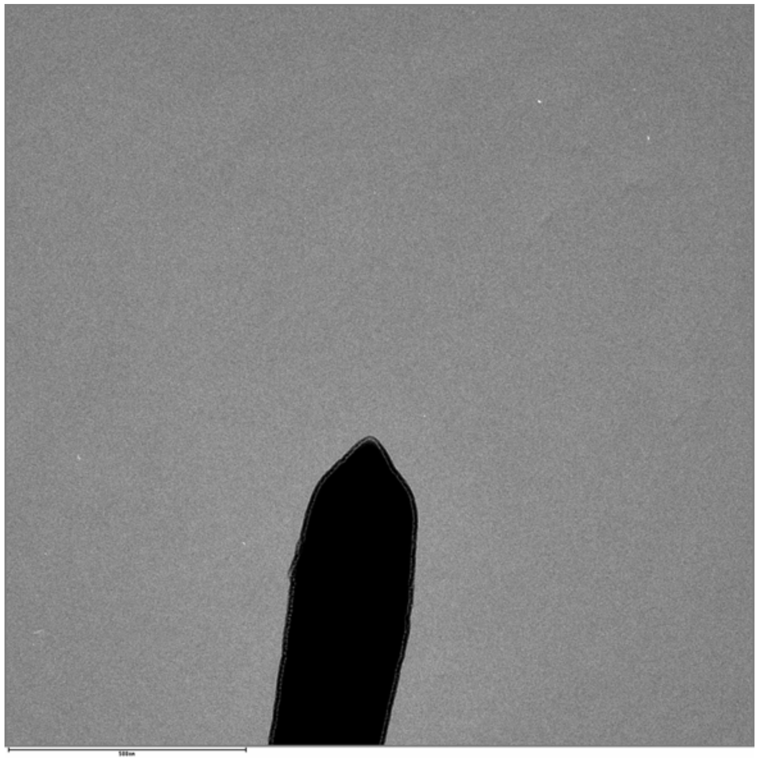


Figure A.2. 1300 V plasma, 50 V tip bias

Before



After

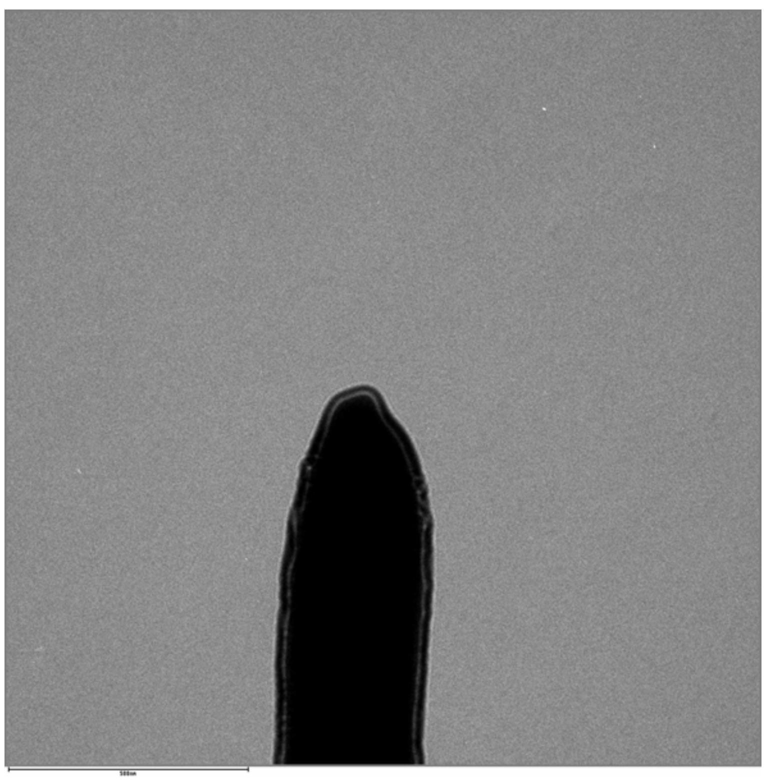


Figure A.3. 1400 V plasma, 150 V tip bias

Before

After

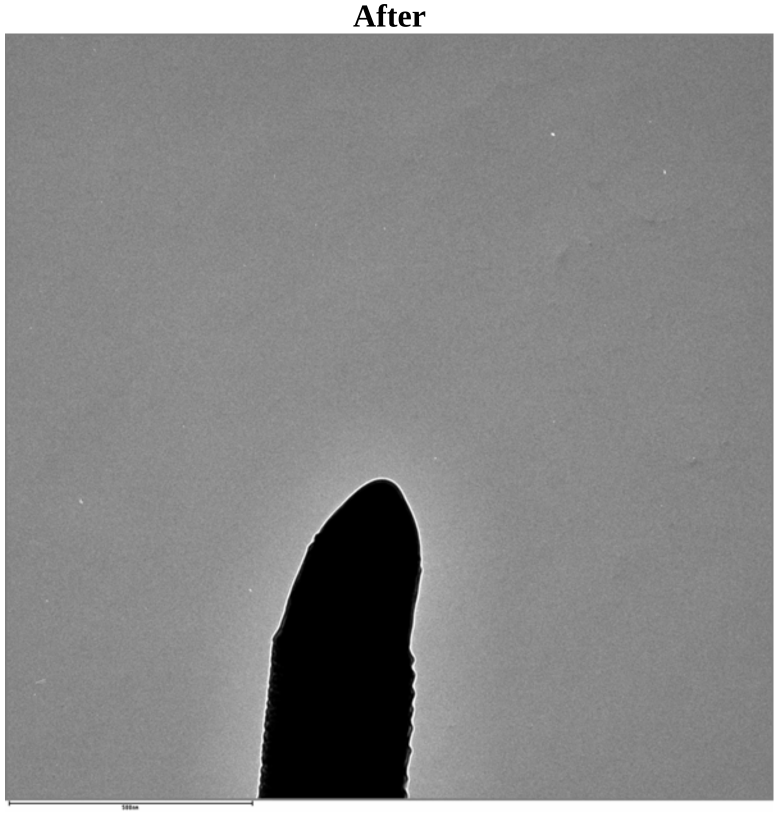


Figure A.4. 1450 V plasma, 200V tip bias