Experimental Investigation of Reproducible Electrochemical Etching Technique of Tungsten Probe at Domestic level for SPM

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Abstract- One of the great challenges of Scanning Tunneling Microscopy (STM) is the production of atomically sharp tips. Tungsten tips in Scanning Probe Microscope (SPM) and Atomic Force Microscope (AFM) are used instead of platinum and iridium (Pt/Ir) tips due to their high quality factor, mechanical stability and produced at low cost. In this work we carried out different experiments on 0.5 mm (0.02 inch) tungsten wire and obtained sharp and reproducible tungsten tips by simple electrochemical etching "Drop-Off" method and we have shown that tips reproduced have better properties i.e. tip diameter and cone angle than earlier. Moreover tips reproducibility is checked by selecting a sample or two from produced tips and experimental work is repeated to show whether the procedure is reproducible or not and as shown that tips production procedure is reproducible by analyzing them, so have best control on tip shape and optimized etching conditions are reported. Similarly tips are produced by varying different voltages and maintaining various concentration of solution constant i.e. 2 molar, 3 molar and 4 molar NaOH concentration of solution and comparison is made between different tips by measuring their cone angle and tip diameter from SEM and OM images. Cone angle and tip diameter are measured from SEM and OM images by using AutoCAD software whose procedure are shown below at experimental work. Finally we have produced atomically sharp tungsten tips by analyzing optical microscope (OM) images and scanning electron microscope (SEM) images and have recommended the optimum one to be used in SPM.

Keywords- Scanning Tunneling Microscopy, Scanning Probe Microscope, Tungsten Tips, Electrochemical Etching, Scanning Electron Microscope

I. INTRODUCTION

Scanning Probe Microscopy (SPM) is a branch of imaging technique that scan the surface with the varying sharp probe whose diameter vary from size of the atom up to 40 nm [1]. Here source is quite far away from the specimen. It works by scattering of light and electrons that produce image of a specimen. SPM technique broadly classified into Scanning Tunneling Microscopy (STM) and Atomic Force Microscopy (AFM) [2, 3].

In STM a tunneling current between atomically sharp probes is used for scanning the surface. STM consist of small sharp conducting tip that scans across the sample. Separation distance b/w tip and sample is close to 1 nm. At this small distance a quantum mechanical tunneling current can flow b/w atoms at tip and surface. The tip is attached to scanning device which move in X, Y, Z direction and scan surface. The magnitude of current b/w tip and sample is constantly measured. It is important to know that only electrically conductive sample can be examined with STM. STM works in two ways, scanning at constant height and scanning at constant current [4, 5].

In AFM a 100-200 micrometer cantilever with a sharp silicon nitride or silicon crystal tip is used for scanning a specimen [6]. AFM is used for measuring non conducting samples i.e. polymers and biological specimens with atomic resolution. AFM works on intermolecular forces (Vander walls or Electrostatic). Here no flow of current b/w tip and specimen occur [7]. Tip diameter ranges from 10-40 nm. It works in three mode contact mode, tapping mode and non-contact mode [KARTHIK SALVAM, www.vit.ac.in].

II. ELECTROCHEMICAL ETCHING

Electrochemical etching method is at present considered the fastest, cheapest, and most convenient and reliable method for fabricating metallic tips for scanning tunneling microscopy (STM) or atom probe field ion microscopy (AFM) [8]. Electrochemical etching is governed by various etching parameters and also depends on the operator's skills and experience. Many researchers have developed customized etching procedures and conditions to improve the stability and reliability, and to obtain the desired high-quality tip profiles. Among these customized techniques, the "drop-off" method is a well-known, robust technique that provides flexibility in terms of control in etching parameters and process. In the "drop-off" technique, electrochemical etching essentially results from the anodic dissolution of metallic wire in an aqueous electrolyte solution. The standard method involves dipping a tungsten wire into an electrolyte, biasing the tip [9, 10]. Etching then begins at the air/electrolyte reaction interface and continues into the immersed metallic wire. Usually, potassium hydroxide (KOH) or sodium hydroxide (NaOH) is used as electrolyte for tungsten wires; meanwhile, calcium chloride ($CaCl2$), sodium chloride (NaCl), or potassium chloride (KCl) is used for platinum-iridium wires. When a positive voltage is applied to the tungsten wire, etching occurred at the wire surface within the meniscus and below the nominal air/electrolyte interface. The basic electrochemical reactions occurring in this process are shown below:

Cathode: $2H2O + 2e \leftrightarrow H2\ (g) + 2OH$

Anode: $W + 8OH \rightarrow WO4^{2}+ 4H2O + 6e$

Overall: $W + 2OH + 2H2O \rightarrow WO4^{2}+ 3H2\ (g)$

Here in the electrochemical reaction, at the anode the tungsten wire is oxidized to form tungstate anions which are soluble in water, and thus the wire is partly dissolved. Similarly the capillary forces and surface tension yield the formation of a meniscus of solution around the tip wire when it is immersed into the electrolyte. The shape of the meniscus plays a very important role in determining the final shape of the tip as the etching rate at the top of the meniscus is a lot slower than at the bottom. Also the concentration of OH^- ions is lower near the top of the meniscus than in the solution overall. Thus, the etching process takes place at a slower rate at the top of the meniscus. Where etching takes place at a higher rate, tungsten anions are formed that will flow downwards. This hinders the electrochemical etching of the tungsten wire submerged below the meniscus. Thus, a necking phenomenon is observed in the meniscus where the etching rate is enhanced. At some point, this part of the wire becomes so thin its tensile strength cannot sustain the weight of the lower end of the wire; the latter breaks off and a sharp tip is left behind. This is commonly referred to as the “drop-off” method [11].

Furthermore, the soluble tungstate produced during the reaction flows towards the lower end of the tip wire, generating a dense viscous layer which prevents this region from being etched away. The reaction is also significantly enhanced as a result of the radial flow of solution from the cathode ring toward the anode tungsten wire. As in the above procedure, when the tips drop off, so the applied voltage is turned off as quickly as possible in order to avoid an increase in tip radius. Finally, the upper portion of the tungsten wire was used as the final product, i.e., the tungsten tip and in some cases both upper and lower portion as Tungsten (W) Tips., increasing the chance on a double tip[12].

So Here we will use electrochemical etching technique for the production of Tungsten (W) tips by supplying DC voltage from a DC power supply. A positive DC etching voltage was chosen to eliminate disruptive gas formation at the etching site [13]. The advantages of this method are short fabrication time, easy setup, and low investment and being controllable with high accuracy [10]. In conclusion, STM tips will be electrochemically etched using an in-house developed method which will offers excellent control over the tip’s shape and consistently produce ultra-sharp probes capable of sub-molecular/atomic resolution STM/AFM images.

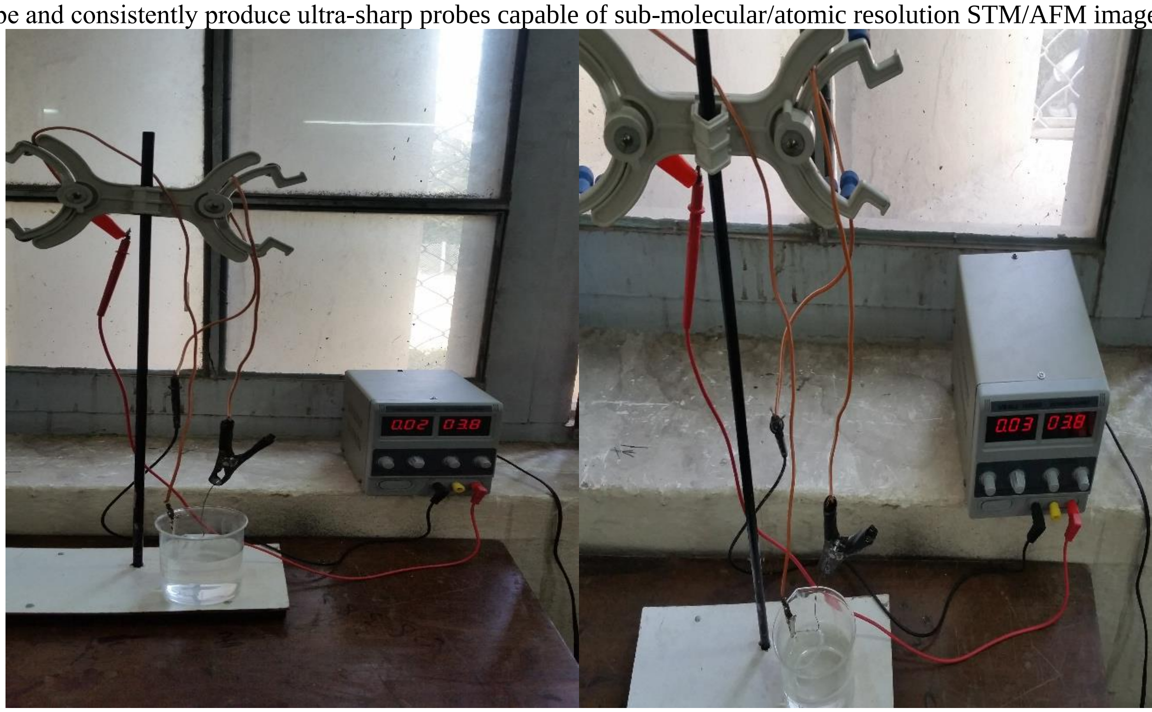


Figure 1: Schematic of the experimental setup for Electrochemical Etching Technique

III. EXPERIMENTAL WORK

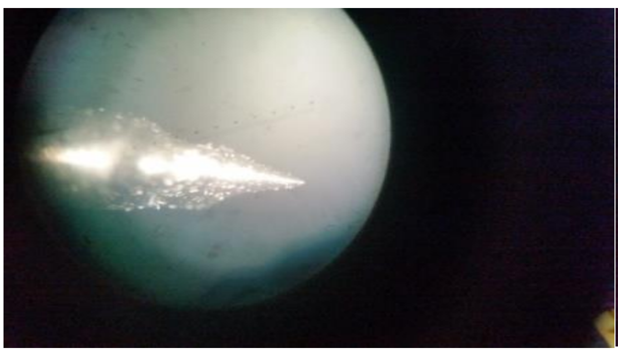
A. Electrochemical etching of 0.5 mm tungsten wire in 2 molar NaOH solution

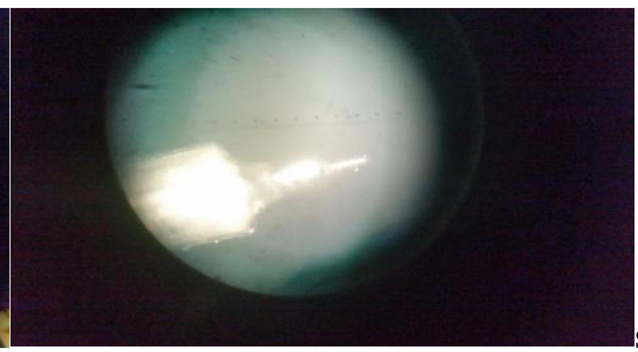
In electrochemical etching, 0.5 mm equivalent to 0.02 inch tungsten wire is dipped into 2 molar NaOH solution containing an electrode and applying a DC voltage, the process have dissolved the wire partly near the air-solution interface until a sharp tip have been formed. In 2 molar solution 16 grams of NaOH is dissolved in 200 ml of distilled water. The DC voltage is applied in such a way that the Tungsten wire (tip) is the anode and the submerged electrode is the cathode in this process. Experimental data for produced tips is given in following table.

Table 1: Experimental data for produced tips of 0.5 mm tungsten wire on Electrochemical Etching in 2 molar NaOH solution

S. No	V	Ti	Tf	A	Cone Angle	Tip Diameter (mm) ≈ Tip Diameter (nm)	
1	3.2	11:08	11:25	0.02	29°	0.000107	107
2	3.8	11:38	11:55	0.04	31°	0.000044	44
3	4.9	12:06	12:18	0.09	38°	0.000048	48
4	5.6	12:19	12:30	0.10	33°	0.000058	58
5	7.3	12:31	12:40	0.11	23°	0.000062	62
6	9.2	12:41	12:48	0.15	17°	0.000087	87
7	11.2	12:49	12:56	0.17	17°	0.000971	971

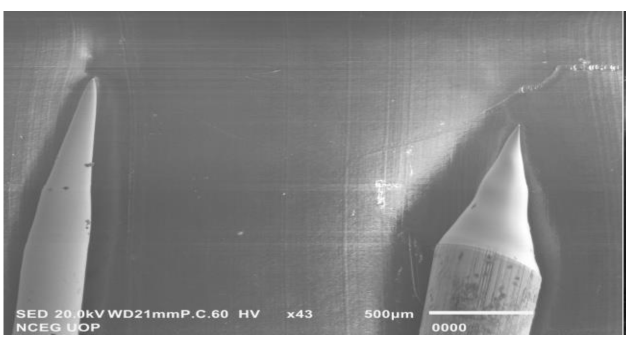
In above table, V is the reference voltage which is selected, Ti is start time of experiment, Tf is end time of experiment when etching completed and A is ampere drawn by supply when etching in progress. OM & SEM images of the produced tips are shown in following figure.

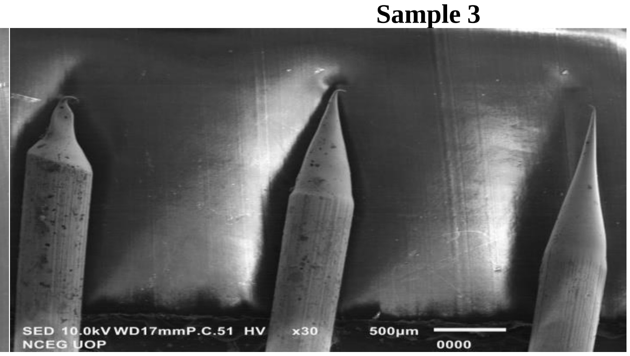




Sample 2

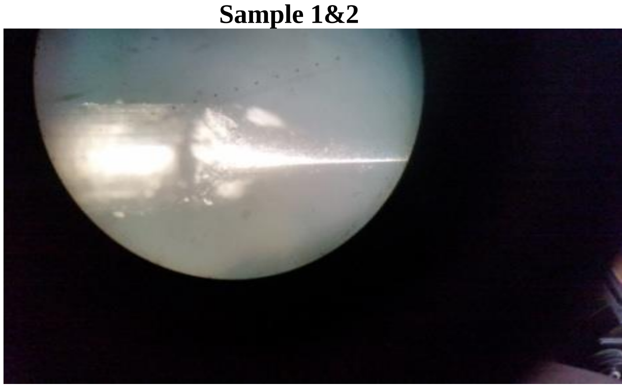
Sample 3

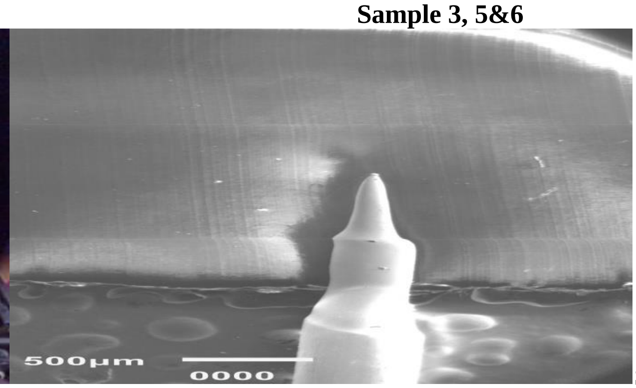




Sample 1&2

Sample 3, 5&6





Sample 4

Sample 7

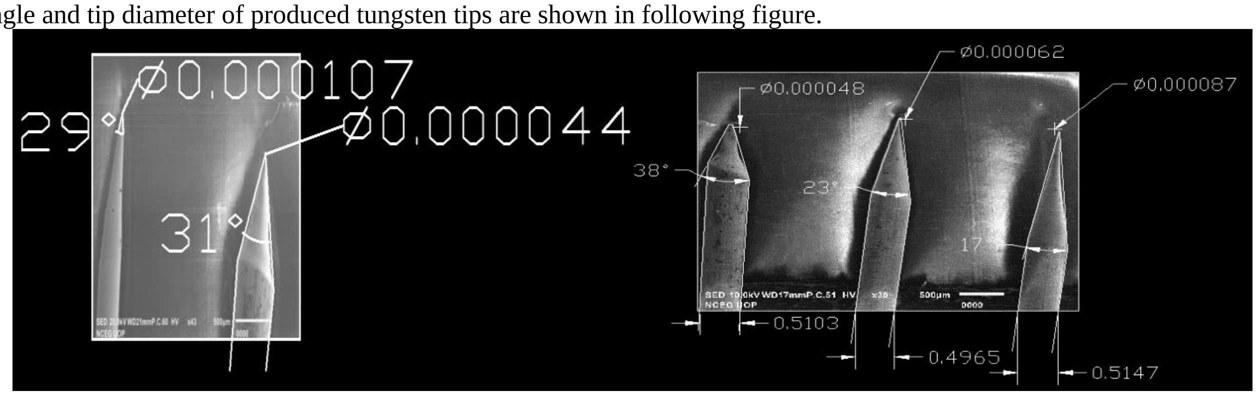
Figure 2: OM & SEM Images of tungsten tips prepared in 2 Molar NaOH solution (16g/200ml)

❖ Finding Cone Angle using AutoCAD

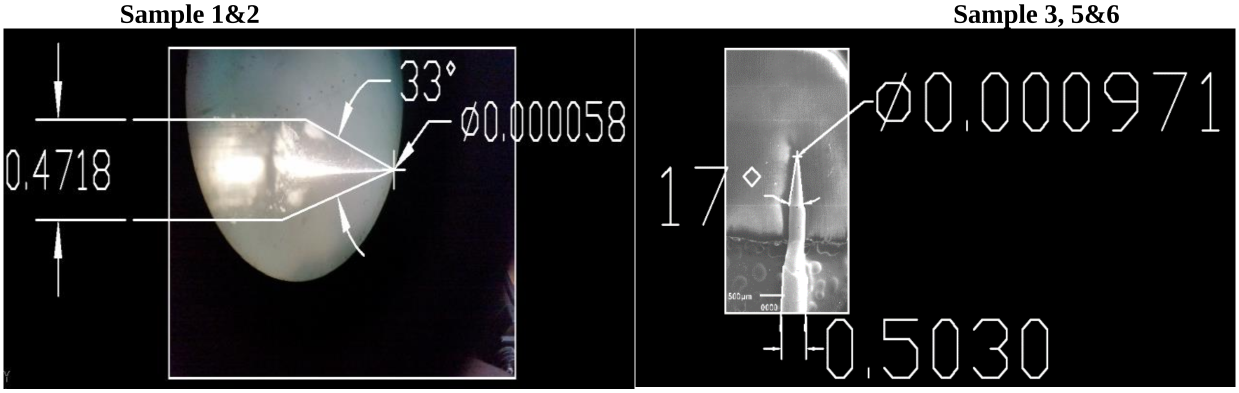
Cone angle and tip diameter of produced tungsten tips are find by using AutoCAD Software whose procedure are given below.

1. First insert the picture to AutoCAD software.
2. Then scale the picture by finding its scale factor as one of the dimension i.e. wire diameter is known to us which is 0.5 mm.
3. At last when the picture is scaled, so find cone angle and tip diameter by drawing imaginary lines and a circle at the tip.

Cone angle and tip diameter of produced tungsten tips are shown in following figure.



Sample 1&2 **Sample 3, 5&6**



Sample 4 **Sample 7**

Figure 3: SEM Images of tungsten tips showing cone angle and tip diameter measured using AutoCAD Software

From the finding data we suggest that sample 2 and sample 3 have best cone angle and tip diameter, so we will reproduce these two samples and will check their reproducibility which is one of our objective.

a. Reproducibility of sample 2 on Electrochemical Etching in 2 molar NaOH Solution

We have performed several experiments keeping the voltage of sample 2 i.e.3.8 v constant to check whether the sample we selected is reproducible or not. We have performed 7 experiments and taken samples. Experimental data for production of tips is given in following table.

Table 2: Experimental data showing reproducibility of sample 2 on Electrochemical Etching

S. No	V	Ti	Tf	A	Cone Angle	Tip Diameter (mm)	≈ Tip Diameter (nm)
1	3.8	10:34	10:54	0.02	17°	0.000108	108
2	3.8	11:04	11:25	0.03	35°	0.000018	18
3	3.8	11:47	12:08	0.03	38°	0.000016	16
4	3.8	12:17	12:39	0.04	58°	0.000014	14
5	3.8	12:52	1:12	0.04	61°	0.000049	49
6	3.8	9:49	10:12	0.02	44°	0.000037	37
7	3.8	10:08	10:20	0.04	63°	0.000065	65

SEM images of the produced tips are shown in following figure.

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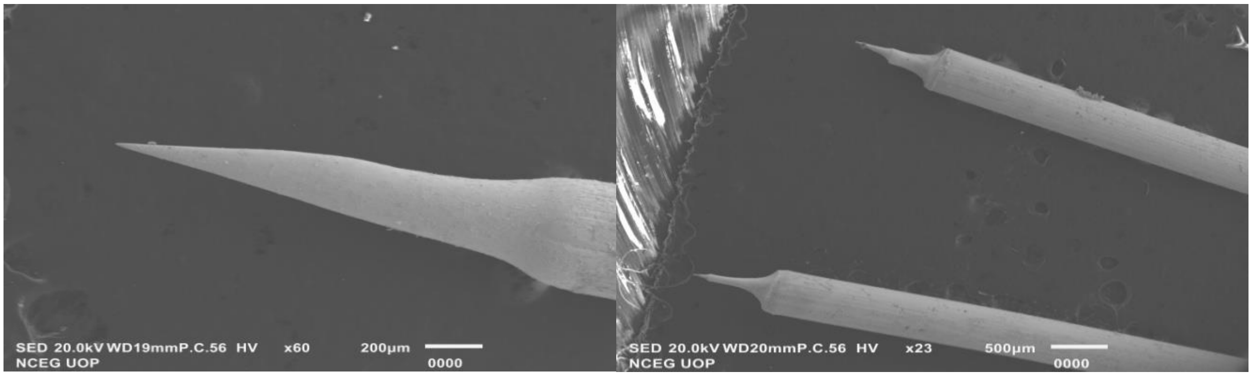
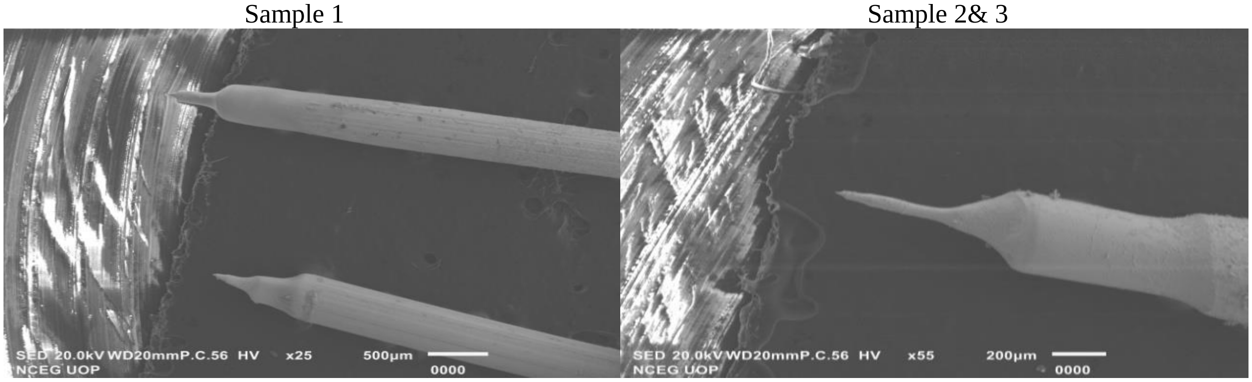
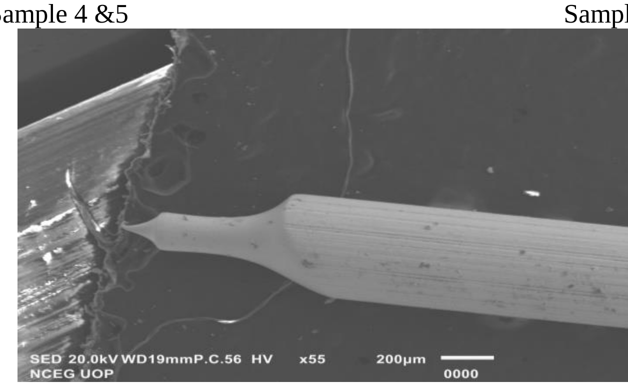


Sample 1 Sample 2& 3



Sample 4 &5 Sample 6

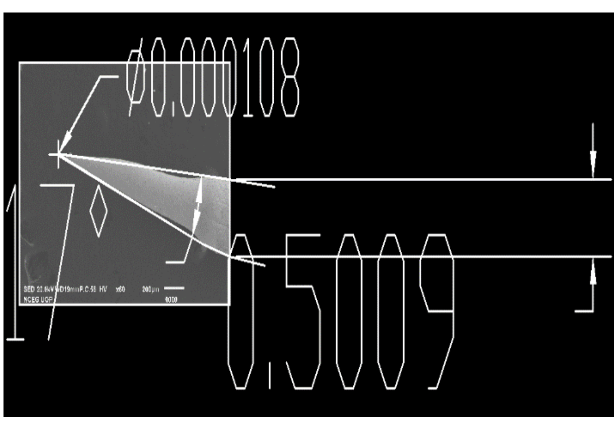


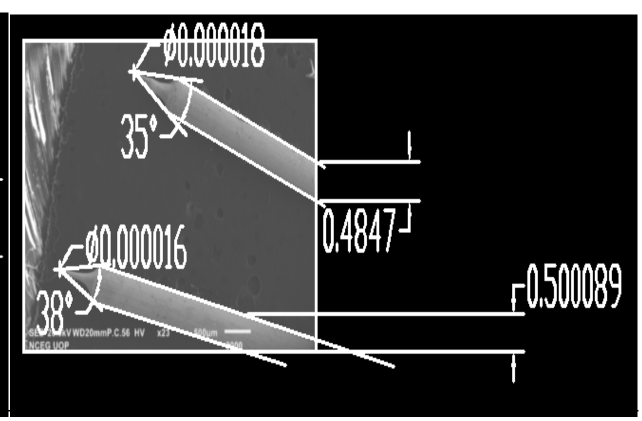
Sample 7

Figure 4: SEM Images showing Sample 2 reproducibility in 2M NaOH solution with an applied voltage of 3.8V

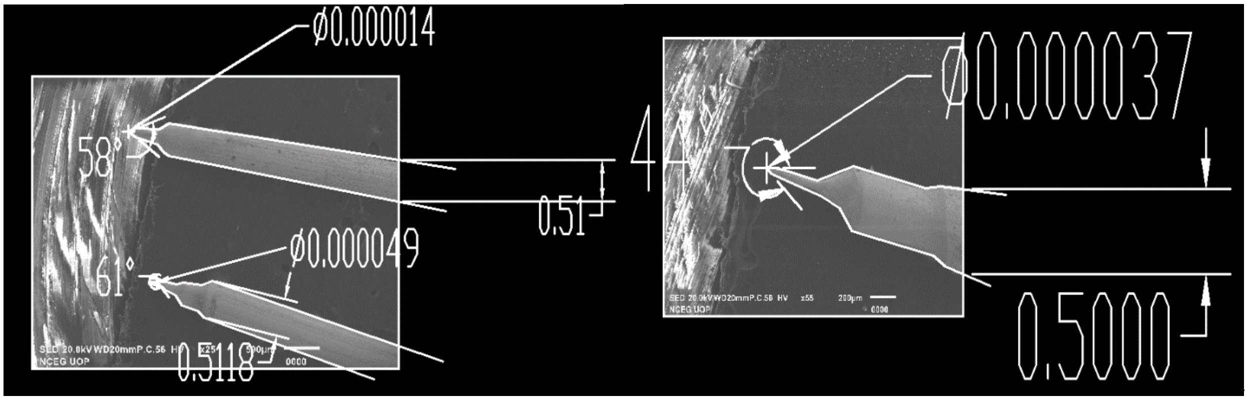
❖ **Finding Cone Angle using AutoCAD**

Cone angle and tip diameter of tungsten tips reproduced on 3.8 V are find by using AutoCAD Software whose details are shown in following figure.

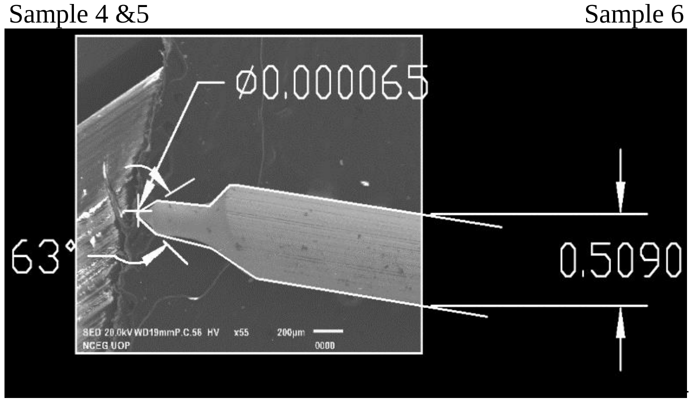




Sample 1 Sample 2& 3



Sample 4 &5 Sample 6



Sample 7

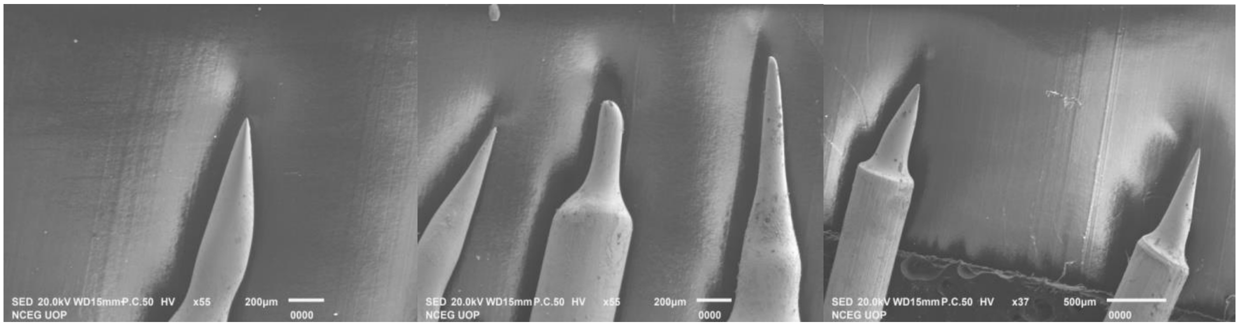
Figure 5: SEM Images of tungsten tips showing cone angle and tip diameter measured using AutoCAD Software

b. Reproducibility of sample 3 on Electrochemical Etching in 2 molar NaOH Solution

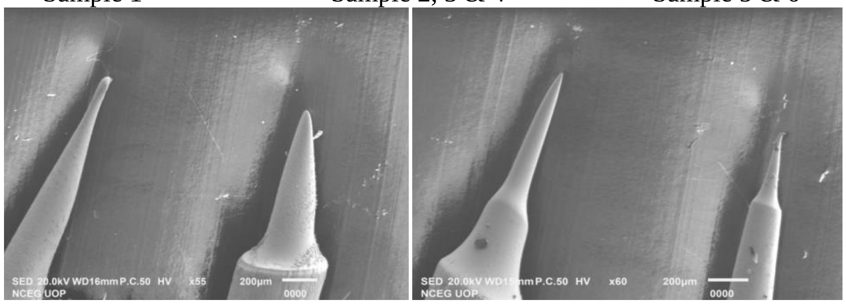
We have performed several experiments keeping the voltage of sample 3 i.e. 4.9 V constant to check whether the sample we selected is reproducible or not. We have performed 10 experiments and taken samples. Experimental data and SEM images for produced tips are given below.

Table 3: Experimental data showing reproducibility of sample 3 on Electrochemical Etching

S. No	V	Ti	Tf	A	Cone Angle	Tip Diameter (mm) ≈ Tip Diameter (nm)	
1	4.9	10:47	10:58	0.07	24°	0.000047	47
2	4.9	11:00	11:14	0.03	19°	0.000038	38
3	4.9	11:21	11:37	0.02	13°	0.003994	3994
4	4.9	11:45	11:59	0.04	16°	0.000276	276
5	4.9	12:28	12:43	0.03	38°	0.000040	40
6	4.9	10:00	10:13	0.02	25°	0.000044	44
7	4.9	10:18	10:31	0.02	13°	0.002859	2859
8	4.9	10:38	10:51	0.02	23°	0.000083	83
9	4.9	10:59	11:14	0.02	22°	0.000045	45
10	4.9	11:22	11:37	0.04	20°	0.000040	40



Sample 1 Sample 2, 3 & 4 Sample 5 & 6

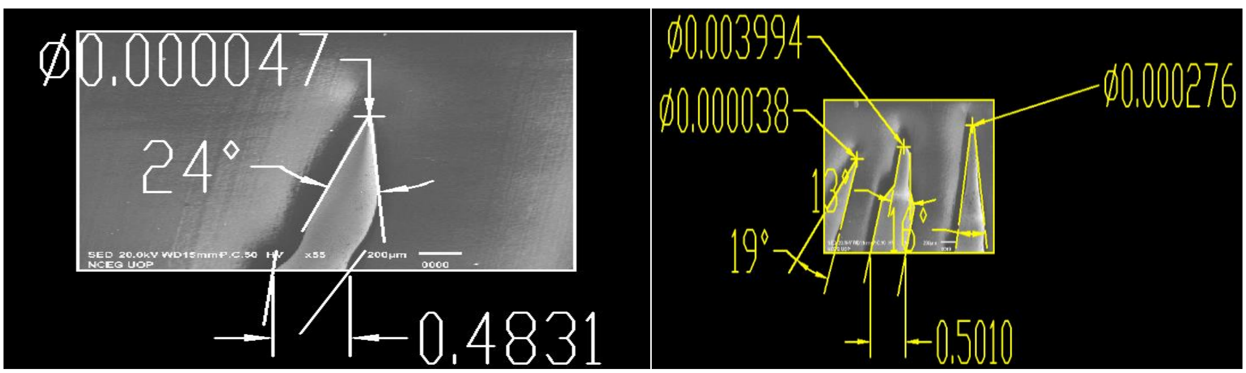


Sample 7 & 8 Sample 9 & 10

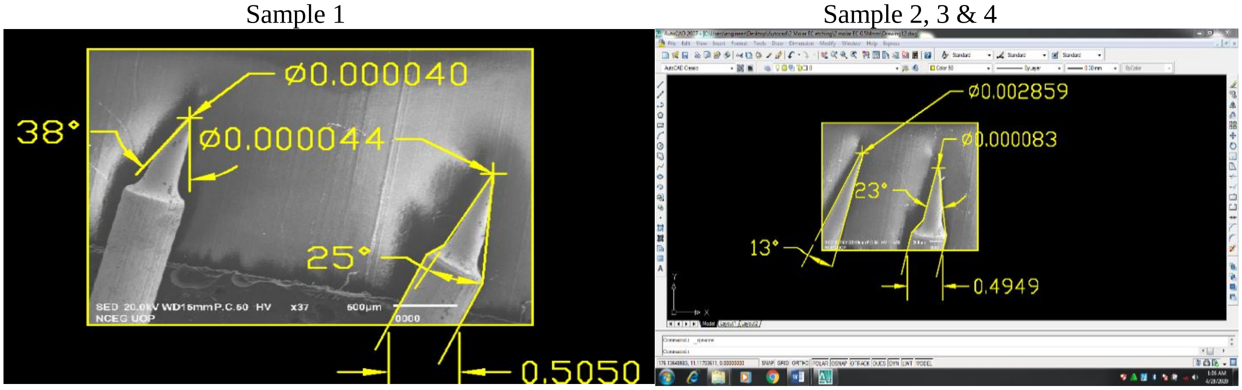
Figure 6: SEM Images showing Sample 3 reproducibility in 2M NaOH solution with an applied voltage of 4.9V

- **Finding Cone Angle using AutoCAD**

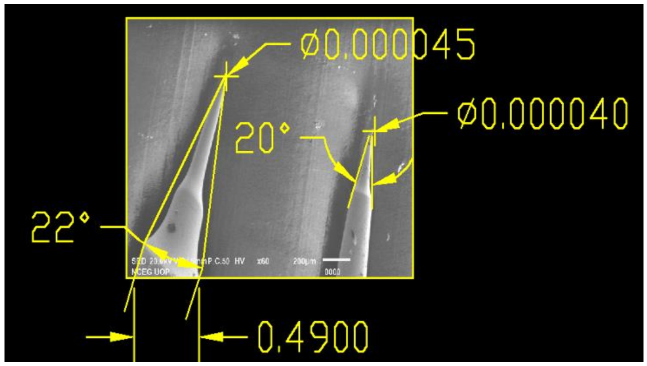
Cone angle and tip diameter of tungsten tips reproduced on 4.9 V are find by using AutoCAD Software whose details are shown in following figure.



Sample 1 Sample 2, 3 & 4



Sample 5 & 6 Sample 7 & 8



Sample 9 & 10

Figure 7: SEM Images of tungsten tips showing cone angle and tip diameter measured using AutoCAD Software

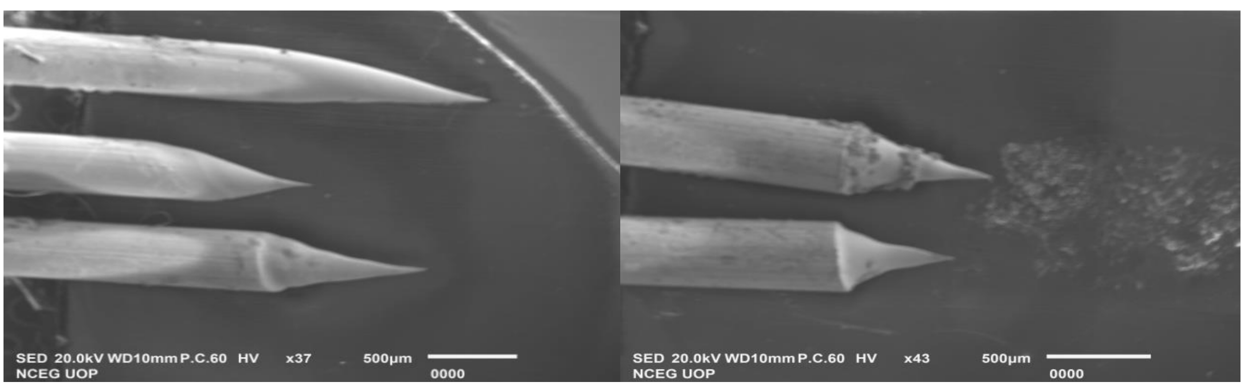
B. Electrochemical etching of 0.5 mm tungsten wire in 3 molar NaOH solution

In Electrochemical etching, 0.5 mm (0.02 inch) tungsten wire is dipped into 3 molar NaOH solution containing an electrode and applying a voltage, the process have dissolved the wire partly near the air-solution interface until a sharp tip have been formed. In 3 molar solution 24 grams of NaOH is dissolved in 200 ml of distilled water. The DC voltage is applied in such a way that the Tungsten wire (tip) is the anode and the submerged electrode is the cathode in this electrochemical etching process. Experimental data for produced tips is given in following table.

Table 4: Experimental data for produced tungsten tips of 0.5 mm on Electrochemical Etching in 3 molar NaOH solution

S. No	V	Ti	Tf	A	Cone Angle	Tip Diameter (mm) ≈ Tip Diameter (nm)	
1	3.8	3:36	3:44	0.17	19°	0.000019	19
2	5.6	3:48	3:55	0.20	31°	0.000017	17
3	7.3	3:58	4:03	0.22	26°	0.000012	12
4	9.2	4:05	4:08	0.37	36°	0.000017	17
5	11.2	4:11	4:14	0.39	41°	0.000012	12

SEM images of the samples obtained are given below.

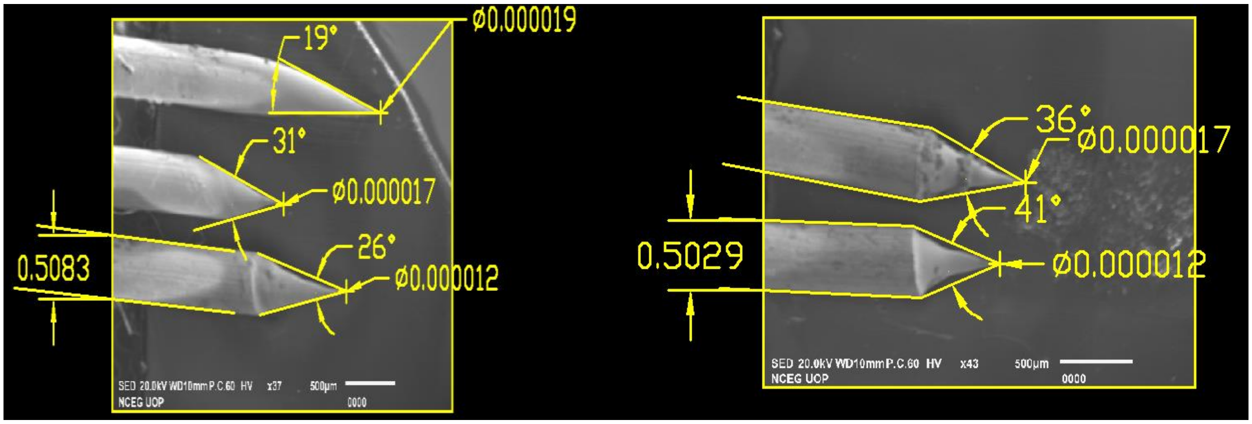


Sample 1, 2 & 3 Sample 4 & 5

Figure 8: SEM Images of tungsten tips prepared in 3 Molar NaOH solution (24g/200ml)

- **Finding Cone Angle using AutoCAD**

Cone angle and tip diameter of tungsten tips produced are find by using AutoCAD Software whose details are shown in following figure.



Sample 1, 2 & 3 Sample 4 & 5

Figure 9: SEM Images of tungsten tips showing cone angle and tip diameter measured using AutoCAD Software

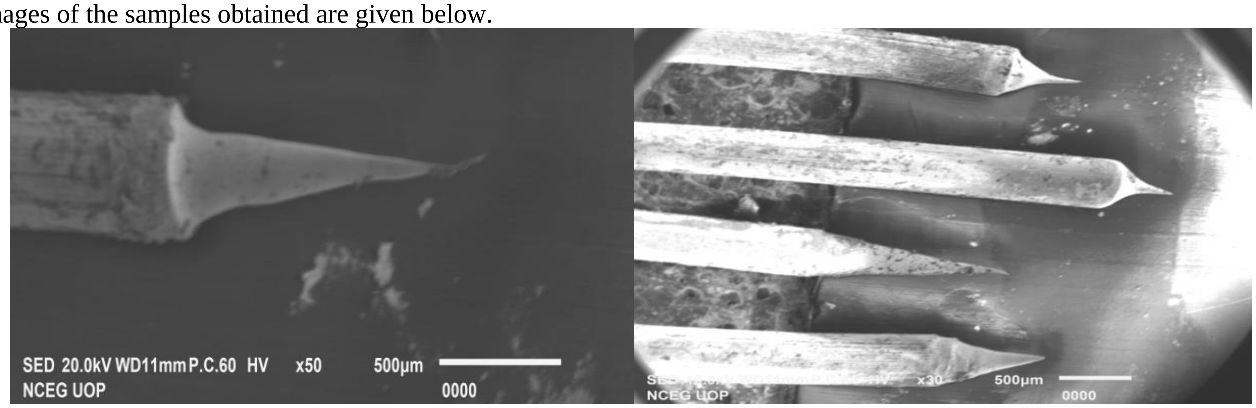
C. Electrochemical etching of 0.5 mm tungsten wire in 4 molar NaOH solution

In Electrochemical etching, 0.5 mm (0.02 inch**)** tungsten wire is dipped into 4 molar NaOH solution containing an electrode and applying a voltage, the process have dissolved the wire partly near the air-solution interface until a sharp tip have been formed. In 4 molar solution 32 grams of NaOH is dissolved in 200 ml of distilled water. The DC voltage is applied in such a way that the Tungsten wire (tip) is the anode and the submerged electrode is the cathode in this electrochemical etching process. Experimental data for produced tips is given in following table.

Table 5: Experimental data for produced tungsten tips of 0.5 mm on Electrochemical Etching in 4 molar NaOH solution

S. No	V	Ti	Tf	A	Cone Angle	Tip Diameter (mm) ≈ Tip Diameter (nm)	
1	3.8	10:42	10:45	0.25	23°	0.000033	33
2	5.6	10:48	10:51	0.28	43°	0.000021	21
3	7.3	11:00	11:03	0.33	50°	0.000018	18
4	9.2	11:07	11:09	0.72	17°	0.000029	29
5	11.2	11:13	11:14	0.84	33°	0.000024	24

SEM images of the samples obtained are given below.

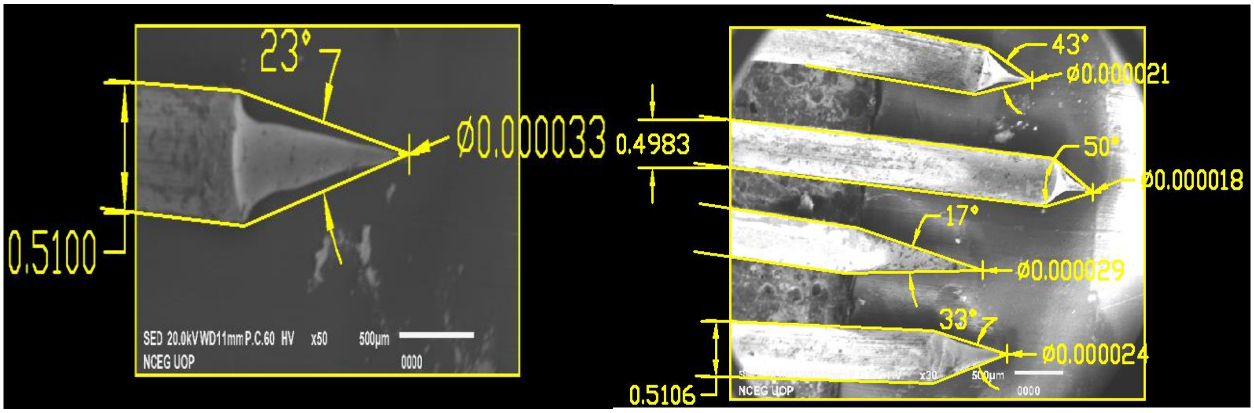


Sample 1 Sample 2, 3, 4 & 5

Figure 10: SEM Images of tungsten tips prepared in 4 Molar NaOH solution (32g/200ml)

- **Finding Cone Angle using AutoCAD**

Cone angle and tip diameter of tungsten tips produced are find by using AutoCAD Software whose details are shown in following picture.



Sample 1 Sample 2, 3, 4 & 5

Figure 11: SEM Images of tungsten tips showing cone angle and tip diameter measured using AutoCAD Software

IV. RESULTS AND FINDINGS

There is a vast amount of reported techniques to make SPM tips, and these techniques do yield really sharp tips. But, reproducibility and controlling tip shape becomes an issue. Recent efforts in stabilizing tip fabrication came in the form of simple electrochemical "drop-off" [14] method. This simple "drop-off" method produces highly reproducible tips with controllable cone angles and tip diameter. We have produced 34 samples on this technique by changing different variables i.e. voltage and concentration of solution. As it is clear from the literature, that tips with a cone angle of ~15° or greater [13] and tip diameter up to 40 nm [1] gave excellent STM images. Also shown that cone angles around 20º were more typical [13]. So tip diameter and cone angles of all samples were calculated and our results shows that about ~80% of tips were produced having cone angle greater than 20° and tip diameter less than 40 nm as shown in experimental data.

Instead of it, we have also checked reproducibility of some samples i.e. sample 2 and 3 reproducibility is checked in 2 molar solution and it can be seen from the results shown in experimental work in table 2 and 3 that our procedure is reproducible and we are able to reproduce the required Tungsten sharp probes for STM at domestic level easily by keeping the reference voltage and experimental condition constant. Note that the largest cone angle which is produced is 63° as shown in Sample 7 in Reproducibility of sample 2 i.e. figure 5.

Moreover we explored numerous parameters effecting the etching process. Etching voltage was optimized so that it was high enough to create drop-off, but not very high to produce blobbing at tip apex. For all the experiments we performed, we kept some specific values of etching voltage which are 3.2, 5.6, 7.3, 9.2 and 11.2V by keeping constant concentration of solution i.e. 2M, 3M and 4M. Some of the important relations and results which we explored from our experimental work are given below.

A. Impact of Voltage and Concentration of Solution on Tip Diameter

As it is cleared from the following figure 12, that in 2 molar concentration solution as the etching voltage is increases, so tip diameter increases. Similarly in 3 molar concentration solution as the etching voltage increases so tip diameter decreases and in 4 molar concentration solution as the etching voltage increases, so tip diameter decreases up to 7.3V and then increases again. Moreover best tip diameter which is obtained is 12 nm from 3 molar concentration solution on 7.3V and 11.2V and collectively also best tip diameter samples are produced on 3 molar solution. Following table and figure shows impact of voltage and concentration of solution on tip diameter.

Table 6: Experimental data for tip diameter produced in 2M, 3M and 4M NaOH solution on Electrochemical Etching

S. No	V	Tip Diameter (2M)	Tip Diameter (3M)	Tip Diameter (4M)
1	3.8	44	19	33
2	5.6	58	17	21
3	7.3	62	12	18
4	9.2	87	17	29
5	11.2	-	12	24

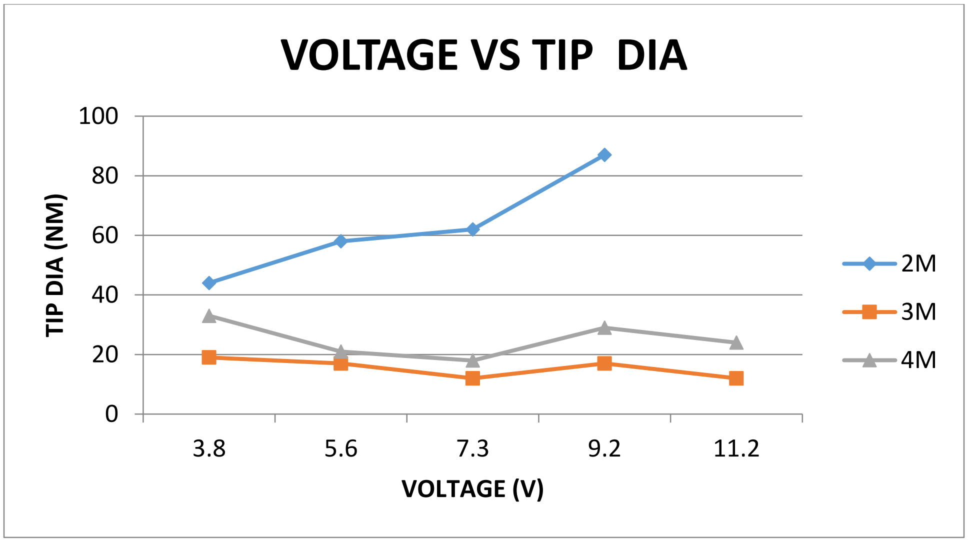


Figure 12: Figure showing Impact of Voltage and Concentration of Solution on Tip Diameter

B. Impact of Voltage and Concentration of Solution on etching time

As it is cleared from the following table 7 & figure 13, that in all 2 molar, 3 molar and 4 molar concentration solution as the etching voltage is increases, so etching time decreases means that there is inverse relationship between applied voltages and etching time. Moreover best etching time which is obtained is 5 minute in which 12 nm tip diameter is produced in 3 molar concentration solution. Following table and figure shows impact of voltage and concentration of solution on etching time.

Table 7: Experimental data for etching time produced in 2M, 3M and 4M NaOH solution on Electrochemical Etching

S. No	V	ΔT (2M)	ΔT (3M)	ΔT (4M)
1	3.8	17	8	3
2	5.6	11	7	3
3	7.3	9	5	3
4	9.2	8	3	2
5	11.2	7	3	1

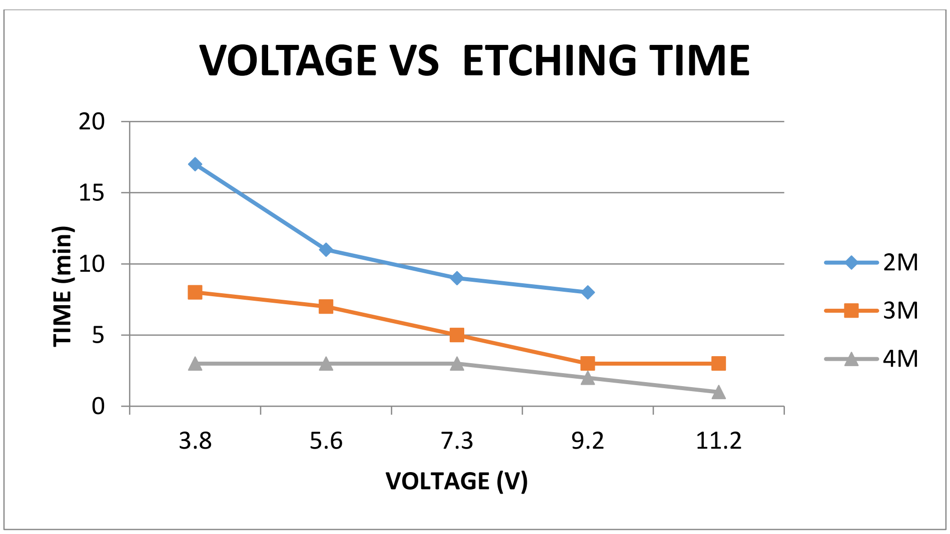


Figure 13: Figure showing Impact of Voltage and Concentration of Solution on Etching Time

C. Impact of Voltage and Concentration of Solution on Cone Angle

As it is cleared from the following table 8 & figure 14, that in 2 molar concentration solution as the etching voltage is increases, so cone angle decreases. Similarly in 3 molar concentration solution as the etching voltage is increases, so cone angle also increases and in 4 molar solution, cone angle increases up to 7.3V and then decreases again. Best cone angle which is obtained from this result is 50° on 7.3V in 4 molar concentration solution and on which tip diameter is also applicable mean equal to 18 nm and etching time is 3 minute. Following table and figure shows impact of voltage and concentration of solution on etching time.

Table 8: Experimental data for Cone Angle produced in 2M, 3M and 4M NaOH solution on Electrochemical Etching

S. No	V	CONE ANGLE (2M)	CONE ANGLE (3M)	CONE ANGLE (4M)
1	3.8	31°	19°	23°
2	5.6	33°	31°	43°
3	7.3	23°	26°	50°
4	9.2	17°	36°	17°
5	11.2	17°	41°	33°

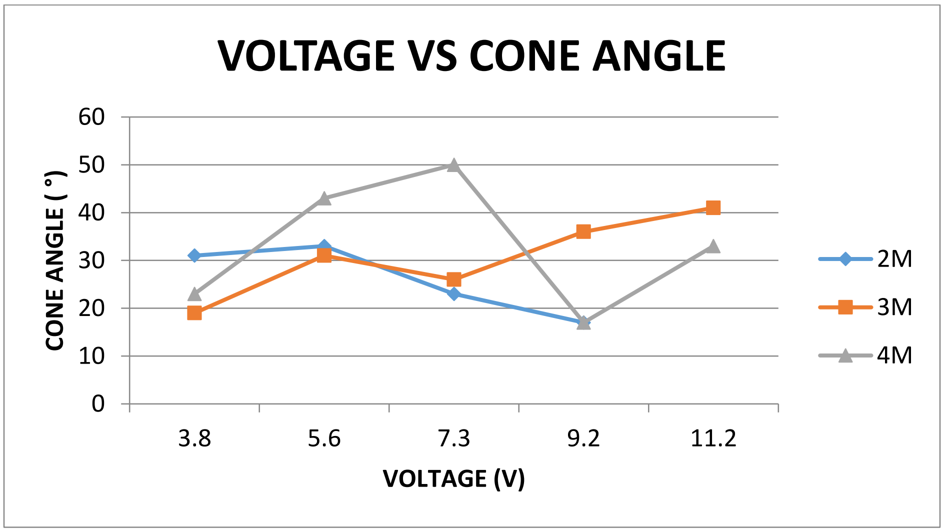


Figure 14: Figure showing Impact of Voltage and Concentration of Solution on Cone Angle

V. CONCLUSIONS

Extremely sharp tips with low aspect ratio are vital for scanning tunneling microscopy, and eventually atomically precise manufacturing [15]. At the conclusion of described "drop-off" method, it produced highly reproducible tips with controllable cone angles. Tip diameter less than 40 nm with ~80% success rate was obtained. High NaOH concentration and applied voltage was found to create turbulent bubbles on the cathode side, therefore, NaOH concentration was kept to an adequate level where reaction rate was not violent. As concentration of solution and applied voltage increases so reaction rate become faster and it takes much less time to complete the etching. Finally we says, that electrochemical etching is governed by various etching parameters and also depends on the operator's skills and experience. Overall, in this work, we performed a complete study to produce tips reproducibly with control on tip shape, and optimized etching conditions reported in literature. In conclusion, we have developed a simple procedure for the preparation of very sharp tungsten tips with tip diameter of 12 nm. This method produces tips in a very controlled and reproducible manner which are useful for STM and other probe applications.

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