DEVELOPMENT OF ATOMIC FORCE MICROSCOPE

A thesis submitted towards partial fulfillment of

BS-MS Dual Degree Programme

By

VIBHAM SHUKLA

Under the guidance of

Dr. SHIVPRASAD PATIL

Assistant Professor

DEPARTMENT OF PHYSICS

IISER PUNE

INDIAN INSTITUTE OF SCIENCE EDUCATION AND RESEARCH

PUNE

Certificate

This is to certify that this thesis entitled ''DEVELOPMENT OF ATOMIC FORCE MICROSCOPE'' submitted towards the partial fulfillment of the BS-MS dual degree programme at the Indian Institute of Science Education and Research Pune represents original research carried out by ''VIBHAM SHUKLA'' at ''INDIAN INSTITUTE OF SCIENCE EDUCATION AND RESEARCH, PUNE'', under the supervision of ''Dr. SHIVPRASAD PATIL'' during the academic year 2011-2012.

Student	Supervisor
VIBHAM SHUKLA	Dr. SHIVPRASAD PATIL

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Abstract

Since AFM was developed in 1986, much work has been done to enhance its resolution. In recent years Tuning forks are being used as a cantilever. Tuning fork as a tip-sample separation detector provides excellent stability and low thermal noise while measuring forces in pN range. A piezoelectric quartz tuning fork allows tip-sample separation detection without use of any optics.

A tuning fork based dynamic Atomic Force Microscope for force measurements have been developed. An X-Y-Z nano-positioner for sample coarse and fine approach has been developed. Current measurements have been done to test the overall stability of the set-up and tip-sample junction. The typical amplitude-distance curves are recorded for on-resonance operation with a tungsten tip mounted on micro-machined quartz tuning forks commercially available in the market. Under the on resonance condition, interaction force changes amplitude, frequency and phase of the tuning fork. A servo controller for the AFM and its automation using LABVIEW is developed. Once we are ready with the LabVIEW programme for scanning, we want to use this AFM to image in liquid.

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Chapter 1

Introduction

In order to study atomic scale processes, you need an instrument which can study and work at atomic scale. Physicist Richard Feynman said in his famous lecture in 1959: "If you want to make atomic-level manipulations, first you must be able to see what's going on.'' The Optical microscopes are limited by the diffraction limit of light, and hence have a resolution limit of $\lambda/2$ (~ 150-400 nm for visible radiation). Electron beam was used to overcome this limit, as we can control wavelength of electron beam by controlling its kinetic energy. Max Knoll and Ernst Ruska invented Transmission Electron Microscope (TEM) in 1931, and developed Scanning Electron Microscope (SEM) in 1942. Though SEM achieved success in studying samples at nano level, the most optimized resolution that we could get from SEM was few nm. This was far from imaging an atom. Another disadvantage of these microscopes was use of high energy beam for high resolution imaging, which can be destructive while studying soft biological samples. Müller developed a Field Ion Microscope (FIM) in 1951, first microscope to resolve atoms. The need of forming a sharp tip of the sample surfaces restricted the class of samples that could be studied using FIM. Then the Scanning Probe Microscope (SPM) technique was invented. The two important techniques of SPM are Scanning Tunneling Microscope (STM) and Atomic Force Microscope (AFM).

The history

STM was first developed by Binnig and Rohrer in1982 in IBM, Zurich [1] in which instead of using beam they used sharp tip to scan the sample surface. This was awarded with the Nobel Prize in 1986. STM works on the principle of quantum tunneling. A bias is maintained between the tip and the sample, when the tip comes close to the sample surface (~ nm), electrons tunnel between the surface of the sample and the tip, producing a current which strongly depends on the tip-sample separation. The sample surface can be scanned by two methods
(i). Maintaining constant current (ii). Maintaining constant separation. A feedback controller is used to maintain the separation/current constant and the tip is scanned over the surface to generate the topography. Since the current depends exponentially on the distance between the tip and the sample surface, it gives very good vertical resolution and lateral resolution depends on the sharpness of the tip.

Although in STM, we get atomic resolution of the sample surface morphology, it has a limitation; it works only for conducting and semi conducting samples. This limits the materials which can be imaged using STM and led to the development of Atomic Force Microscope in 1986, by Binnig, Quate and Gerber [2].

AFM works on the principle of atomic interaction between two surfaces. It measures the interaction force between the tip attached to the weak cantilever and the sample by measuring the deflection in the cantilever. Hence, can be used to study both conductors and insulators at atomic level.

In the first AFM, developed by Binnig, Quate and Gerber, they used a diamond tip attached to a gold cantilever, and used STM to measure the deflection of the cantilever [2]. Later, many methods came in existence to measure the deflection including fiber optic interferometer and optical deflection detection. Binning, Quate and Gerber anticipated the atomic resolution with the AFM from the beginning (1986) [14]. Giessibl in 1995, first resolved the sample surface Si (111)-(7 X 7) with atomic resolution [3]. Initially, AFM worked on contact mode only.

Although contact mode operation proved successful, the constant downward force often damages the sample surfaces. Also, soft or biological samples like cells and DNA need to be placed on a substrate for imaging surfaces [14]. These complications led to development of Tapping and Non-Contact modes.

Much work has been done to further enhance the imaging by AFM. Research has been done on the shape and size of the cantilever and material of the tip. Among the latest developments, quartz tuning fork is being used as a cantilever for force measurement. Tuning fork, because of its high stiffness, the piezoelectric property of the quartz crystal, and large quality factor resulting in high sensitivity is proving to be a better alternative. Giessibl in 1996 pioneered the use of tuning fork in qPlus configuration (mechanical excitation of tuning fork) in AFM and has been able to image atomic orbital of Si atoms [12].

AFM is not only an instrument for imaging; it is also being used in several other experiments by researchers. Researchers are using it not only for generating topography of the sample surfaces, but a variety of other experiments are being done using AFM. AFM has huge implications in biology, and has been used to generate topography of many biological macromolecules, cells, DNA, protein. Protein pulling experiments are also being done using AFM. AFM can also be used to measure physical properties like elasticity, adhesion, hardness, friction and chemical functionality. AFM has been used to perform liquid confinement experiments.

We have developed an X-Y-Z nano-positioner and have performed current measurements to test the overall stability of the set-up and sample-tip junction. The same setup is used for the tuning fork based AFM developed by us in the lab. We have also fabricated atomically sharp tungsten tip by electrochemical etching. Typical amplitude-distance curves have been recorded for on-resonance operation with the tungsten tip mounted on micro-machined quartz tuning forks commercially available in the market. The servo controller for the AFM and the approach mechanism has been fully automated using LABVIEW.

Chapter 2

Theory

In Scanning Probe Microscope (SPM), a sharp probe scans over a surface whilst monitoring some interaction between the probe and the surface. They give resolution of the order of an nm. In principle, any quantity that varies with the probe-sample separation can be used for imaging in Probe Microscope.

The two most important techniques of SPM are

- Scanning Tunneling Microscope (STM)
- Atomic Force Microscope (AFM)

Scanning Tunneling Microscope (STM)

STM works by scanning a sharp metal probe over the sample surface. A bias is maintained between the tip and the sample, when the tip comes close to the sample surface (~ nm), electrons tunnel between tip and the sample. We can image the sample surface with atomic level resolution.

STM works on the principle of quantum tunneling due to which we can see the sample surface. Another principle is piezoelectric effect, which allows us to scan over the sample surface precisely. A feedback controller maintains the separation constant and the tip is scanned over the surface to generate the topography.

Figure 1 shows schematic of STM [15].

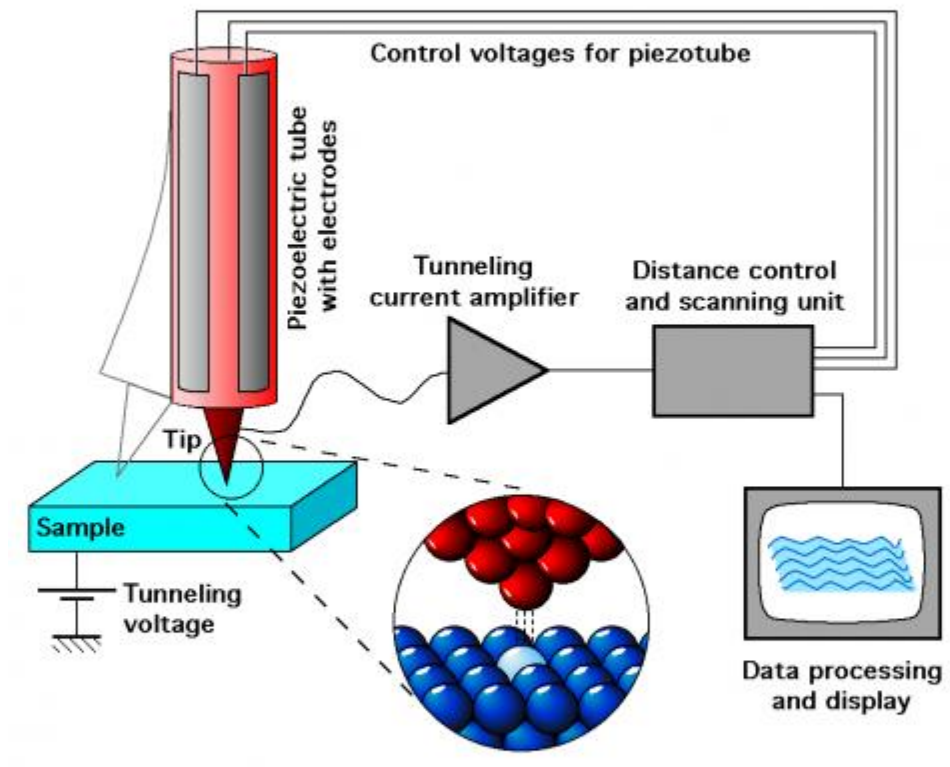


Figure 1: Schematic of STM

Quantum Tunneling

A tunneling current occurs when an electron tunnels through a potential barrier which classically they shouldn't be able to go through.

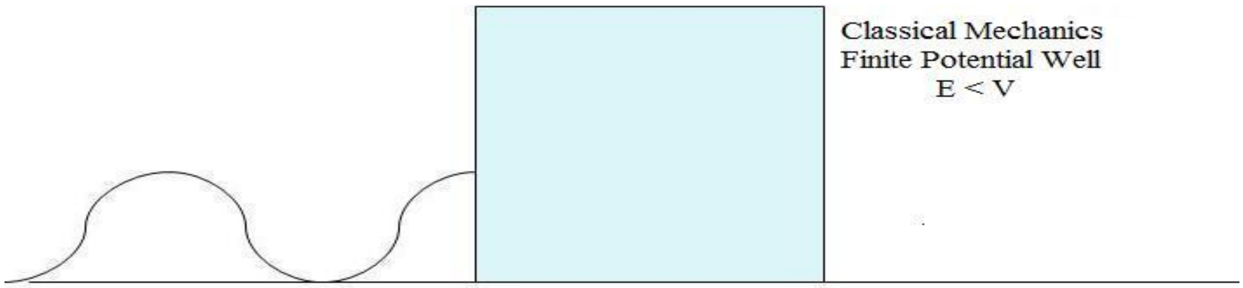


Figure 2: Classical Potential Well

Classically, when an object hits a potential barrier for which it doesn't have enough energy to pass, it will never go through that potential barrier, it always bounces back [5].

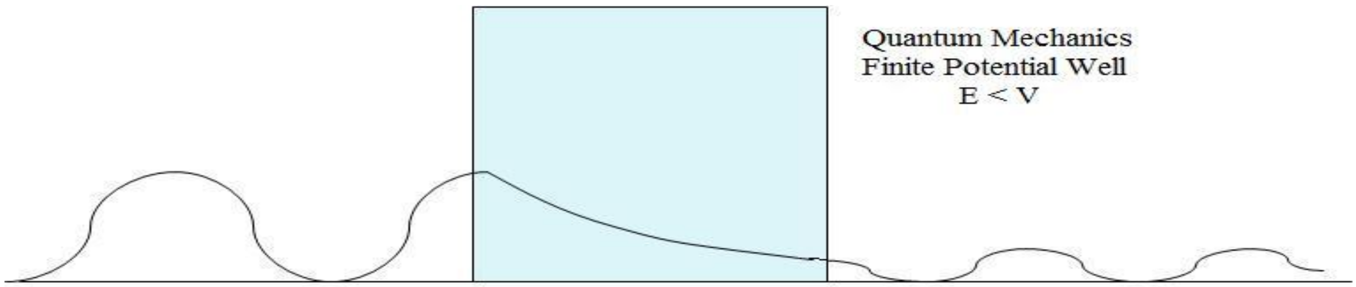


Figure 3: Quantum Potential Well

However in quantum mechanics, electrons possess wavelike properties. These waves don't end at a barrier, but they die exponentially with the thickness of the barrier. If the barrier is thin enough, there will be a noticeable probability of finding electrons on the other side. When the electron tunnels through a barrier in this fashion, it is called tunneling. Because of the exponential decay of the current with the barrier, the number of electrons that will cross the barrier is very much dependent upon the thickness of the barrier [5].

Piezoelectric effect

Pierre Curie discovered the piezoelectric effect in 1880. Piezoelectric effect is the generation of an electric potential on the sides of some materials (crystals), such as quartz when applied to mechanical stress and vice versa. When you apply an electric potential to a piezoelectric crystal, the crystal distorts. The distortions of a piezo are usually of the order of micrometers.

Figure 4 shows diagram explaining piezoelectric effect [16].

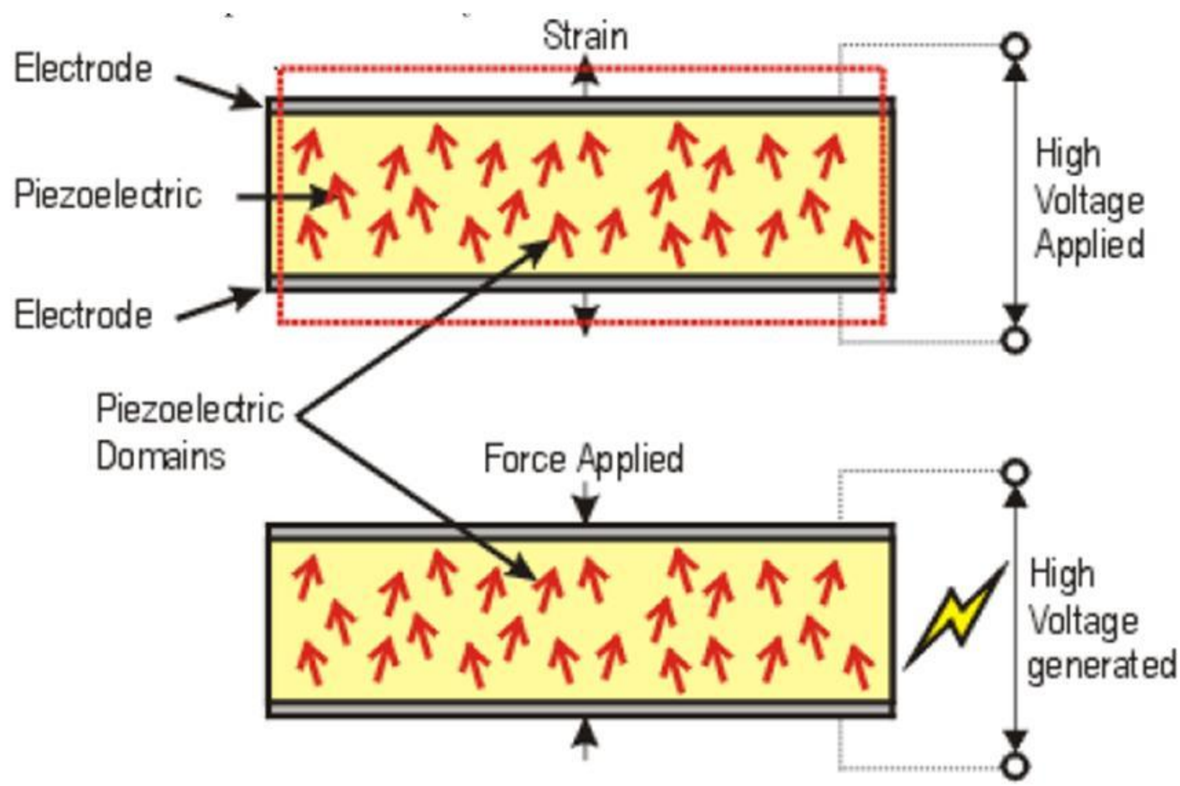


Figure 4: Piezoelectric Effect

These Piezos are also being used in other Probe Microscopes. The piezoelectric material that we used is PZT (Lead Zirconium Titanate).

Feedback controller

A feedback controller monitors the tunneling current, and adjusts the probe-sample separation to maintain a constant current during the scanning of the sample surface. Tip approaches towards the sample until a tunneling current is detected; at that point the feedback controller is turned on. The feedback controller responds to the changes in the tunneling current and if necessary, varies the voltage applied to the tip-positioning piezoelectric crystal until the current reaches the desired value (the set point).

A sample surface can be scanned by two different methods using STM:

- **Constant current**

  In this setup, the feedback controller makes adjustments with the probe-sample separation to maintain the constant value of the tunneling current. These adjustments are recorded by the computer and generate the topography of the sample surface.

- **Constant height**

  In this setup, we turn off the feedback loop and just record the variations in the tunneling current. And then with the help of current distance relation, we generate the topography of the sample surface.

$$I \propto \exp(-d)$$

Where,

I = Tunneling current

d = Distance between the tip and the sample surface

Atomic Force Microscope (AFM)

Instead of measuring tunneling current, AFM measures forces between the tip and the sample surface [7]. The AFM probe is normally attached with the cantilever. Once contact between the tip and the sample surface has been established, the sample moves laterally relative to the tip, while the distance between the tip and the sample is monitored. Variations in sample surface height cause up and down deflections in the cantilever. We record this data and generate the topography of the surface.

Unlike the tunneling current, which has a very short range, Force between the tip and the sample surface has both long and short-range forces contributions, like short-range chemical forces (few nm) and long-range van der Waals, electrostatic and magnetic forces (up to 100 nm) in vacuum.

Figure 5 shows schematic of AFM [17].

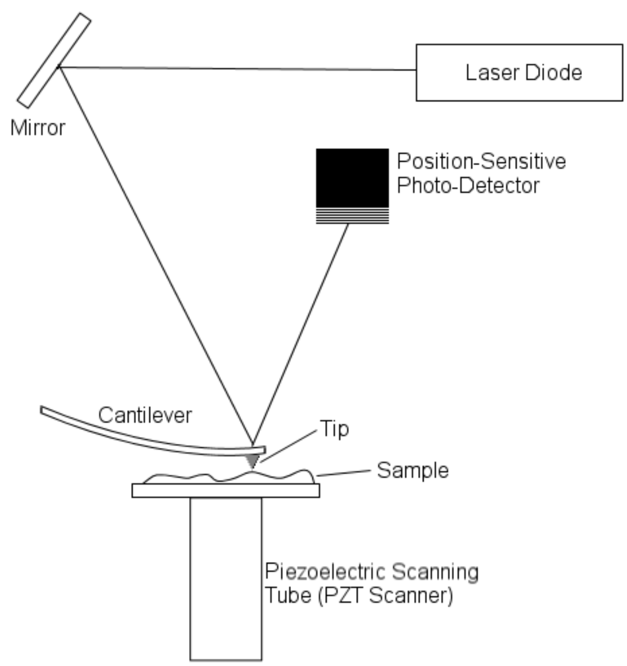


Figure 5: Schematic of AFM

There are two ways of imaging

- **Constant height**

  In this setup, the height of the tip is fixed, and due to change in the sample-probe distance, cantilever deflects.

- **Constant force**

  In this setup, we maintain constant force between the tip and the sample by keeping the constant cantilever deflection. The motion of the scanner piezo in z-direction is recoded.

Modes of operation

There are two modes of operation for AFM

- **Static Mode**

In this mode of operation, the cantilever is kept at rest. When the sample surface approaches towards the tip, the cantilever bends due to the attractive force between the tip and the sample surface, which follows the Hooke's law [8].

Hooke's law:

$$F = -k.x$$

Where,

F = Force
k = Spring Constant (Stiffness) of the cantilever
x = Cantilever Deflection

The force on the cantilever can be calculated, if the stiffness of the cantilever is known. The sample-probe distance can be measured from the extension of the piezo. The force versus distance curve is known as a force curve.

While approaching of sample surface towards the tip, if the force gradient becomes equal to the spring constant of the cantilever, a mechanical instability occurs making the tip to jump to the surface. This is called jump-to-contact instability in AFM.

For static mode AFM imaging, a soft cantilever is needed, which can deflect by very small forces. To achieve greater sensitivity and to prevent jump-to-contact instability, restriction on the range of spring constant of the cantilever applies.

- **Dynamic Mode**

The cantilever is oscillated at or near its resonance frequency. Feedback loop ensures the constant oscillation amplitude, which ensures the presence of constant force between the tip and the sample during scanning.

Forces between the tip and the sample surface will not only affect the oscillation amplitude, but will also change the resonant frequency and phase of the cantilever.

The shift in frequency is tracked using a Phase Lock Loop (PLL) and is kept constant using a feedback controller while sample is being scanned. This is called frequency modulation (FM) proposed by Albrecht et al. in 1991 [9].

If the excitation frequency is kept constant, shift in resonance frequency causes variation of amplitude and phase. The variation of amplitude with tip-sample separation is used in amplitude modulation (AM). The amplitude is used as a feedback and the z-positions of the scanner piezo are recorded. This technique was introduced by Martin et al. in 1987 [6].

Chapter 3

Method

In this chapter, the development of the each parts of the AFM is described.

Tuning Fork

Tuning fork can be used for force measurement with AFM dynamic mode, such as amplitude modulation/phase modulation (AM/PM) and frequency modulation (FM). First we use a lock-in-amplifier to separate amplitude and phase signal from the original signal. From these two signals, we can obtain force. Then, with PLL controller we obtain the resonant frequency of the tuning fork. With the shift in the resonant frequency, the gradient of the force can be obtained.

Quartz tuning fork (QTF) possesses high stiffness of about 1000 N/m, which avoids thermal noise. They show large quality factor Q (~1000) and can achieve pN force sensitivity. The routinely used force sensor in AFM need optics for data acquisition, we can take Electrical read-out from tuning fork due to its piezoelectric property. The sensitivity and the precision of our measurement depend upon the sensitivity of the quartz tuning fork.

Figure 6 shows equivalent circuit of Tuning fork [13].

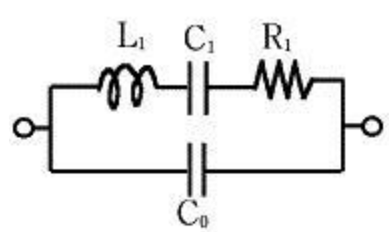


Figure 6: Equivalent circuit of Tuning Fork

Tuning forks are commercially available in the local market since it is the time-keeping element in a normal watch. We procured tuning forks which are sealed inside a metal cap. After opening this cap we glued one of the prongs to a piezoelectric crystal as shown in the Figure 7. The two electrodes are used to measure the charge developed across the other prong. The current generated on the tuning fork due to the piezoelectric property of quartz is of the order of nano amperes, and collected by the electrodes on the arms of the fork. The signal is amplified and converted to voltage signal with the help of a pre-amplifier circuit with a gain of 10^7.

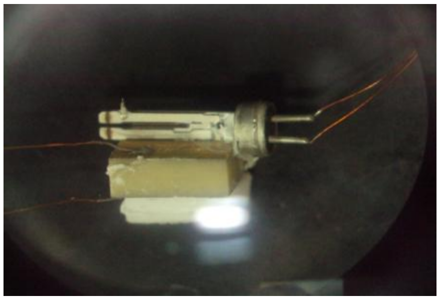


Figure 7: Tungsten tip mounted on Tuning Fork

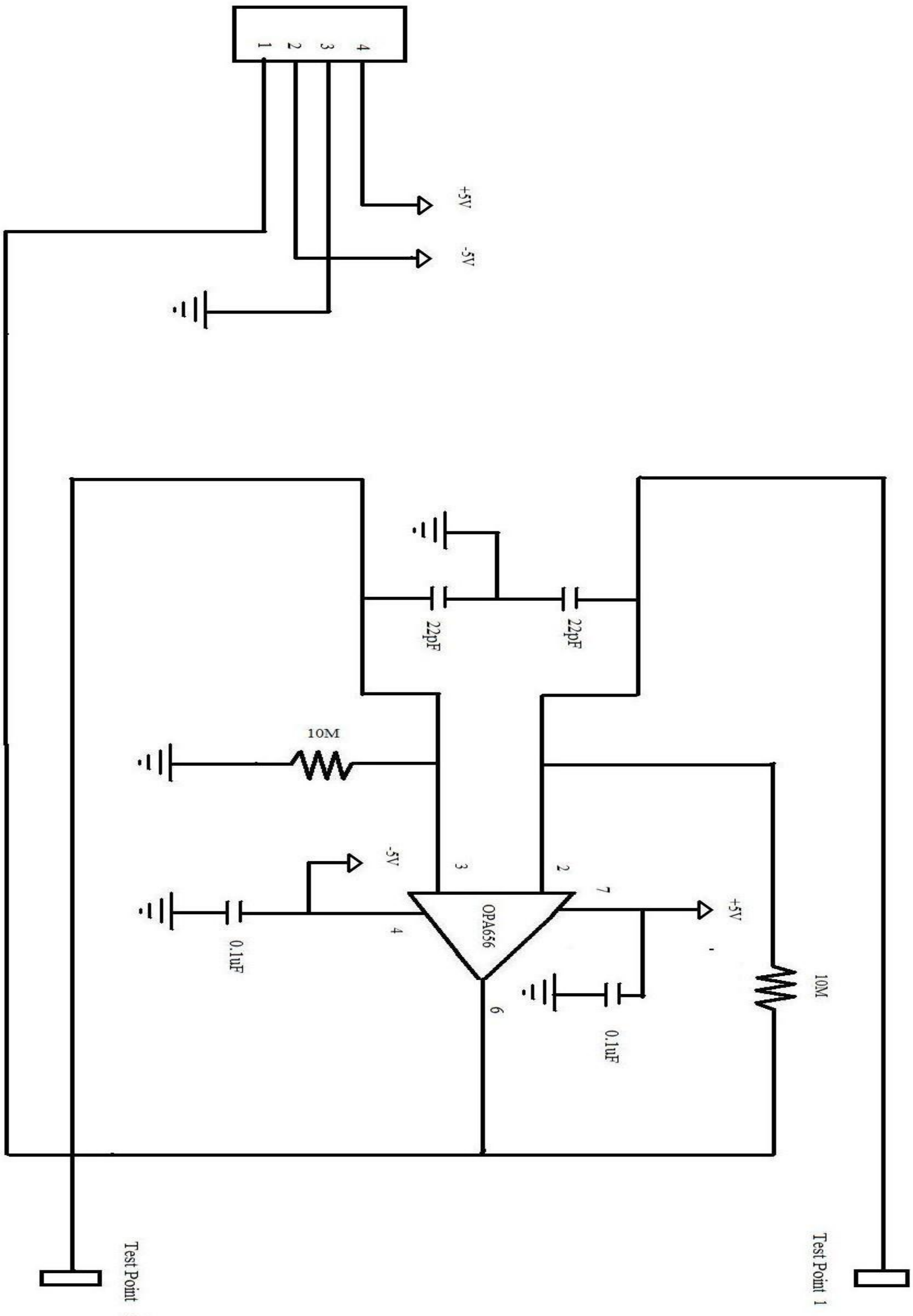


Figure 8: Pre Amplifier circuit

Fig. 9 shows typical resonance characteristics of the tuning fork with and without the tip.

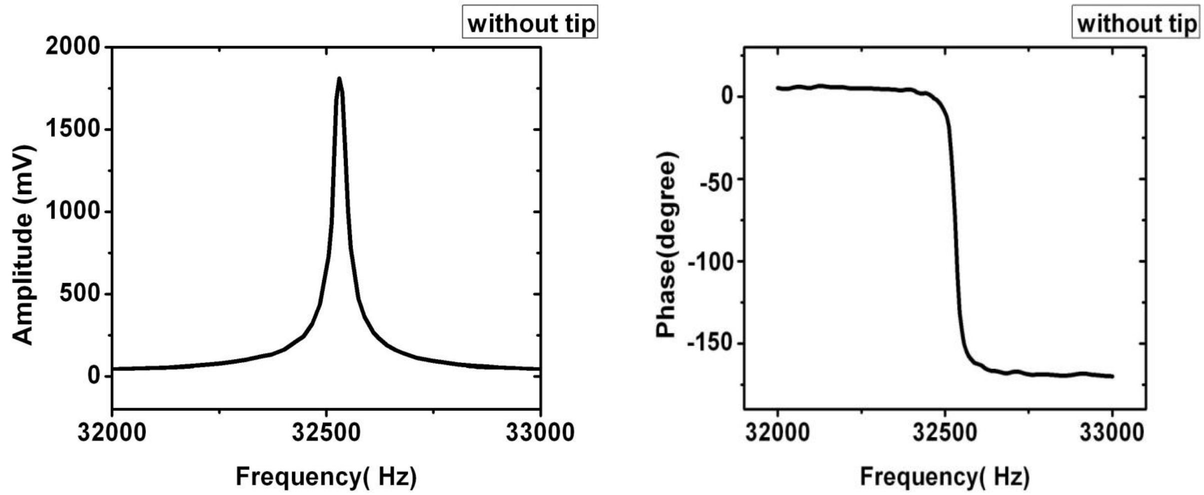


Figure 9: Amplitude and Phase vs Frequency curve of Tuning Fork

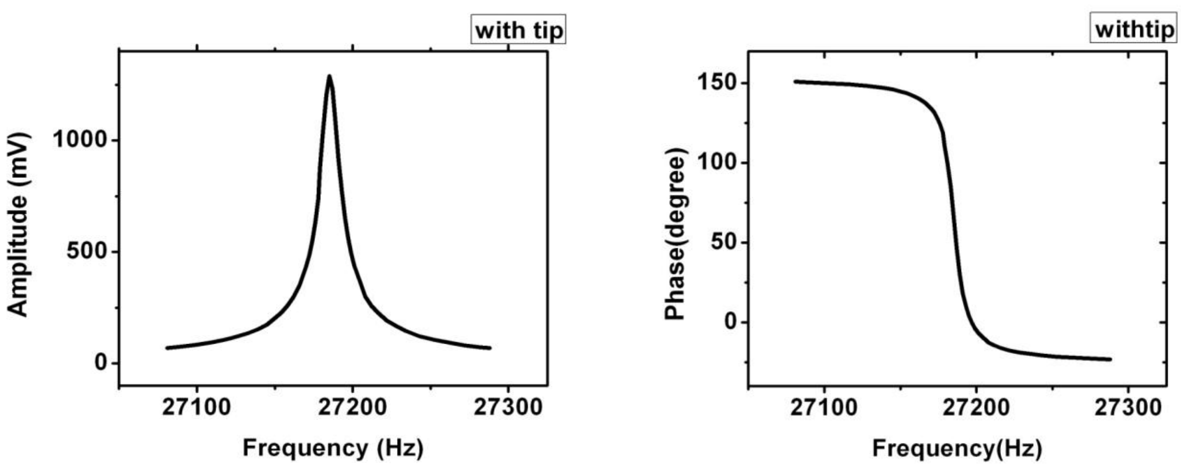


Figure 10: Amplitude and Phase vs Frequency curve of Tuning Fork with tip

Calculations

With the dimensions and geometry of the tuning fork we are using, we have done calculations to calculate the sensitivity of the tuning fork as the force sensors.

The following calculations were done to calculate the sensitivity of the tuning fork:

Figure 11 shows a Quartz Tuning Fork [4].

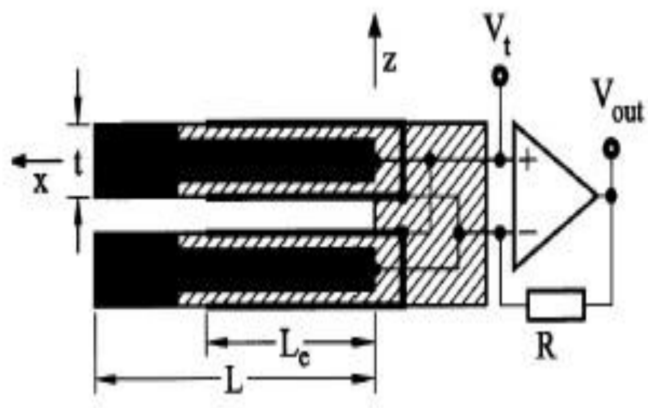


Figure 11: Tuning Fork

For our geometry of the tuning fork, the spring constant (k) is given by

$$k = 0.25Ew(t/L)^3$$

Where,

E = young's modulus

w = width

t = thickness

L = length of one beam of the tuning fork.

When a force $F = kz'$, where z' is the deflection along the Z direction, acts on the upper prong.

The strain is given by

$$\in(x, z = t/2) = \frac{t}{2} F(x - L) \frac{1}{EJ}$$

Where,

J = moment of inertia for the given geometry.

$$J = wt^3 / 12$$

The strain results in the development of stress given by

$$\sigma_{mech} = \in E$$

The stress leads to the charge density

$$\sigma_{charge} = \sigma_{mech} d_{21}$$

Where,

d_{21} = piezoelectric coupling constant.

For Quartz

$$\rho = 2650 kg / m^3$$

$$E = 7.81 \times 10^{11} N / m^2$$

$$d_{21} = 2.31 \times 10^{-12} C / N$$

We integrate σ_{charge} from x = 0 to x = L_e (length of the electrodes) and from y = -w/2 to y = w/2 and taking into account the equal amount of charge being generated on the lower end of the prong, we get the total charge developed as [4]

$$q / z' = 12 d_{21} k L_e (L_e / 2 - L) / t^2$$

For the tuning fork we are using

$$L = 3.5mm$$

$$w = 350\mu m$$

$$t = 700\mu m$$

$$L_e = 3mm$$

The theoretical sensitivity is given by

$$S_{theory} \approx 2\pi f \times R \times q / z'$$

For $R = 10M\Omega$ in our gain circuit and for

$$f = 27187Hz$$

We have,

$$S_{theory} = 319.42mV / nm$$

Tip Preparation

Sharp tungsten tips are very important for scanning with STM, AFM and other related techniques. Electrochemical etching process produces atomically sharp tips from wires.

Techniques of Tip Fabrication

There are two types of procedures for tip fabrication.

- **Mechanical procedures**

  Mechanical procedures involve cutting a metal wire at an angle with a wire cutter or a scissor, fragmenting bulk pieces of material into small pieces and grinding a metal rod in order to get a sharp tip shape.

- **Physicochemical procedures**

  Physicochemical procedures involve electrochemical etching, ion milling and chemical vapour deposition /electron beam deposition onto pre-existing tips.

With mechanical procedures, we get atomic resolution. But it leaves a ragged surface with many tiny tips rising from the apex. Because of which we get multiple image signals from tips. With physicochemical procedures, we can get extremely sharp and symmetric tips in a more reproducible fashion.

For AFM/STM purposes, we need very sharp probe of the order of nm (sharper the tip better the resolution). We use electrochemical etching process to etch the tip.

Electrochemical etching process

In this process we dipped tungsten wire and a small ring electrode into the solution of KOH. A dc voltage is applied for etching. Tungsten wire acts as anode and the small ring electrode as cathode. After sometime (10-20min), the wire becomes so thin that the lower portion of the tungsten wire falls down. The etched wire ends in a uniform manner since gravity is the only force felt by the wire. As the lower portion of the wire falls down, the circuit breaks. Hence we don't need any control to switch off the voltage power supply to prevent further etching [10].

Figure 12 shows schematic of the setup [10].

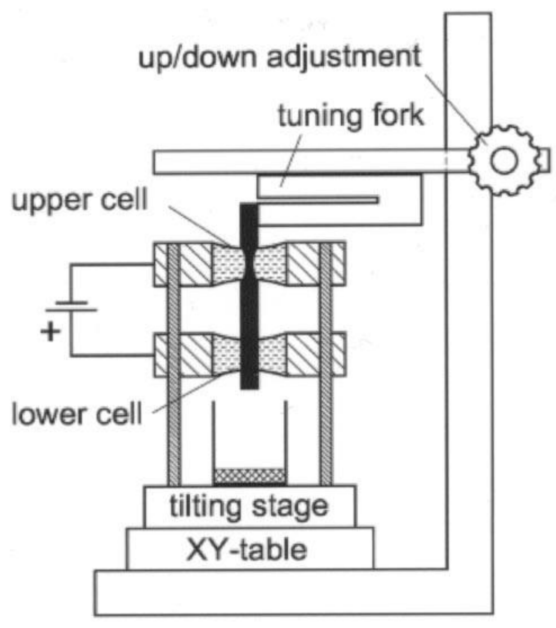


Figure 12: Schematic of Etching Setup

Following electrochemical etching reaction for Tungsten takes place:

Anode: $W(s) + 8OH^- \longrightarrow WO_4^{2-} + 4H_2O + 6e^-$

Cathode: $6H_2O + 6e^- \longrightarrow 3H_2(g) + 6OH^-$

Overall: $W(s) + 2OH^- + 2H_2O \longrightarrow WO_4^{2-} + 3H_2(g)$

Figure 13 shows various steps of etching process.

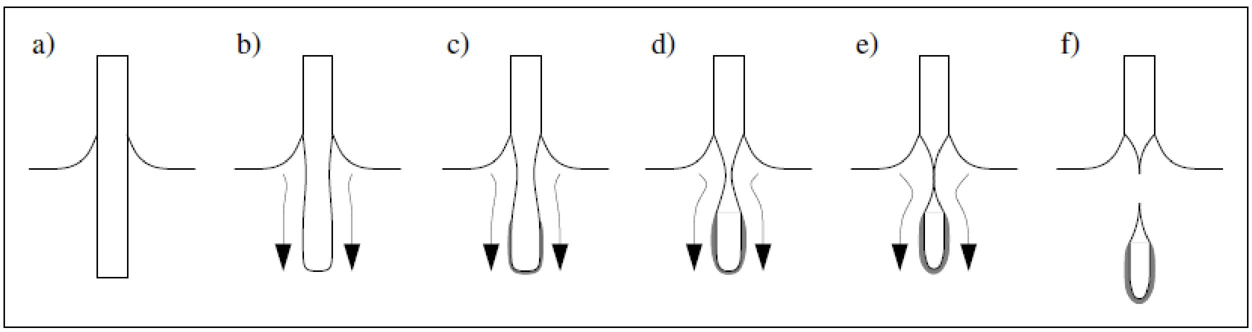


Figure 13: Various steps of Etching Process

Shape and symmetry of tip depends upon shape and stability of the meniscus. For a successful etching process the shape of the meniscus should stay intact throughout the process.

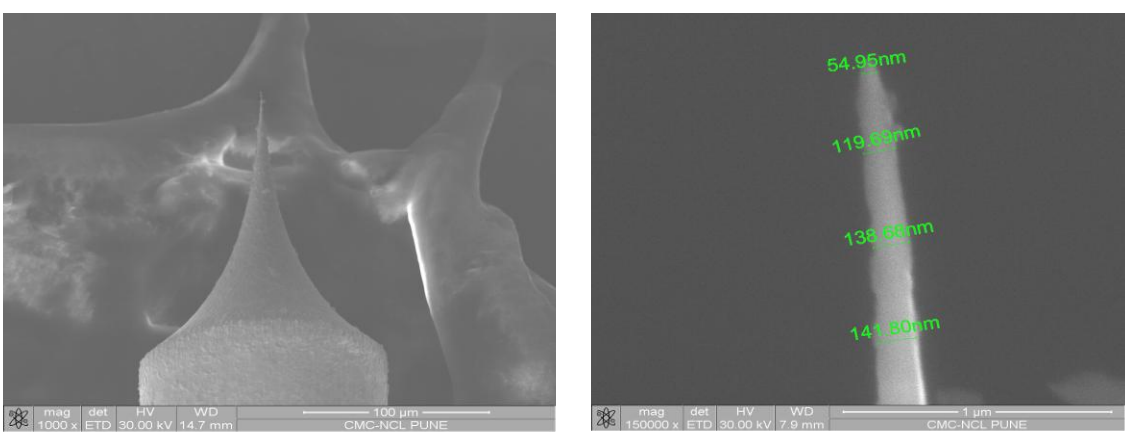


Figure 14: Tip fabricated by Etching Process

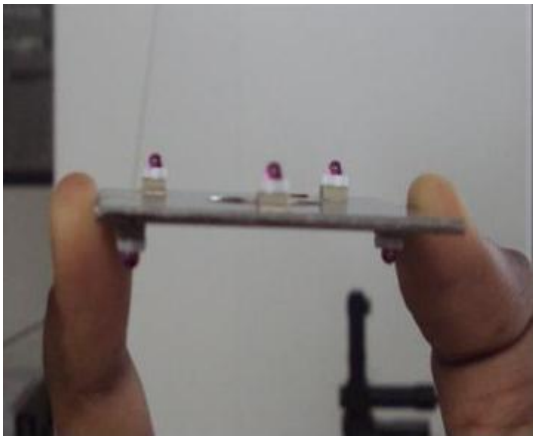
X-Y-Z Nano Positioner

Instrumentation

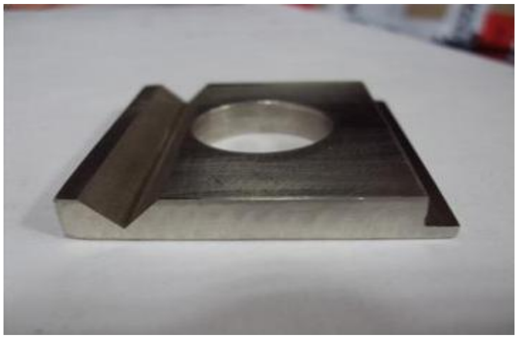
X-Y-Z Positioner is built on a heavy base. The central part of the base is made hollow to accommodate the vertical coarse and fine approach. The sample coarse approach in z direction works on the principle of inertial slider. On the lower end of the hammer piezo tube a heavy steel piece ('hammer') is glued. On the upper part of the hammer piezo tube the glass tube is attached, which is held by a leaf spring on a holder. Inside the glass tube is the scanner piezo tube, which has four quadrants electrode section outside to allow x and y direction motion. The sample holder is attached to the upper end of the scanner piezo [11].

The X-Y sample positioner also works on the inertial slider principle. In this case shear piezos are mounted on heavy steel plate. The X-Y sample positioner consists of grooves cut into the AFM base, a top plate with grooves perpendicular to the grooves in the AFM base and a middle plate with a set of three shear piezos on each side. Macor piece is glued on top of each shear piezo mounted on the middle plate which serves as a seat for a 2mm ruby ball. The ruby ball can slide on the glass plate that is glued inside grooves. Since the grooves on the base and the top plate are perpendicular to each other, X and Y motion can be generated. There is hole in the middle of each plate to accommodate the Z- approach assembly. The sample holder consists of an aluminum plate which has three magnets glued into recessed holes which serve as holders for the sample [11].

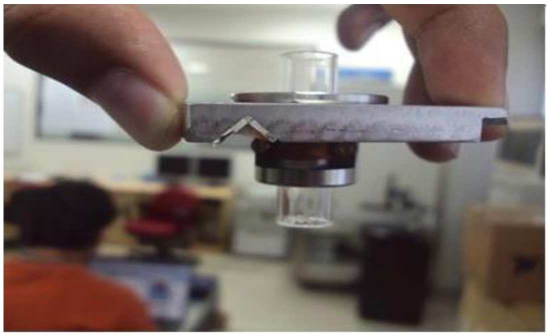
AFM Base



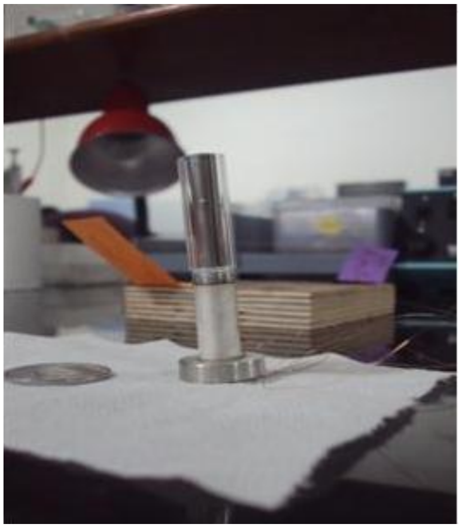
Middle Plate



Top Plate



Leaf Spring



Hammer and Scanner Piezo

Figure 15: Different parts of X-Y-Z Nano Positioner

Figure 16 shows the schematic of X-Y-Z Nano Positioner [11].

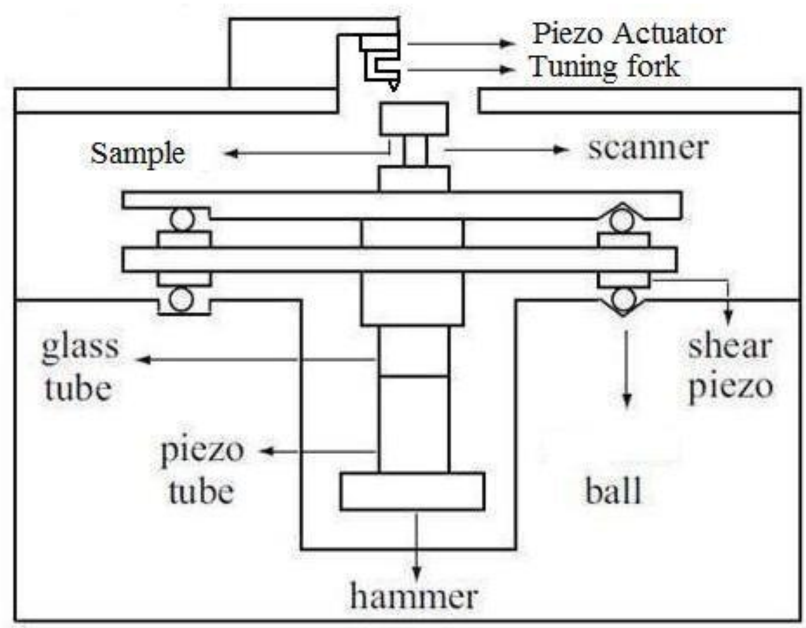


Figure 16: Schematic of X-Y-Z Nano Positioner

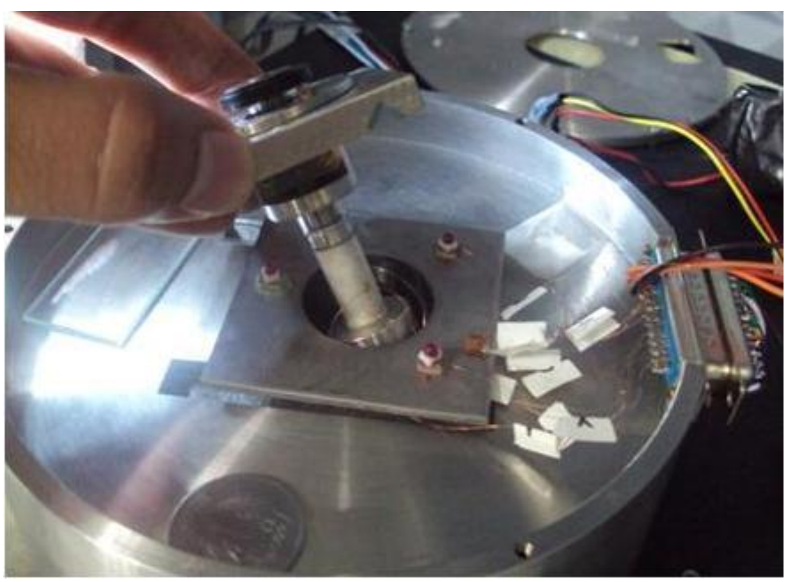


Figure 17: X-Y-Z Nano Positioner Setup

Working

For coarse z motion, exponentially rising and rapidly falling pulses are given to the hammer piezo. During the slow, rising part of the pulse, the tube piezo contracts or expands, moving the hammer up (or down). The upper end remains clamped as the glass tube is held fixed by the leaf spring. During the rapid falling part of the pulse, the inertia of the hammer keeps it in place forcing the glass tube to slide on the leaf spring. A synchronized ramp is applied to the scanner piezo. This combination forms the coarse and fine approach mechanism. Scanner has a linear response to the voltage applied. The amplitude of the voltage ramp of the scanner and that of the pulse for the hammer is kept such that, the distance travelled by the scanner is greater than that of the hammer piezo. The amplitude signal from the tuning fork is recorded through lock-in-amplifier. Amplitude decreases as the tip approaches towards the sample surface due to the force between the tip and the sample surface. For constant force AFM imaging, amplitude of the tuning is kept fixed at some value, that value is called set point. After setting a desired set point, pulse is given to scanner piezo, scanner piezo will go and come back to the position if the set point is not reached. Since, scanner piezo has come back; pulse is now given to hammer piezo. The pulses and ramps keep running till the set-point is not reached. Once the set point is reached, scanner piezo stops at that point. Scanner piezo is taken back to zero voltage, and the voltage of the hammer piezo is decreased such that the voltage of scanner piezo remains between 0 and 1 at set point. Since scanner piezo voltage is less than 1 at set point, its full range can be used at the time of scanning. Once this is done, PID is started to maintain the same set point throughout the scanning. Same set point assures the fixed distance between the tip and the sample.

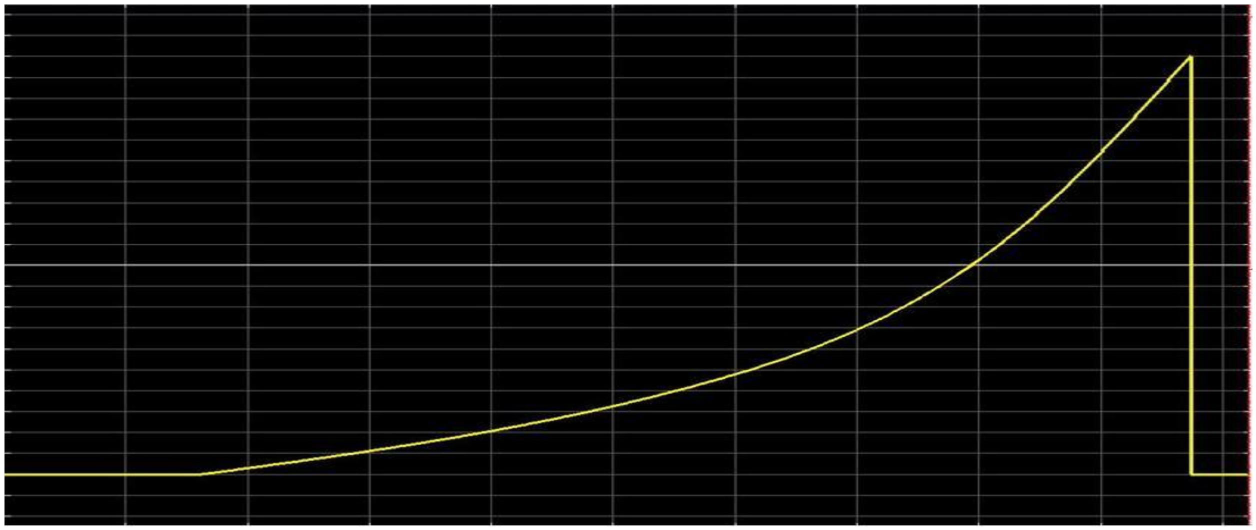


Figure 18: Pulse given to the hammer piezo

Fig. 19 shows the schematic of the approach mechanism.

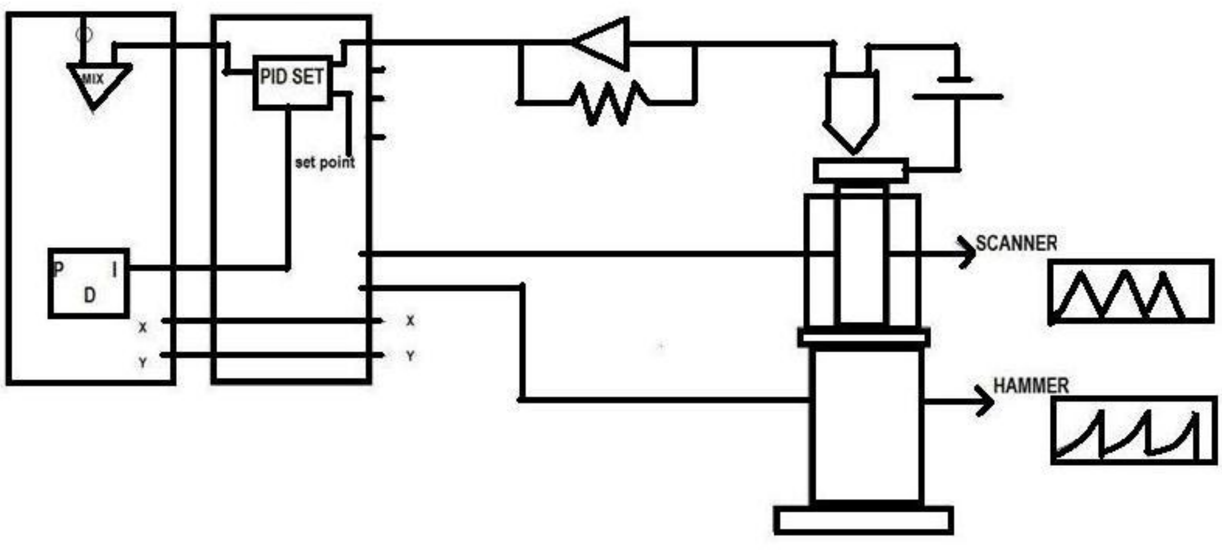


Figure 19: PID connections of the piezos and the pulses

Since scanner piezo is only attached with the sample holder. For scanner piezo, we use standard value for expansion and contraction i.e.45 nm/V. But since, hammer piezo is attached to the hammer at the lower end and on the top of it is lot of load; we calibrate its expansion and contraction with three parameters, Frequency of the pulse, time interval between the pulses and the voltage given to the pulses.

We get maximum motion of 18µm/pulse at

Frequency = 1.1 KHz

Time Interval = 10ms

Voltage= 3 V

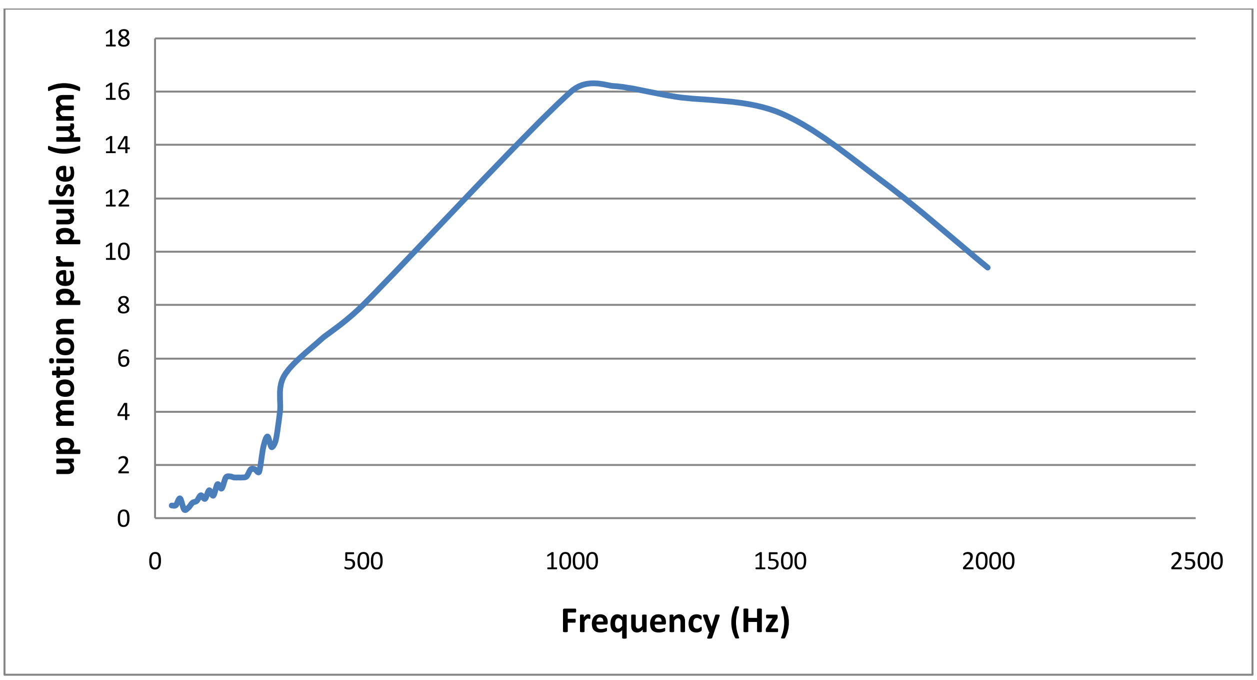


Figure 20: Frequency Calibration

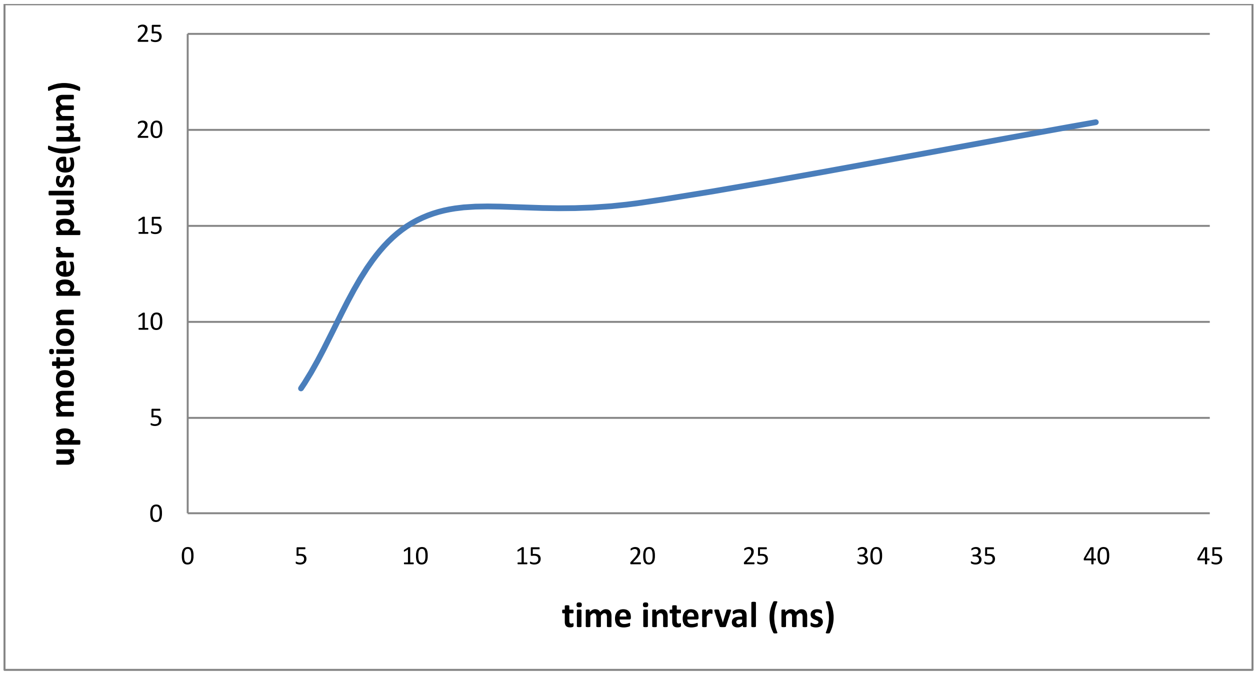


Figure 21: Time Interval Calibration

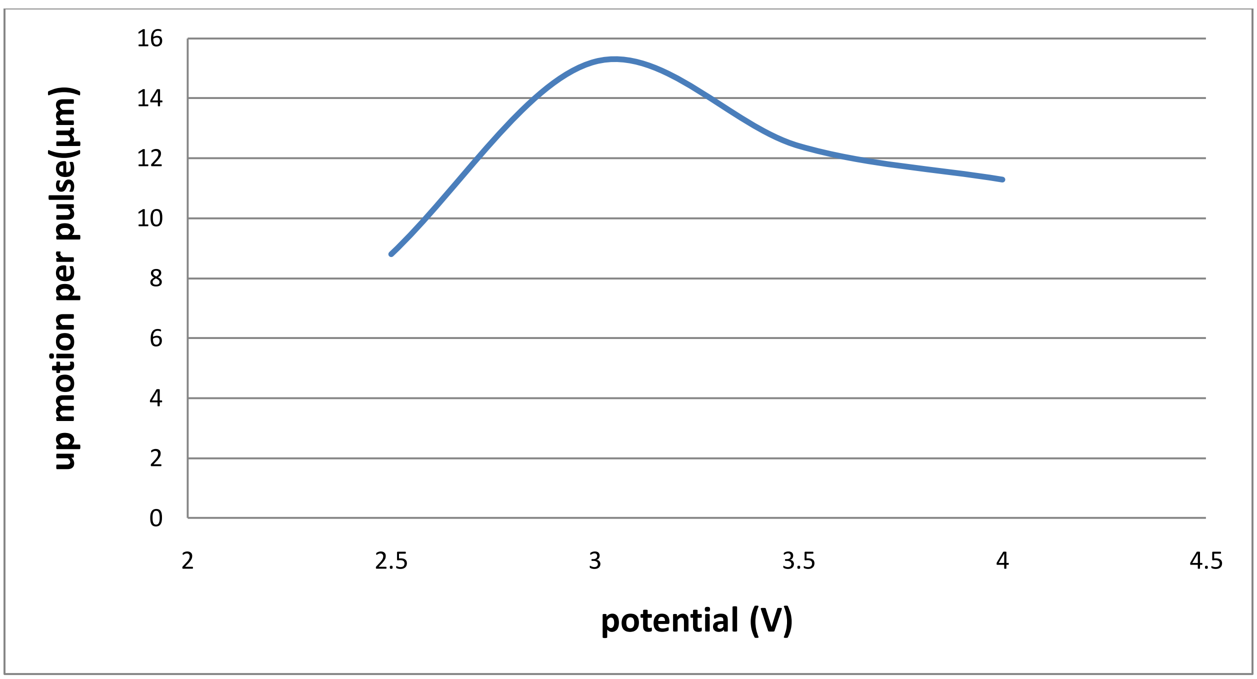


Figure 22: Voltage Calibration

Manual approach is impossible as distances are of the order of nm; we have developed automated auto approach mechanism using LabVIEW software. The program contains the generation of waveforms for the scanner and hammer piezo tubes, auto approach and data acquisition system to obtain the force curves.

Complete Set up

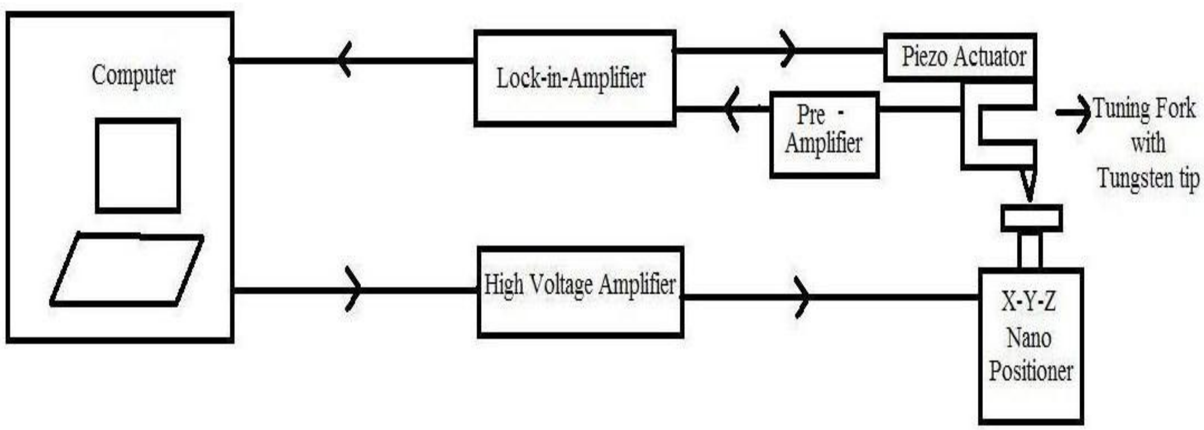


Figure 23: Complete Setup

The resonant frequency of the tuning fork with the tip is checked through lock-in-amplifier and then the piezo is exited with the same frequency. Signals from the tuning fork passes through the pre amplifier having a gain of 10^7 to the lock-in-amplifier. Lock-in-amplifier separates the signals of amplitude and the phase of the piezo. For vertical approach, signals are given to the hammer and the scanner piezo through the high voltage amplifier with controlled gain. With the help of the amplitude signals from the tuning fork, the topography of the sample surface is generated.

Chapter 4

Results

Current Measurements

After developing all parts and their calibration, they are assembled to perform current measurements to test the stability of the set up and the tip-sample junction.

Figure 24 shows current curve obtained with the STM.

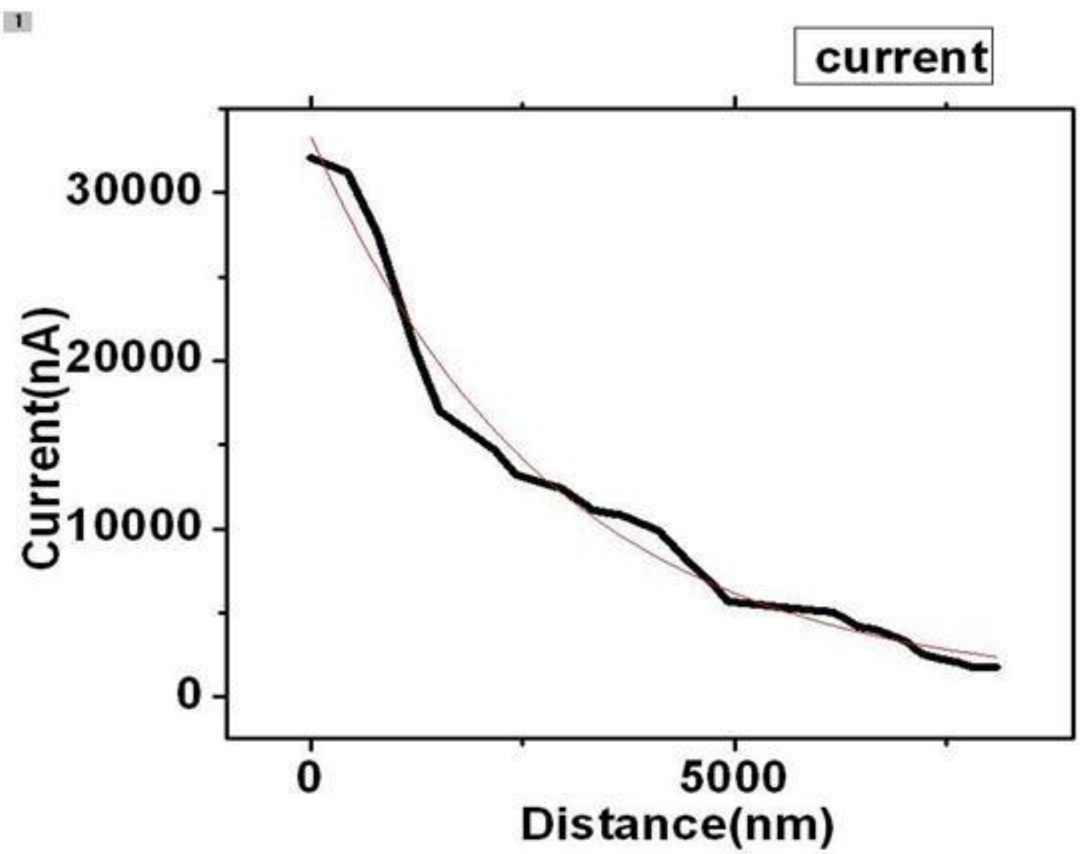


Figure 24: Current curve obtained with STM

Amplitude-Distance curves

After testing the stability of the set up and the tip-sample junction, amplitude distance curve is taken both while approaching towards and retracting from the sample surface.

There are two modes for AFM imaging, namely static mode and dynamic mode.

Static mode

In this mode of operation, we keep cantilever at a position of rest. When the sample surface approaches towards the tip, the cantilever bends due to the attractive force between the tip and the sample surface, which follows the Hooke's law.

Hooke's law:

$$F = -k.x$$

While approaching of sample surface towards the tip, if the force gradient becomes equal to the spring constant of the cantilever, a mechanical instability occurs making the tip to jump to the surface. This is called jump-to-contact instability in AFM.

For static mode AFM imaging, we need a soft cantilever, which can deflect by very small forces. To achieve greater sensitivity and to prevent jump-to-contact instability, restriction on the range of spring constant of the cantilever applies.

Dynamic mode

The cantilever is oscillated at or near its resonance frequency. Feedback loop ensures the constant oscillation amplitude, which ensures the presence of constant force between the tip and the sample during scanning. Upon approaching towards the sample, resonant frequency changes and amplitude of tuning fork decreases because of the interaction force between the tip and the sample surface. The excitation frequency is kept constant and the change in the amplitude of the tuning fork is measured while approaching towards and retracting from the sample surface.

Figure 25, 26 shows the approach and retract curve obtained with AFM in dynamic mode oscillating tuning fork near its resonant frequency.

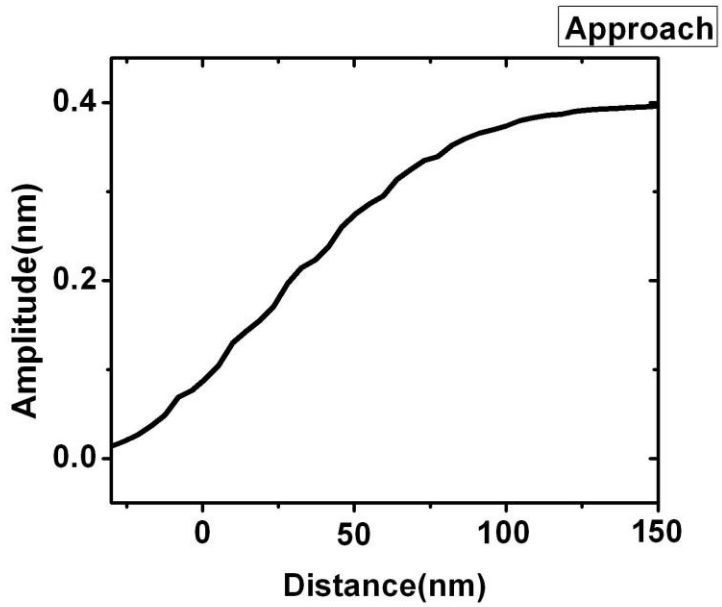


Figure 25: Approach curve obtained with AFM

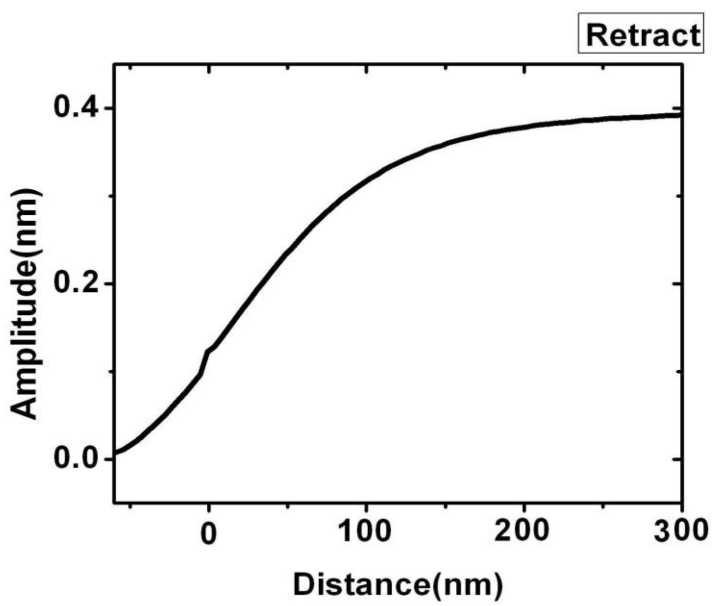


Figure 25: Retract curve obtained with AFM

Chapter 5

Discussion

Current we have measured on oxide coated Si sample. The range of current values and the separation indicates that there could be other processes involved in this measurement such as field emission and possible indentation of oxide layer by the tip. Usually the tunneling current is in the range of nA and dies out to zero at the separation of less than a nm. However these measurements are used to test the stability of our system. It is noteworthy that we could not get stable currents on HOPG or Silver. The gain used for current amplification is clearly not enough for tunneling current measurements and this need to be increased by another two orders. This measurement only shows the stability of the set up and the tip-sample junction. Approach and Retract curves are in the range of 100s of nm, which shows the tip is finely fabricated, and system is able to gather data points for so much short distances (~10nm). With AFM it's now a standard practice to image a flat surface with atomic resolution, regardless of their electrical conductivity. Forces can be calculated easily from the frequency-shift data.

After calibrating X-Y-Z nano positioner, programming is done in LabVIEW software to gather data points and generate topography of the sample surface while scanning. It was done previously with the DAQ card, which was too slow to control PID for maintaining a constant distance between the tip and the sample surface. It is replaced by real time programming FPGA card. Work is still going on to write a programme to work with the PID at a desired frequency to start imaging with the AFM.

Future aspects

Our instrument is ready for imaging at a constant value of amplitude. However we have to develop PID controllers that operate in real time using an FPGA. We have built such a PID. The next step is to build the scanning signals from using this FPGA. We have procured the FPGA card and the programming is underway. Once this is achieved we will start imaging the standards for the AFM imaging to check resolution and X-Y calibration. The final goal of this project is to use AFM with tuning forks in liquid environments. In coming month we are hoping to obtain few images using the home-built tuning-fork based AFM.

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