Fabrication of two dimensional MoS_2 sensors for piezoelectric applications

by

Jasmine Kaur

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Name:	**Jasmine Kaur Gill**
Degree:	**Masters of Engineering**
Title:	**Fabrication of two dimensional MoS2 sensors for piezoelectric applications**
Examining Committee:	**Chair:** Mr. Steve *Senior Lecturer*

Michael Adachi

Senior Supervisor

Assistant Professor, School of Engineering Science, Simon Fraser University

Ash M. Parameswaran

Supervisory Committee Member

Professor, School of Engineering Science, Simon Fraser University

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Abstract

With the growing popularity of miniaturization of high performance piezoelectric devices, the research on applications of Transition Metal Dicalcogenide (TMD) monolayers has also grown a lot. This is because of the suitable piezoelectric properties exhibited by these two dimensional materials. In this project, a piezoelectric sensor, which could be used for various applications, is being designed which could receive both low and high frequency signals well. This project is in collaboration with Think Sensor Research, an organization working towards the development of marine and aerospace sensor applications. The present version of this device, which is being used by the collaboration company, possesses Lead zirconate titanate (PZT) as the piezoelectric material. Our purpose was to design and fabricate this device using Molybdenum Disulfide (MoS_2) instead of PZT and observe the response and piezoelectric properties of the device.

Keywords: Piezoelectric devices; Transition Metal Dicalcogenide (TMD); Molybdenum Disulfide (MoS_2); marine and aerospace sensor; Lead zirconate titanate (PZT)

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I also want to thank my team members Hope Xi and Steven Wang who are working towards designing and manufacturing the testing setup in this project. I got an opportunity to learn the circuit design and fabrication while I assisted them in building the testing setup. They guided me a lot. I want to thank M. Bakhtiar Azim and Abdelrahman Askar for their guidance in operations of cleanroom. I appreciate everyone who helped me and contributed in this project.

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List of Acronyms

TMD	Transition Metal Dicalcogenide
MoS_2	Molybdenum Disulfide
PZT	Lead Zirconate Titanate
MEMS	Micro-Electro-Mechanical Systems
NEMS	Nano-Electro-Mechanical Systems
Cr	Chromium
Si	Silicon
SiO_2	Silicon Dioxide
PDMS	Polydimethylsiloxane
IV	Current-Voltage
Op-amp	Operational Amplifier
PCB	Printed Circuit Board
mV	Milli Volts
cm	Centimeters
DIY	Do It Yourself

Chapter 1.

Introduction

Two dimensional materials such as TMD monolayers are reported to possess high crystallanity and capability to endure enormous strain, because of which they are suitable candidates as high performance piezoelectric materials.[1] For a material to be piezoelectric, it should be non-centrosymmetric i.e. not possess a point through which the spatial inversion leaves the structure invariable. So, TMD monolayers are ideal candidates as low dimensional piezoelectric materials because of their structural non-centrosymmetry. [2]

There has been a rapidly growing demand for high performance miniaturized devices in Micro-Electro-Mechanical Systems (MEMS) and Nano-Electro-Mechanical Systems (NEMS).[3]This demand also makes Transition Metal Dicalcogenide Crystals (TMDC) such as $MoS_{2,}$ an ideal candidate for this device as they can be used to manufacture nanoscale piezoelectric sensors. Reducing dimensionality of bulk MoS_2 enhances its piezoelectric properties due to breaking of its inversion symmetry and MoS_2 as well as other TMDC's can retain their atomic structures even till monolayer, without lattice reconstruction.[4] Due to reported promising characteristics of $MoS_{2,}$ this material is being used to fabricate the sensor and observe the results.

Chapter 2.

Proposed Design of the Device

The proposed design was to use a pre patterned silicon substrate. This substrate consists of a layer of Silicon Dioxide over Silicon. Gold contacts have been patterned on top of Silicon Dioxide layer such that there is a spacing of 3, 4 or 5 microns between two adjacent contacts. There is a layer of Chromium under Gold contacts. A monolayer or few layers of MoS_2 has been stamped between two adjacent gold contacts in order to create a free standing MoS_2 layer. Some part of the gold contacts has to be left uncovered for electrical probing and connections. The proposed design has been depicted in Figure 1.

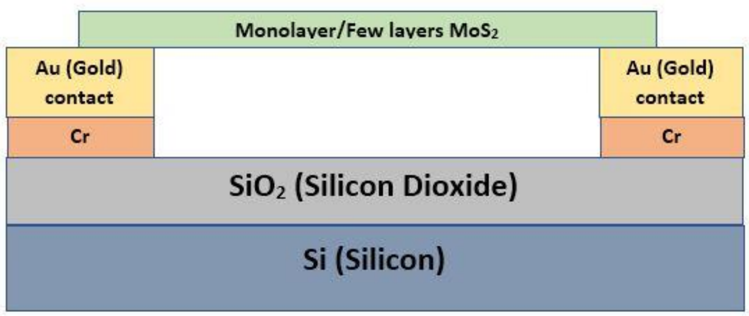


Figure 1 Proposed design of the receiver

Chapter 3.

Device Fabrication

3.1. Fixing the Bulk Crystal:

The thin layer of MoS_2 had to be obtained from the bulk crystal. In order to exfoliate thin layer, the bulk had to be affixed to a surface to make the process easy. So, the bulk crystal was fixed on a glass slide using a double sided tape. The strategy was to hold the glass slide while exfoliating thin MoS_2 and reduce the risk of contaminating the bulk crystal. [5] The process of fixing the bulk crystal has been shown in figure 2.

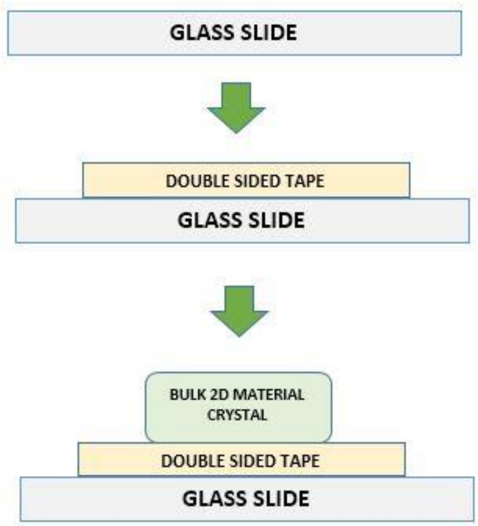


Figure 2 Process of fixing the bulk crystal on glass slide [5]

3.2. Obtaining Flake of Bulk Crystal:

While obtaining a flake from the bulk crystal, it is very important to make sure that there is minimum contamination. So, we used a blue nitro tape to obtain the flake as this tape has much cleaner adhesive layer and it transfers minimum amount of adhesive while exfoliating the material. [5] The setup has been shown in figure 3.

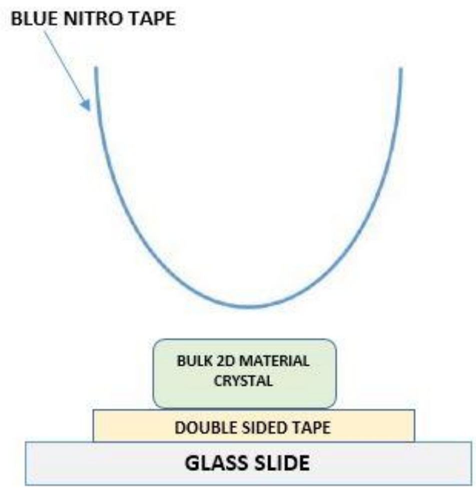


Figure 3 Setup to obtain the flake of MoS2 on the tape [5]

The tape was brought in contact with the bulk crystal affixed to the glass slide and was pressed gently as shown in figure 4.

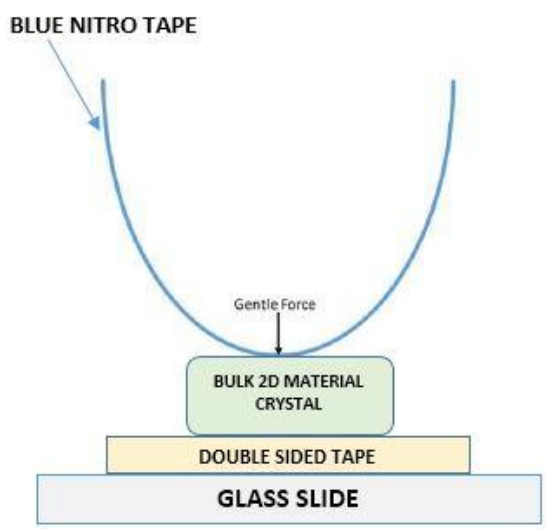


Figure 4 Obtaining the flake on the tape [5]

Then the tape was gently lifted as shown in figure 5. As a result, the flakes were obtained on the tape.

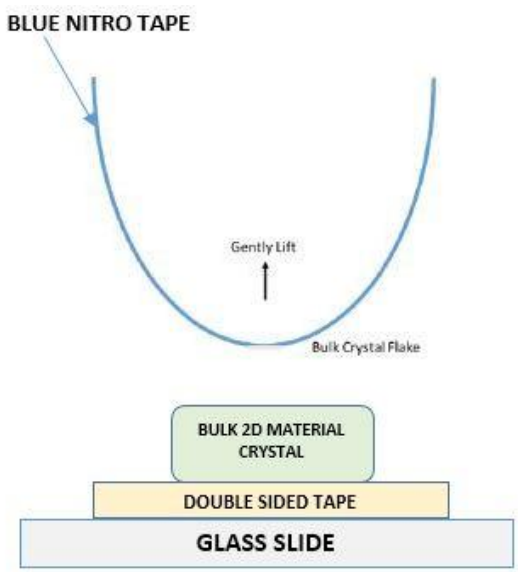


Figure 5 Gently lifting the tape to obtain flakes [5]

A little pressure can be applied on the tape to obtain the desired crystal on the tape, but the idea is to strike a balance and not apply too much or too less pressure. The desired crystal for a piezoelectric device is a wide but thin layer.

3.3. Thinning down Bulk flake to a few layers:

The next step was to thin down the number of layers of the crystal as it still has macroscopic number of layers. So, once a flake of reasonable size was picked up, the tape was repeatedly folded back upon itself to thin down the flake, and obtain a material that is only a few atomic layers thick. [5] This process has been depicted in figure 6.

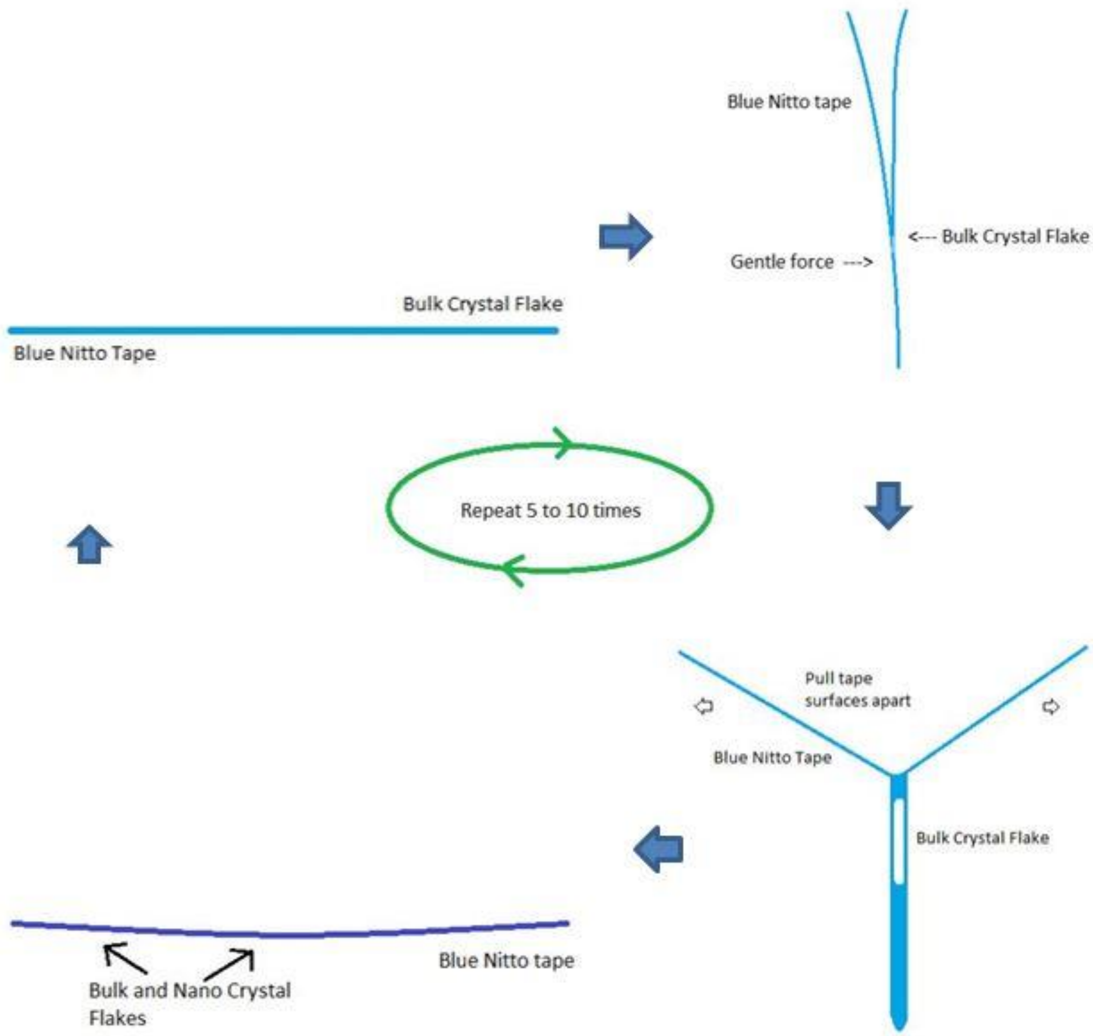


Figure 6 Process of thinning down the bulk flakes. [5]

The number of repetitions done has significant implications on the resulting material. For example, if more repetitions are performed, around 10 times, it leads to flakes becoming thin and there is more abundance of few layer material, but it also decreases the flake size as the flakes break down into smaller pieces during these repetitions and these flakes become less useful due to their small size. On the other hand, if lesser number of iterations are performed, around 3 or 4, there is less abundance of thin layer materials but the flake size is bigger. So, we had to maintain a balance between these two to obtain the desired material. [5]

3.4. Transfer to Polydimethylsiloxane (PDMS) substrate:

The next step was to transfer the desired set of flakes onto a fair sized (~ $1cm^2$) PDMS substrate. This size of the PDMS allows us to transfer more flakes from the tape, hence increasing the likelihood of obtaining the desired flake as exfoliation is a random process. A piece of PDMS was cut from the large sheet and after removing the top thin covering, it was placed on top of the desired flake on blue tape as shown in Figure 7.

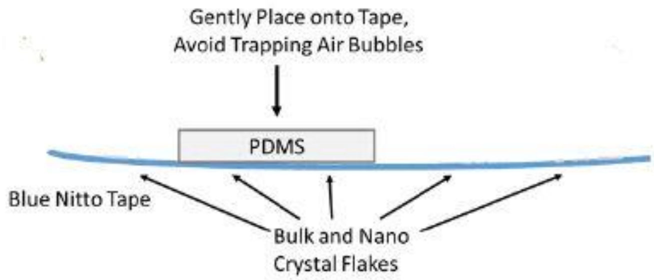


Figure 7 PDMS placed on the tape [5]

The ideal method to transfer the maximum amount of the flakes is to let the PDMS sit on the tape for approximately 2-3 minutes and then remove it very quickly as shown in Figure 8.

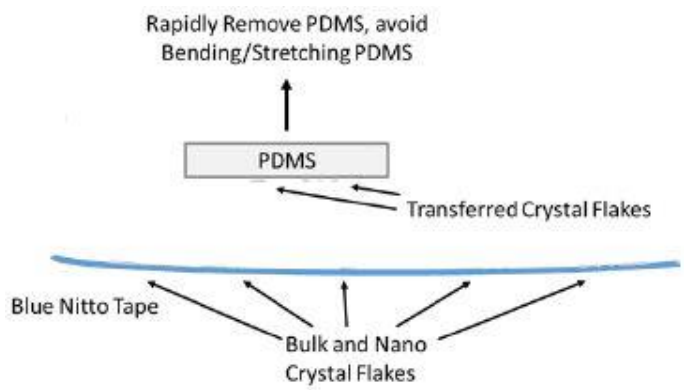


Figure 8 Rapid removal of PDMS [5]

We used tweezers to hold the PDMS and tape and removed it very quickly.

3.5. Deterministic Dry Transfer to the stamp:

A dry transfer method makes use of a microscope and a micromanipulator to precisely place the desired flake on a specific location on the substrate. It involves a few steps. To setup the system, the three axis micromanipulator is affixed with a glass slide and then a very small piece, roughly 1mm – 2 mm, of PDMS stamp is placed on that glass slide as shown in figure 9.

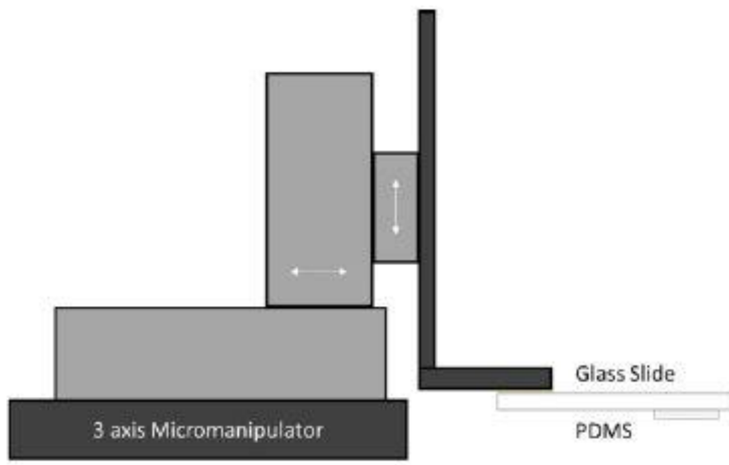


Figure 9 Affixation of glass slide with the PDMS stamp on the micromanipulator [5]

The PDMS stamp actually consists of two layers - A thick (~ 1-2 mm) homemade PDMS film made of 2 part mixture (Sylgard elastomer for Owens Corning) and a Gelfilm x 4 retention, a thin PDMS film from Gelpak..The thick protective layer was removed from the gel film and the gel film was placed on top of the PDMS and they were cut together using a knife. This stamp was placed on the glass slide. [5]

3.5.1. Finding the Flake:

The PDMS that has the flakes on it, was also pasted on a glass slide using its adhesive side and this slide was pasted on the microscope stage using the scotch tape. Then the desired flake was found using the microscope. To obtain piezoelectric properties, we look for dark or almost transparent looking flakes on the PDMS. These are mostly found on the sides of flakes with more number of layers. Also, the micromanipulator is fixed on the side of the microscope stage using a double sided tape as shown in figure 13.

3.5.2. Transferring the desired flake to the stamp:

Before the material can be transferred to the pre patterned silicon substrate, it must first be transferred to a PDMS stamp affixed to the micromanipulator. The actual substrate is a pre patterned silicon substrate which has a coating of Silicon dioxide and gold contacts patterned on top of Silicon dioxide. Once the desired flake is transferred to the stamp, our purpose is to place that stamp on the actual substrate in such a manner that desired flake gets placed between the targeted gold contacts. [5] The reason behind making the stamp so small is to not cover the gold contacts completely during this step and allow easy electrical probing later. Hence, the desired flake was aligned with the PDMS stamp using the axis controls of the micromanipulator as shown in figure 10.

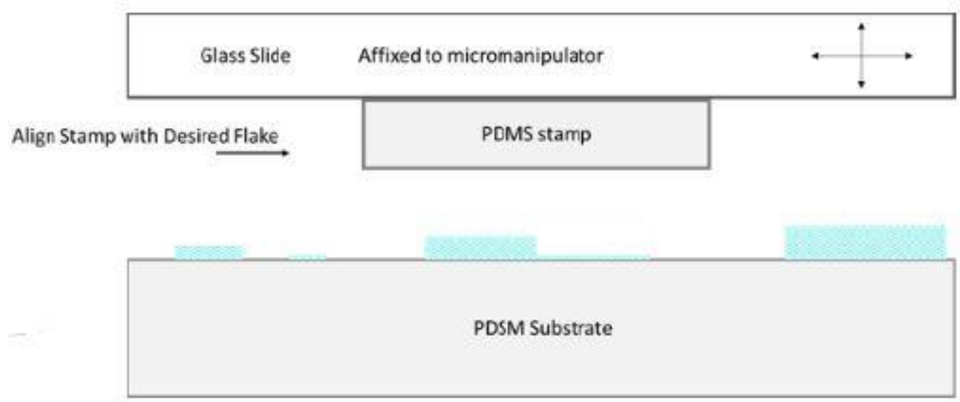


Figure 10 Alignment of the stamp and the desired flake [5]

Initially, we used 5 times zoom to align them roughly and eventually moved to higher zoom. The microscope can only focus on one object at a time, so the focus had to be adjusted between the flake, and the PDMS stamp to observe their alignment precisely. [5] Once, we were satisfied with alignment, the stamp was brought in contact with the PDMS very slowly as shown in figure 11.

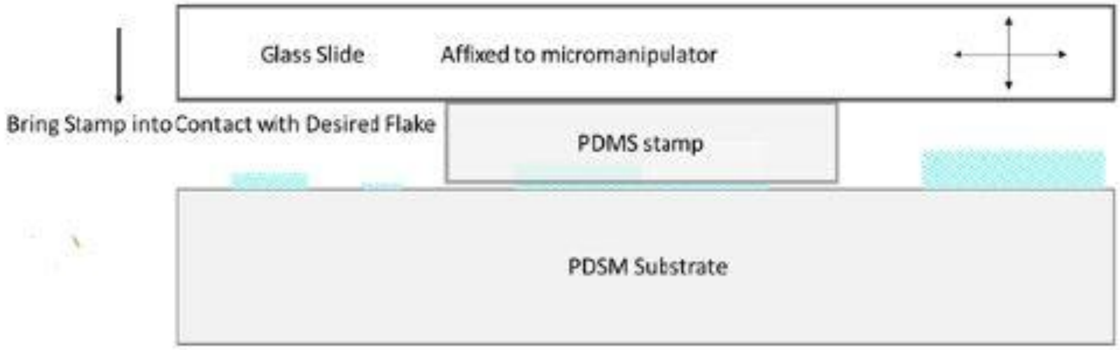


Figure 11 Stamp in contact with the desired flake [5]

When they come in contact, a meniscus becomes visible at their point of contact and the area in contact and the area not in contact become differentiable. This difference was exploited to obtain only the desired part of the flake onto the stamp and leave the rest. Once the desired part comes in contact, there is a visible color change outside the meniscus. The color appears duller or darker when the flake is properly adhering to the stamp.

So, once we confirmed the adherence, we slowly raised the stamp using the y-axis control of the micromanipulator as shown in figure 12.

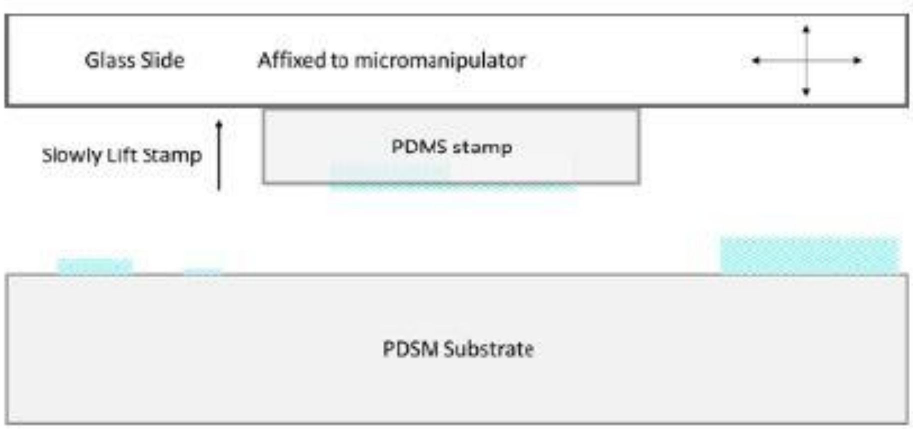


Figure 12 Flake transferred to the stamp [5]

The slower we move, the higher the chance of picking up the desired flake. Hence, we raised it slowly to obtain the flake. After the stamp was not in contact with the PDMS, we brought it up higher and the microscope was refocused on the stamp to confirm that the desired flake has been obtained.

The complete transfer setup has been shown in figure 13.

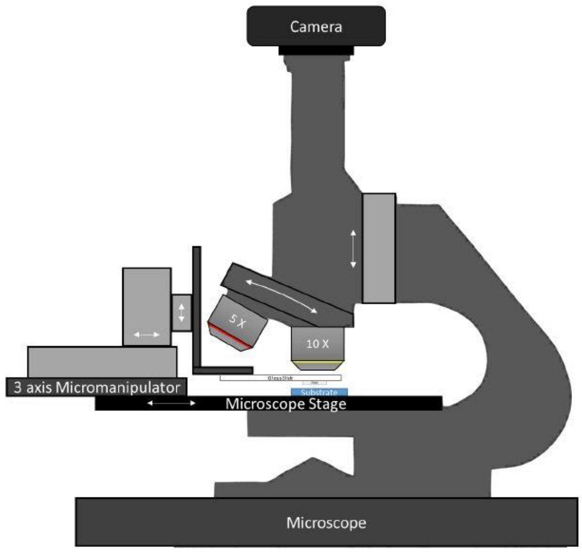


Figure 13 Complete transfer setup [5]

3.6. Deterministic Transfer to the pre patterned substrate:

The next step is to put the stamp on the desired spot on the pre patterned substrate. The same microscope and micromanipulator setup was used for this step. The glass slide with the PDMS is removed from the microscope's surface and the substrate is placed over there. The substrate is rotated to obtain the gold contacts on the desired place according to the shape and location of the desired flake on the stamp. This step has to be done slowly, while checking the alignment and making the adjustments at the same time to avoid any mistake. A 5 times zoom lens is used initially. To avoid lowering the stamp too far, too quickly, the focal plane of the microscope can be set somewhere in between the flake and the substrate, and the flake can be lowered until it comes into focus. This procedure can be repeated until the flake is almost in focus at the same time as the substrate. Then a 10 times zoom was used to precisely adjust and confirm the position of the flake before bringing the stamp in contact with the PDMS.[5] Once it was confirmed, we brought it in contact as shown in figure 14.

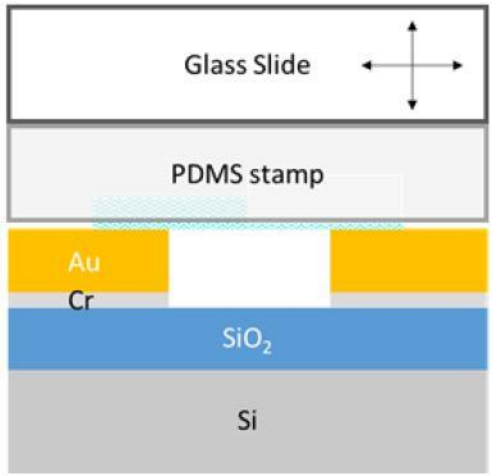


Figure 14 Transfer of PDMS stamp, possessing the flake, onto the substrate [5]

Then it is quickly raised, the substrate being raised along with the stamp. Once it was raised a bit, the edge of a knife was used to detach the stamp from the glass slide as shown in figure 15.

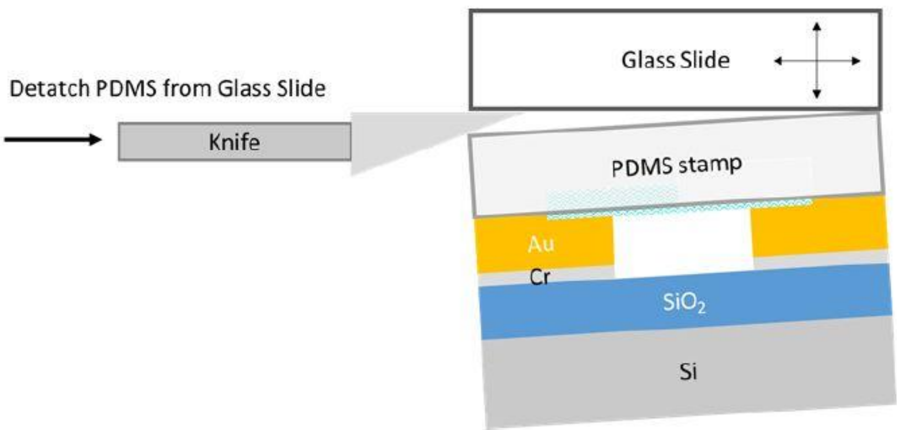


Figure 15 Detachment of PDMS stamp from the glass slide [5]

The device fabrication was completed after this step.

Chapter 4.

Actual Device

The microscopic view of the actual device that was fabricated has been shown in figure 16.

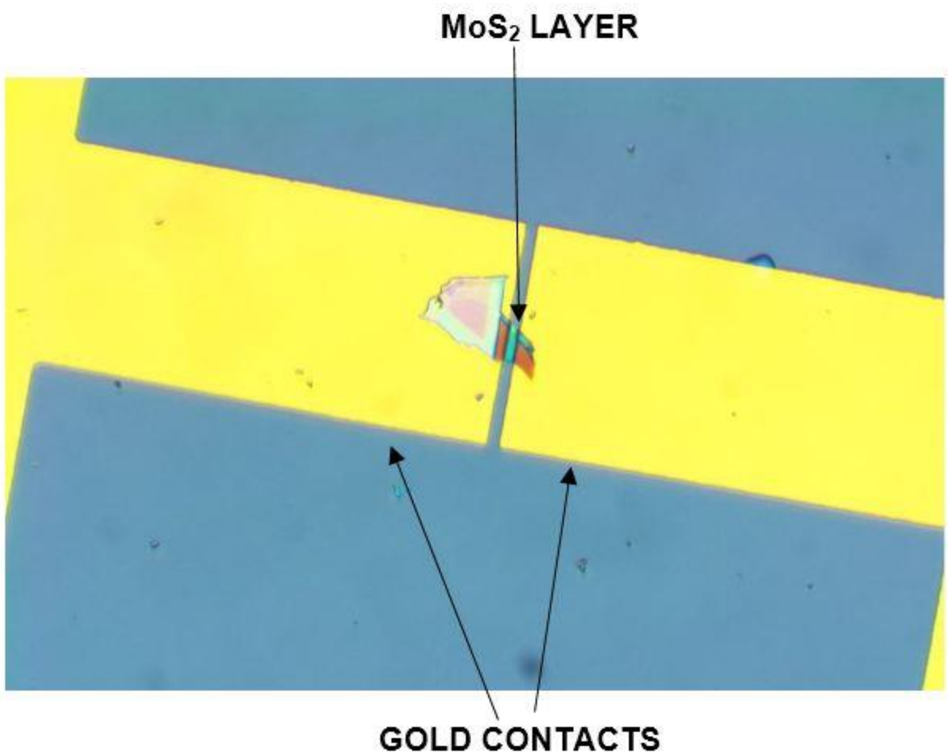


Figure 16 Microscopic view of the actual device

Chapter 5.

Application of electrical current to the device

After the device was fabricated, we tested it by applying electrical current and obtaining the Current Voltage (IV) curves. A voltage range of -15 V to +15 V volts and reverse sweep from of +15 V to -15V was supplied and the curves obtained have been shown in figure 17.

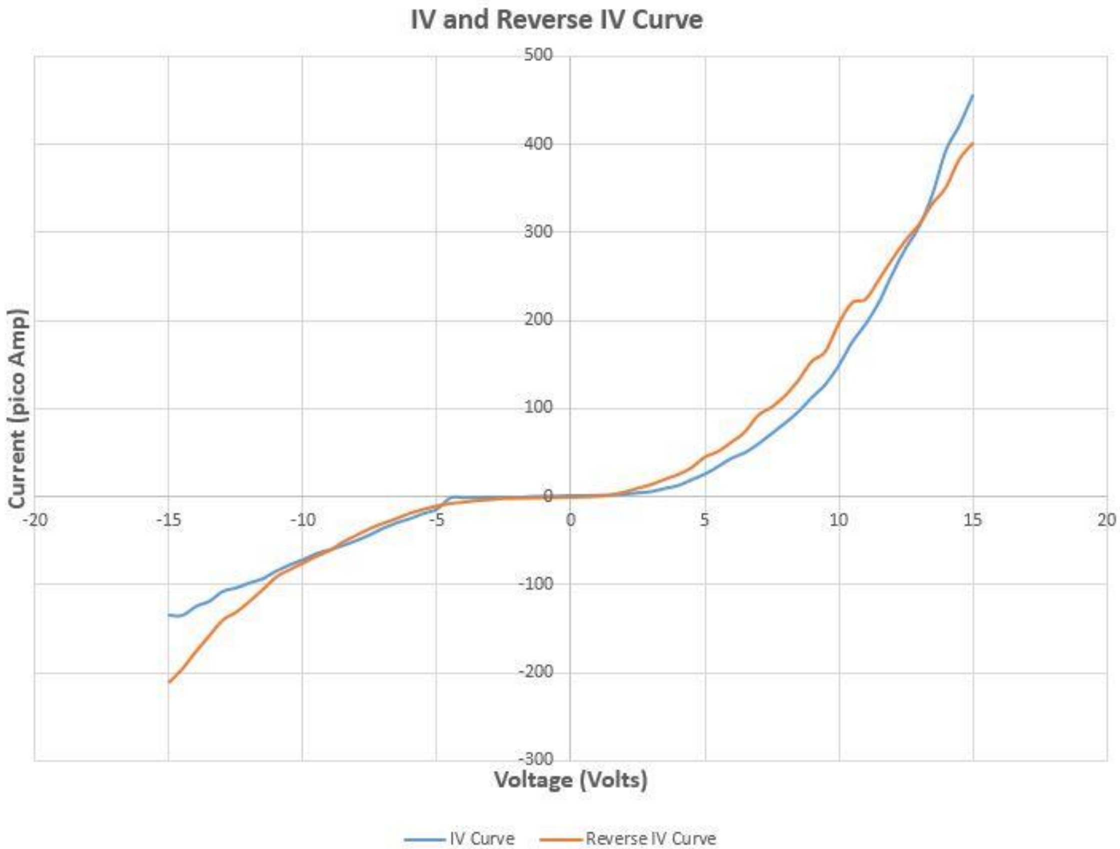


Figure 17 IV and Reverse IV Curve

Chapter 6.

Testing Setup

A testing setup was designed to test the piezoelectric sensor which was fabricated. The goal was to use an actuator as a signal sender and determine how well our device receives that signal at different values of distance between the actuator and the receiver. This section states how the testing circuit was built and what modifications were made for optimization. At the initial stage, a ThorLabs PA4FKW 150V piezo chip was used as a sender and a sin wave was provided to this sender using a function generator. A ThorLabs TA0505D024W 75V Multilayer Piezo Actuator was used as a receiver and the idea was to use an operational amplifier to amplify the signal received by this receiver. This was done, keeping in mind that the signal received by the original receiver, that was being designed, will be very weak and will need amplification. Figure 18 depicts the circuit design. The two PZT are sender and receiver respectively. A sinusoidal wave of peak to peak 5 volts and 10 kHz frequency is being supplied to the sender.

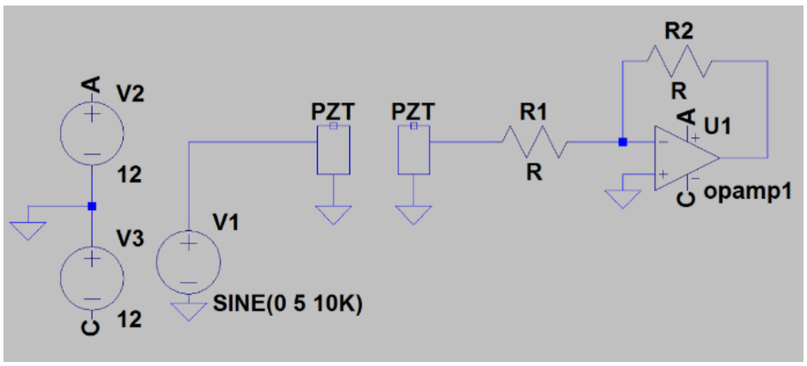


Figure 18 Initial design of the circuit

Figure 19 shows the actual circuit setup on a breadboard. V_{cc} is the positive input voltage being given to the op amp and V_{ee} is the negative input voltage being given to the op amp. G_{nd} is ground. PZT Node (CH1) is the signal input node and Output (CH2) is the output node that is connected to the oscilloscope to obtain the resulting signal.

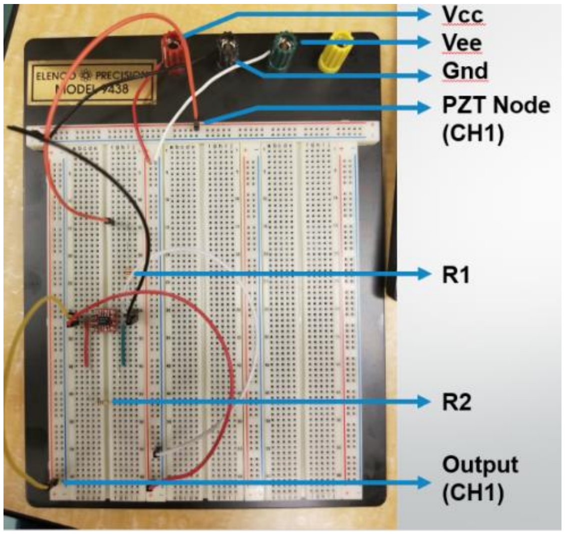


Figure 19 Actual circuit on the breadboard

Initially the op-amp used in this setup was TL072. But with this op-amp, no significant results were found. The output, at many different input voltages, was a bad signal. Then op amp LTC6240 was used. The op amp was attached on top of a PCB board in order to make connections on breadboard. Figure 20 depicts the opamp connections. Figure 21 depicts the connections of PCB board. Figure 22 depicts the connection relations between op-amp and printed circuit board (PCB) board. An input voltage of 5 volts is provided to the Node 7 of op amp, an input voltage of -5V is provided to node 4 of op amp, output signal is received from node 6, and input signal is provided through node 2 and 3.

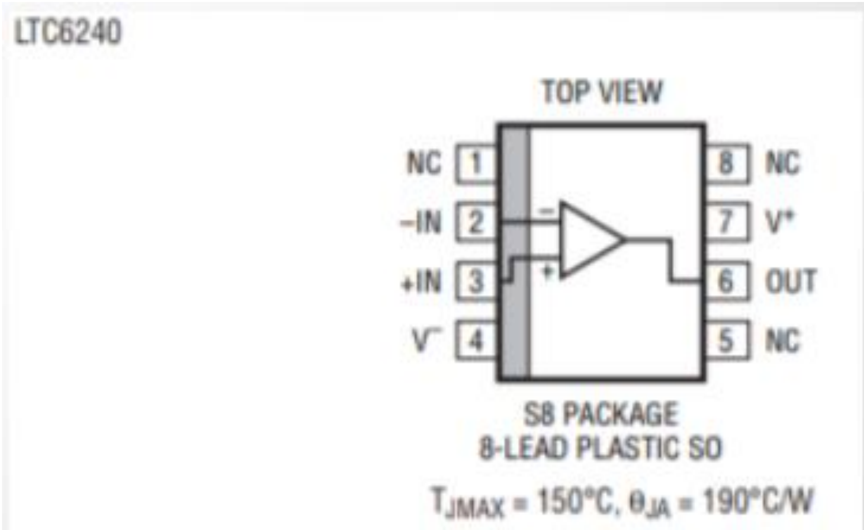


Figure 20 Opamp connections [7]

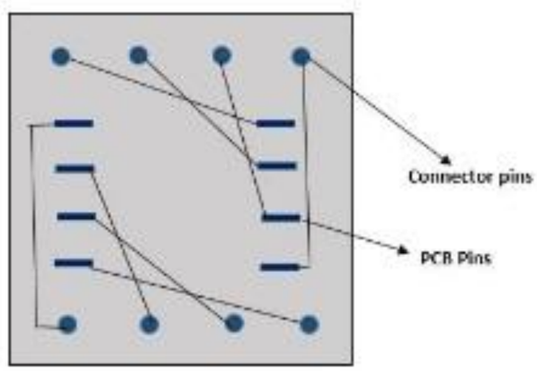


Figure 21 Connections between PCB pins and connector pins that connect the PCB to the breadboard

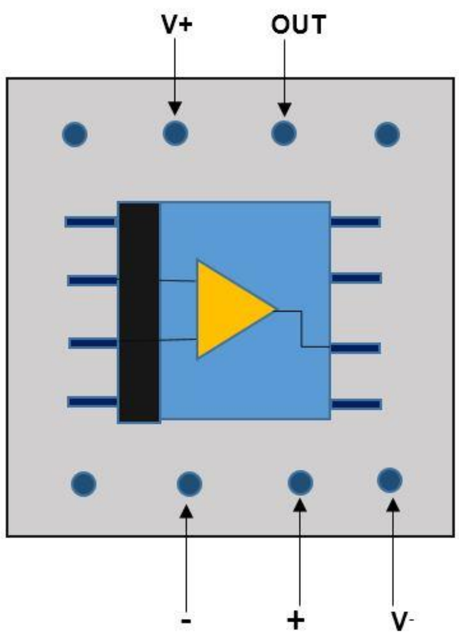
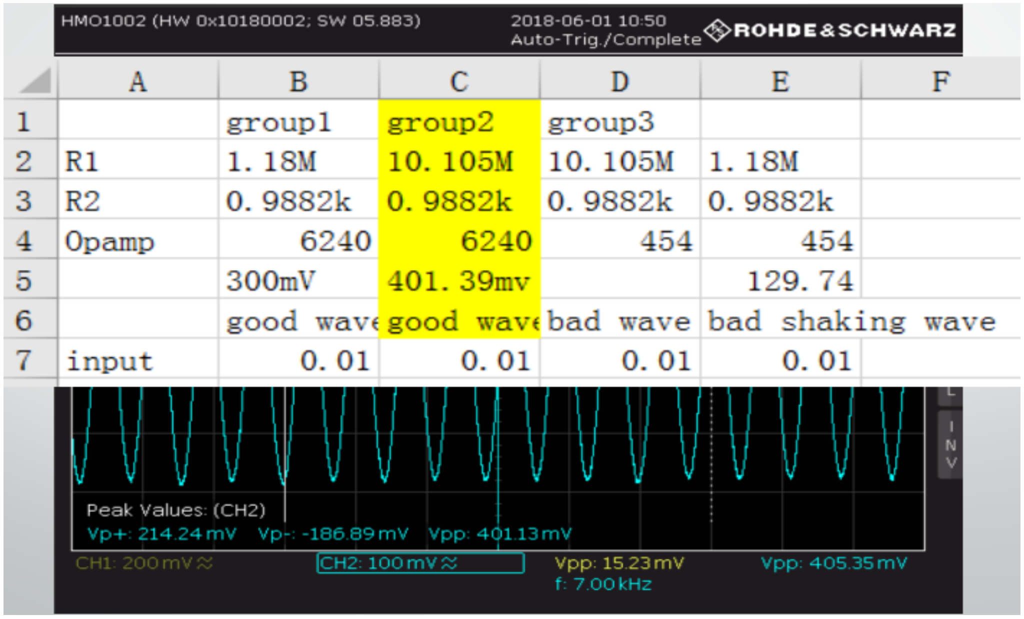


Figure 22 Connection relations between op-amp and PCB board

Two op-amps, LT6240 and 454 with different resistor combinations were tried to test and obtain the combination which would maximize the voltage gain in the circuit. Figure 23 depicts three major result groups, in which output voltage has been shown for a set of resistance values for both the resistors used in the circuit, the input signal voltage of 0.01 V and op-amp's used.



	A	B	C	D	E	F
1		group1	group2	group3		
2	R1	1. 18M	10. 105M	10. 105M	1. 18M	
3	R2	0. 9882k	0. 9882k	0. 9882k	0. 9882k	
4	Opamp	6240	6240	454	454	
5		300mV	401. 39mv		129. 74	
6		good wave	good wave	bad wave	bad shaking wave	
7	input	0. 01	0. 01	0. 01	0. 01	

Figure 23 Three major result groups [7]

It turned out that the output voltage was maximum for the values of group 2 as highlighted in figure 23. Then for the selected group of values, the effect of distance between the sender and receiver to the output voltage was interpreted for different dielectric materials through which the signal was travelling. The parameters for this testing were as follows: Vcc: 3V, Vee: -3V, R1: 0.9882kOhms, R2: 10.105M Ohms and a sin wave of frequency 7kHz was input, the input voltage being 10V.

Figure 24 depicts the change in output as the distance between sender and receiver increases. The unit for output voltage is milliVolts (mV’s) and that of distance is centimeters (cm’s) in all the scenarios. The dielectric material through which the signal is passed, in this case, is air.

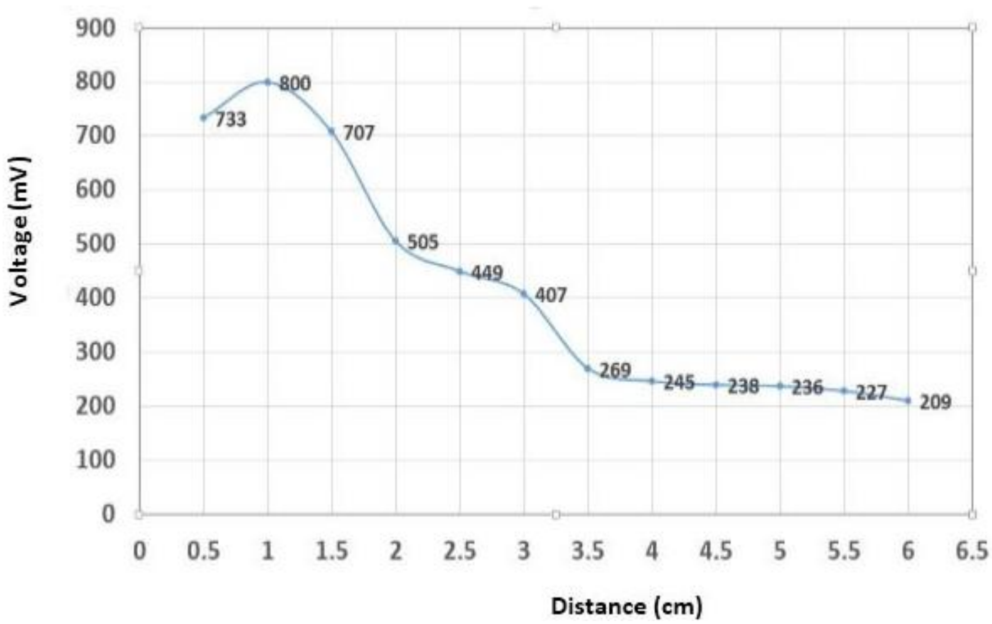


Figure 24 Change in Output with the change in distance between the sender and receiver, when dielectric material is air

Figure 25 depicts the change in output when the dielectric material is resin (a ruler was used for experiment)

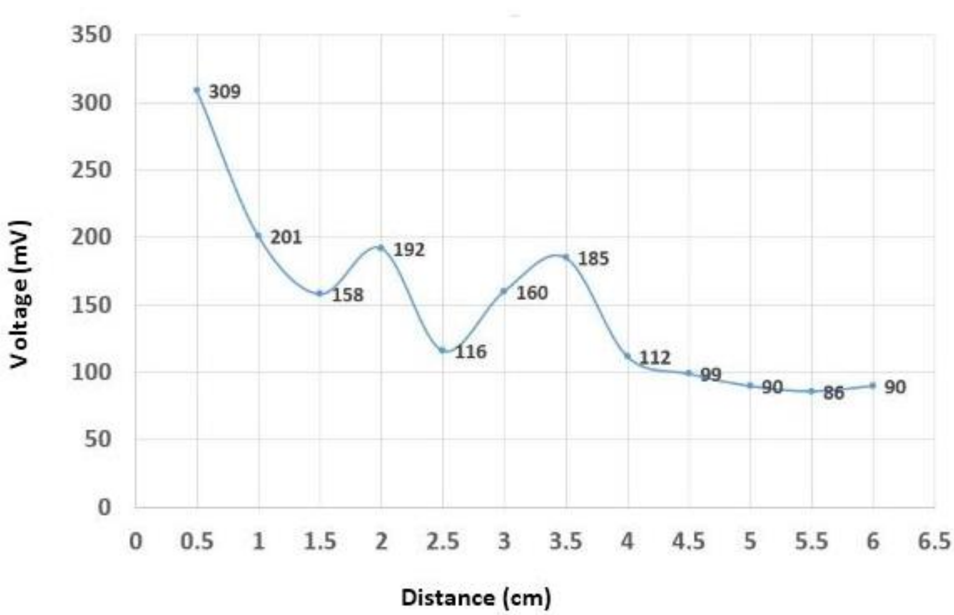


Figure 25 Change in Output with the change in distance between the sender and receiver, when dielectric material is resin

Figure 26 depicts the change in output when the dielectric material is metal (back of breadboard was used for experiment)

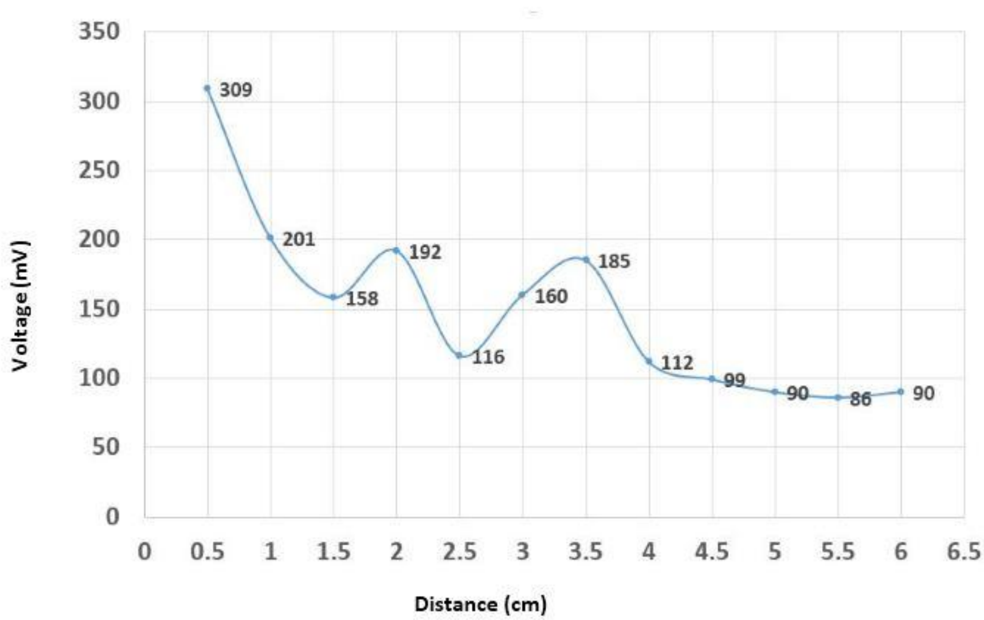


Figure 26 Change in Output with the change in distance between the sender and receiver, when dielectric material is metal

It was found that the change in voltage in all the three cases was non-uniform. So, a Do it yourself (DIY) holder was deigned to accurately maintain the required distance between the sender and the receiver and therefore avoid the noise which was being produced because of the unsteady movement (increase/decrease of distance) of the PZT's. Figure 27 shows the holder design.

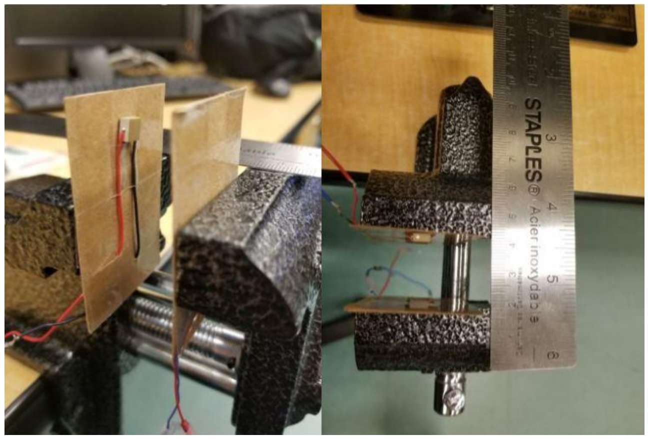


Figure 27 Holder Design

The goal was that the receiver should be able to sense weakest of the signals. So, the purpose was to amplify the received signal so that a significant output could be obtained for interpretation. Hence, we tried to look for op-amp with higher open loop gain but could not find a good option. So, a multistage voltage amplifier was designed using multiple LTC6240.

A two stage voltage amplifier, using two LTC6240 inverting amplifiers was used and the circuit has been shown in figure 28.

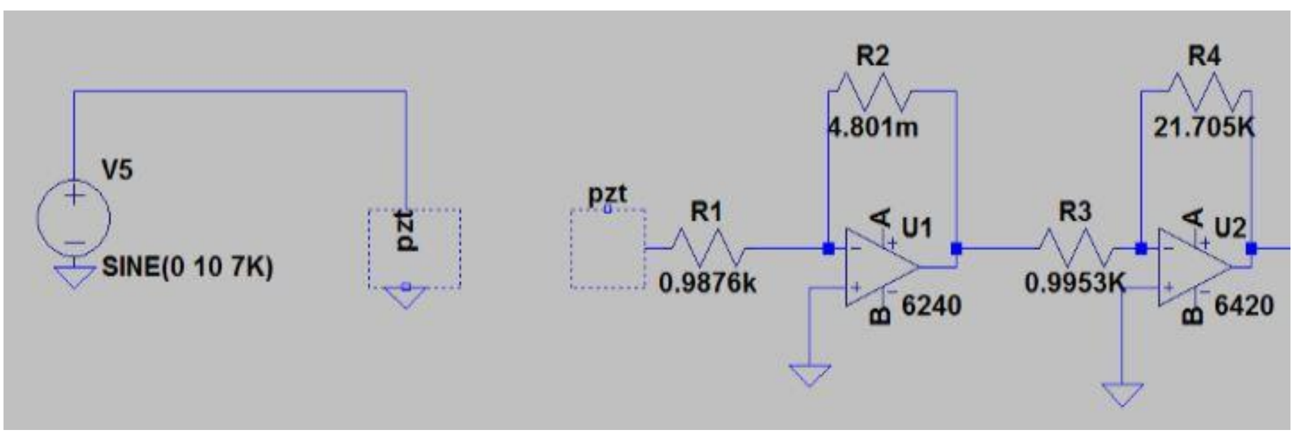


Figure 28 Circuit diagram of the two stage voltage amplifier [7]

The actual circuit on the breadboard is shown in figure 29.

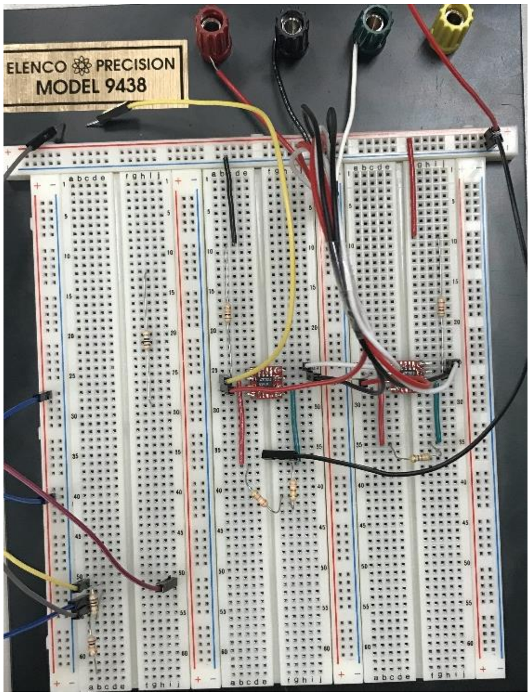


Figure 29 Actual 2 stage amplification circuit on the breadboard

A ±5V was supplied to the opamps. For the resistor values being R1 = 0.9876kΩ, R2 = 4.801mΩ, R3 = 0.9953kΩ, R4 = 21.705kΩ, the second stage gain was 2. The change in output voltage with the change in distance between the sender and receiver, with the two stage gain, has been shown in figure 30.

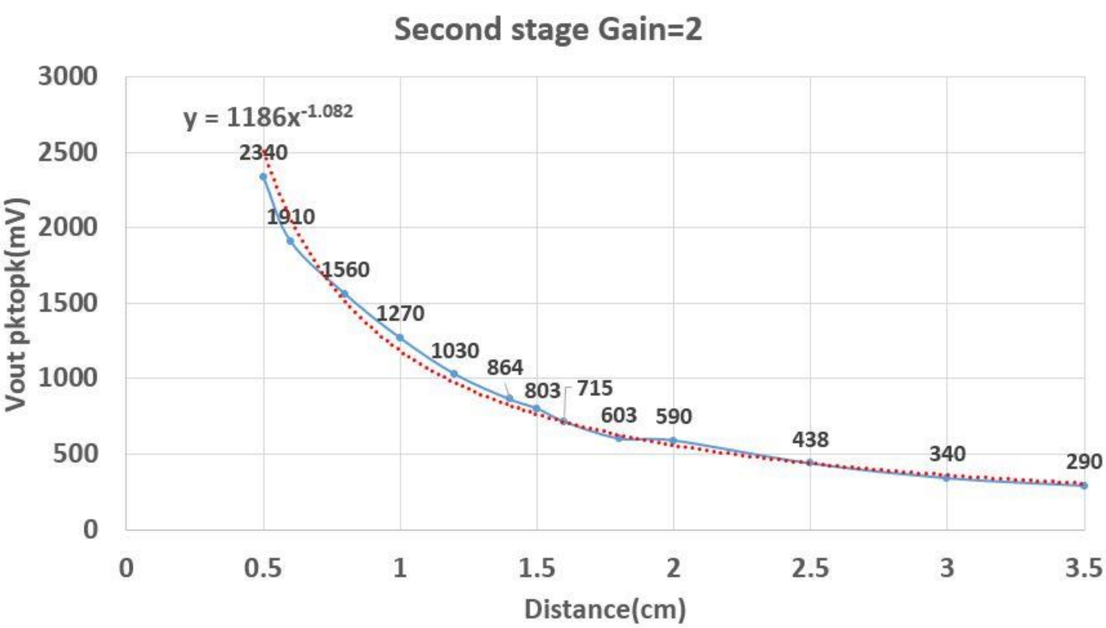


Figure 30 Change in output voltage with change in distance, second stage gain = 2 [7]

Then a three stage amplifier was built with the third stage opamp being TL072 in the inverting mode and other two being LTC6240 in inverting mode. The circuit diagram has been depicted in figure 31.

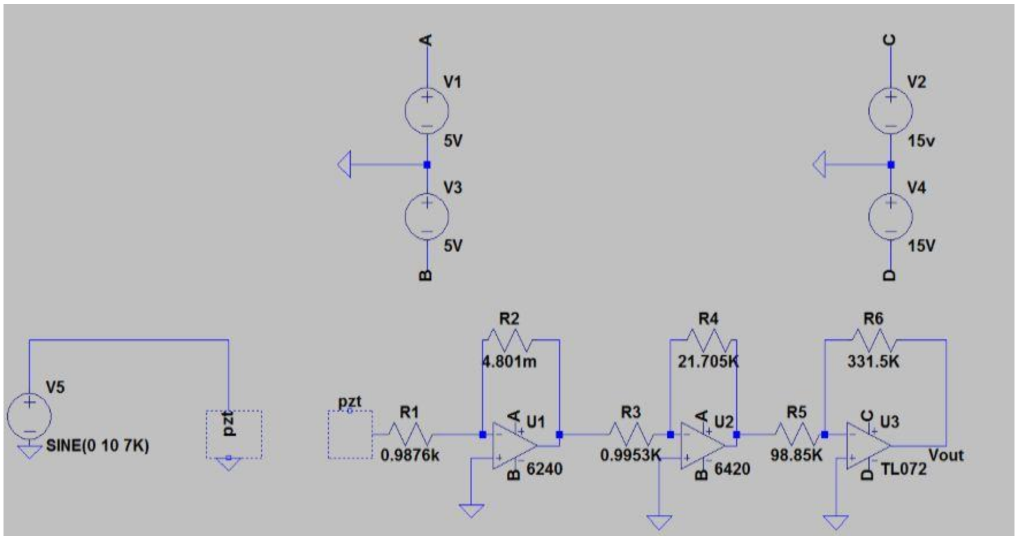


Figure 31 Circuit diagram of three stage voltage amplifier [7]

A ±15V was supplied to the third stage opamp. For the resistor values R1 = 0.9876kΩ, R2 = 4.801mΩ, R3 = 0.9953kΩ, R4 = 21.705kΩ, R5 = 98.85kΩ and R6 = 331.5kΩ, the third stage gain was 3.364. The change in output voltage with the change in distance between the sender and receiver, with the three stage gain, has been shown in figure 32.

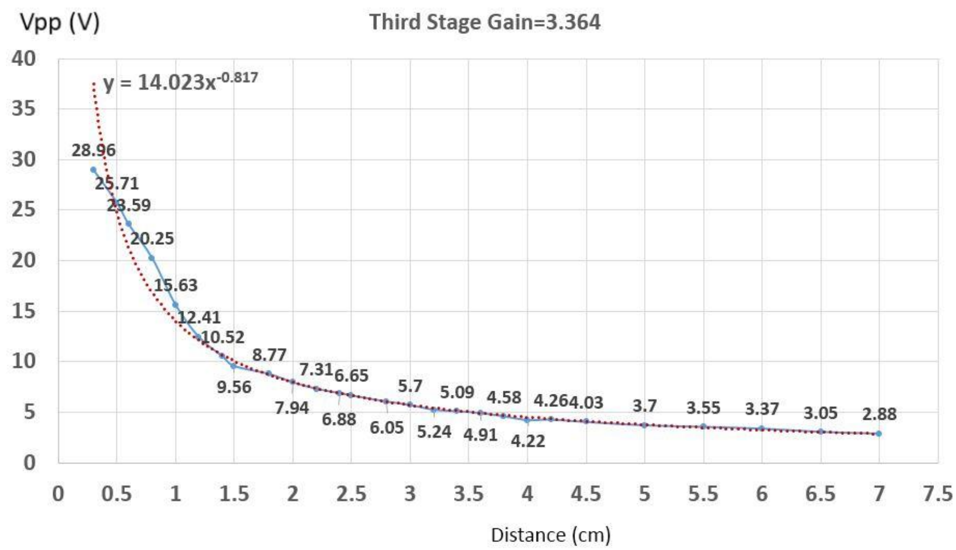


Figure 32 Change in output voltage with change in distance [7]

Chapter 7.

Testing the device

A function generator was used to supply signal to the PZT actuator. The receiver was connected to the amplification circuit using the probe station. The output of amplification circuit was fed to the oscilloscope to read the results. The whole testing setup has been depicted in figure 33.

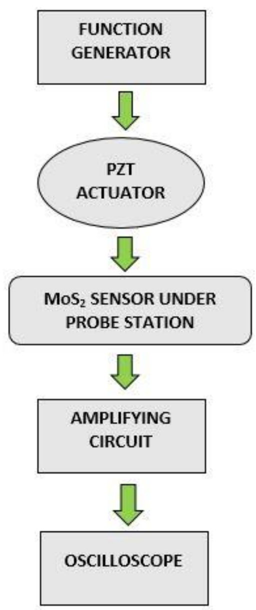


Figure 33 Testing setup

The receiver that was fabricated, was setup on a probe station in the measurement room as shown in figure 34.

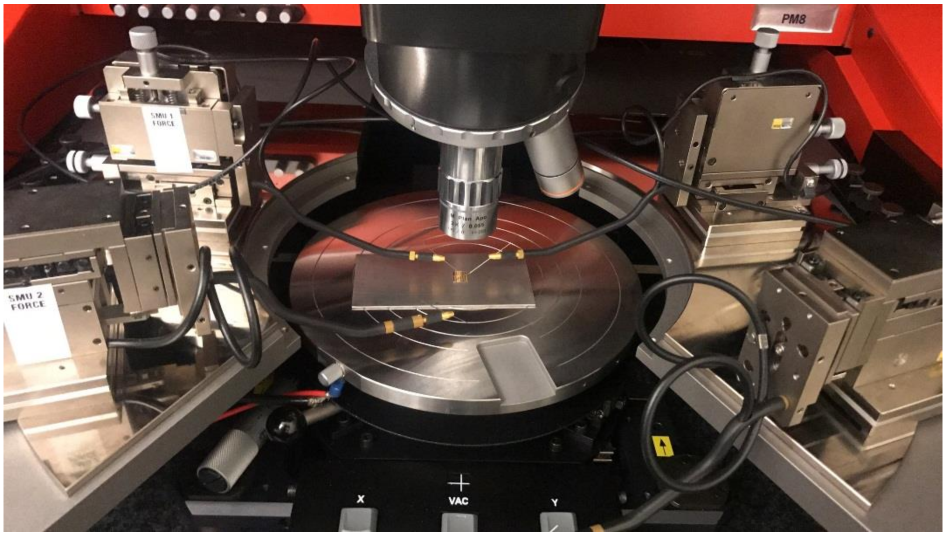


Figure 34 Setup of receiver on the probe station

Two probes were connected to the gold contacts of the device. The connections have been shown in figure 35.

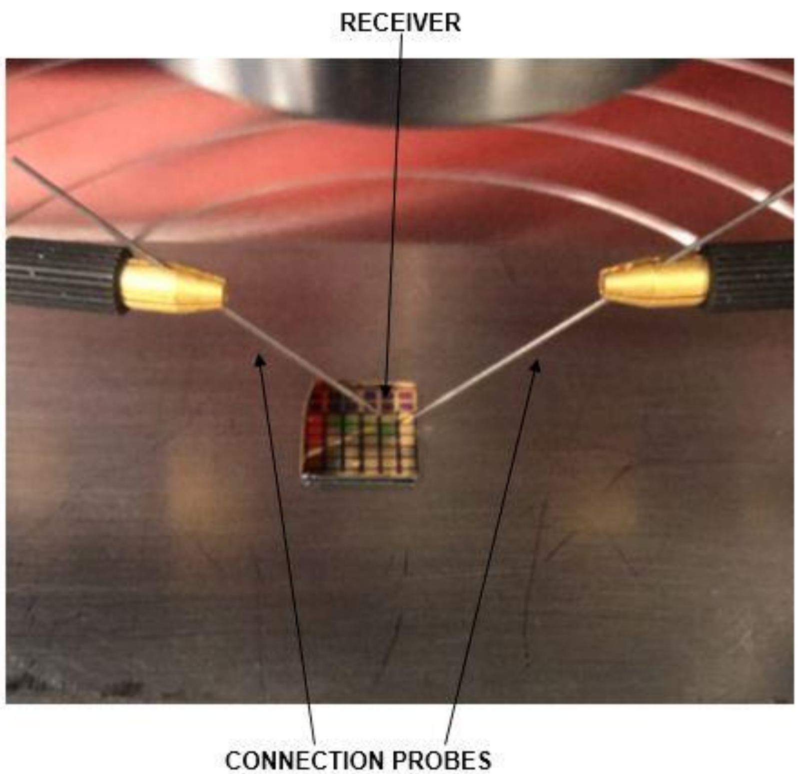


Figure 35 Probe connections

Necessary power connections and connections between the amplification circuit and oscilloscope were made. The circuit has been shown in figure 36.

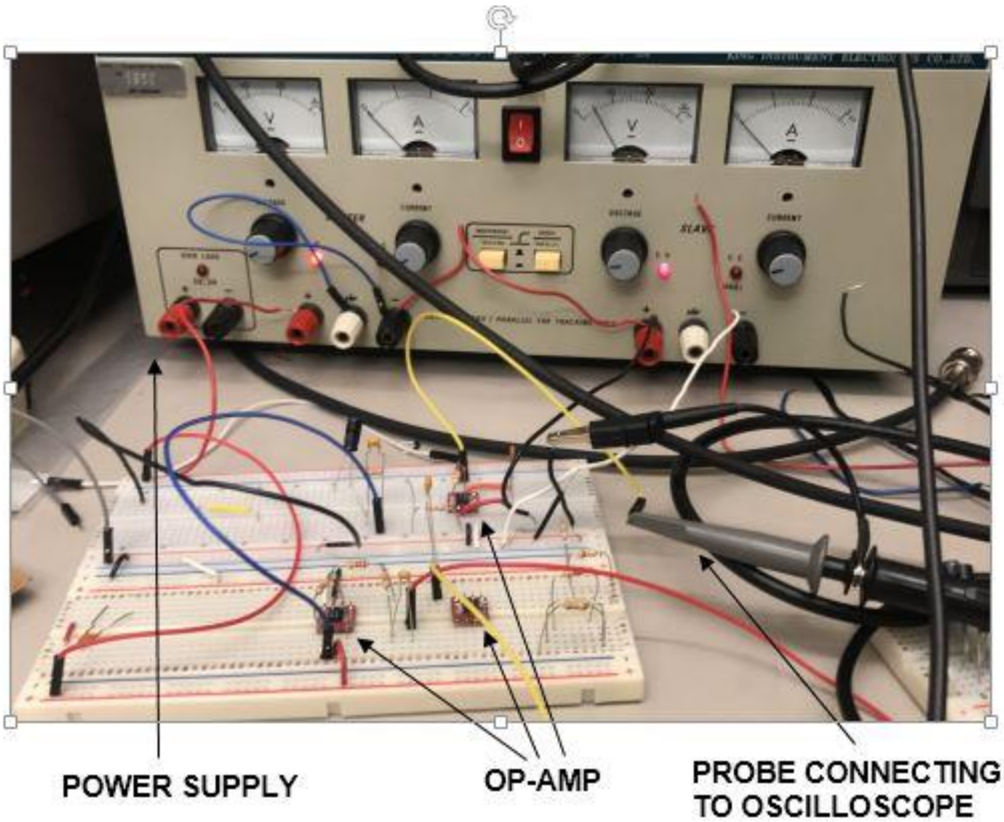


Figure 36 Amplification circuit and some of its connections

After everything was setup, a signal was supplied to the actuator and it was brought close to the receiver to see if the receiver receives any signal. The experimental setup is shown in figure 37.

Figure 37 PZT actuator taken close to receiver

After experimenting and checking the circuit, it was found that there were some issues with the circuit that needed to be resolved to obtain the results. So the team members who were focusing on the circuit design started working to optimize it.

Chapter 8.

Results and Future Work

The device with a few layers of MoS_2 has been successfully designed using the proposed fabrication technique and the IV curves confirm the electrical conductivity of the device. After the testing setup was ready, we tried to test our device using this setup with two stage amplifier in the measurement room as discussed in section VI. But during experimentation, we found out that there was some problem in the circuit and it needed debugging and optimization. The team members who are focusing on the testing circuit are working to optimize the testing setup. But unfortunately, the device that we fabricated has not been tested in this setup yet. So, the future work would be to test this device once the testing setup is ready.

If the results for the test are not as expected, the next step would be to reduce the number of layers of MoS_2 using oxygen plasma or other better techniques and then test the device. Also, the device does not have a completely free standing MoS_2 layer as the MoS_2 bends down in-between the gold contacts after some time. So, some improvements could be made in the fabrication technique in order to fabricate a device with free standing MoS_2 layer.

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