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Dispersing and Depositing MoSe2 onto Metal, Insulating, and Semiconducting Substrates via Voltage-Controlled Deposition Technique

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DISPERSING AND DEPOSITING $MoSe_2$ ONTO METAL, INSULATING, AND SEMICONDUCTING SUBSTRATES VIA VOLTAGE-CONTROLLED DEPOSITION TECHNIQUE

A Thesis

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by
Kalyan Chakravarthy Byrisetty
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Dedicated to my family

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ABSTRACT

In today's world, consumer electronics are getting smaller than ever. These reductions in size are preceded by advancements in electronic materials engineering and related fields. The need for materials that have various properties and are suitable for applications also necessitated research on different materials. Growing research in the applications of graphene led the way to the discovery of materials that have similar properties to graphene. In this way, the transition metal di-chalcogenides (TMDCs) came into use for electrical engineering. Techniques are required to put TMDCs into application, however here we are going to explain the vital focus of our research which is to discuss a successful method of depositing a solution of dispersed $MoSe_2$ using the voltage-controlled deposition technique. Here we are focusing mainly to find an inexpensive, simple and efficient method to deposit the TMDCs onto substrates.

Initially powdered $MoSe_2$ is dispersed in n-Methyl Pyrrolidone (NMP), and the dispersed solution is deposited onto different substrates. The different substrates that we used in this project were conducting substrate (Al foil), semi-conducting substrate (Si wafer), and insulating substrates (Glass, SiO_2, PMMA). At first, $MoSe_2$ was dispersed in NMP using the tip sonication method. Later, by using the voltage-controlled deposition technique the dispersed solution is deposited onto the aforementioned substrates. The final stage of the process is to analyze the deposition of $MoSe_2$ onto the substrates and etched substrates; we used Scanning Electron Microscopy and Raman Spectroscopy.

Through this research we have examined how $MoSe_2$ is dispersed well in NMP, and deposited onto bare and etched substrates as may be useful for future device building surface.

CHAPTER 1. OVERVIEW OF CHAPTER CONTENT

The aim of this chapter is to give a brief description of chapters 2 to 5 of this work.

Chapter 2 (“Motivation and Research Goals”) gives information about why this idea arose and what inspired us to undertake this work. Our goal is to disperse the transition metal dichalcogenide that is $MoSe_2$ in n-Methyl Pyrrolidone, and to deposit the dispersed $MoSe_2$ onto various substrates such as conducting substrate (Al foil), semi-conducting substrate (Si wafer), and insulating substrates (Glass, SiO_2, PMMA) using the voltage-controlled deposition technique.

Chapter 3 (“Introduction and Literature Review”) gives a brief description of the transition metal dichalcogenides (TMDCs), their growth techniques, properties, applications, and characterization techniques. Initially, some of the techniques involved in the growth of TMDCs are explained; this is followed by details of the properties of the TMDCs. Then the applications of TMDCs are explained in detail, considering the individual application and the use of TMDC. The final part explains how TMDCs characterization techniques are used.

Chapter 4 (“Voltage Controlled Deposition of Dispersed Transition Metal Dichalcogenides (TMDCs)”) discusses in detail the process of depositing dispersed $MoSe_2$ using the voltage-controlled deposition technique onto various substrates such as conducting, semiconducting and insulating materials. This chapter consists of six parts: (1) dispersing $MoSe_2$ using NMP, (2) depositing dispersed $MoSe_2$ using voltage-controlled deposition technique, (3) characterization of deposited $MoSe_2$ films, (4) etching of substrates and characterization of deposited $MoSe_2$ films on etched substrates, (5) thickness measurement of deposited films, and (6) conclusion.

Chapter 5 (“Summary and Future Work”) gives a brief description of all the work done to deposit the $MoSe_2$ using the voltage-controlled deposition technique onto different substrates. This method can be used to deposit other TMDCs onto the substrates, which could lead to applications such as using TMDC contacts in transistors.

CHAPTER 2. MOTIVATION AND RESEARCH GOALS

TMDCs have extraordinary electrical and optoelectronic properties which make them applicable in various ways including for use in solar cells, super capacitor electrodes, field effect transistors, flexible electronics, and photodetectors. $MoSe_2$ has a bandgap ranging from visible region to near infrared region. Some of the electronic properties TMDCs exhibit are semiconducting, superconducting, metallic and half-metallic. To know the potentiality of TMDCs in applications, they need to be deposited onto the substrates such that they can be used in devices. Therefore, in this project, $MoSe_2$ as a TMDC has been deposited onto various substrates which are conducting, semiconducting, and insulating.

To attain a better solution for the above, we were driven to conduct research on the TMDCs and found a better way to deposit the TMDC materials. The material which we have chosen here is $MoSe_2$ because of its interesting properties with respect to electronic devices. The first task we needed to undertake was to disperse $MoSe_2$ using n-Methyl Pyrrolidone (NMP). Then, we deposited dispersed $MoSe_2$ onto different substrates such as conducting substrate (aluminum foil), semi-conducting substrate (bare silicon), and three insulating substrates (silicon dioxide, glass sample, and poly methyl methacrylate) using a voltage-controlled technique. The last step was to characterize and analyze the $MoSe_2$ deposited substrates using Raman spectroscopy and Scanning Electron Microscopy (SEM).

The main aim of this work was to find an inexpensive, facile technique and reliable way to deposit the TMDCs onto the aforementioned substrates.

CHAPTER 3. INTRODUCTION & LITERATURE REVIEW

3.1. Background and Overview

The increase of research in the field of graphene is due to its remarkable electronic properties for a zero bandgap. This has motivated investigation to find semiconductors that are similar to graphene but have a bandgap. This has paved the way for research in transition metal di-chalcogenides (TMDCs). TMDCs came into use in electrical engineering because of their useful electronic and optical properties. The weak Vander Waals interactions between these TMDCs allow them to be used in the creation of electronic and optoelectronic devices [1]. In TMDCs, one transition metal atom is sandwiched between two chalcogen atoms; weak Van der Waals forces are experienced between the layers of atoms, but in the layers, bonds are strong. The formed MX_2 is a semiconductor where M is a metal and X is a chalcogen [2]. The single layer TMDCs have direct bandgap where as multiple layer TMDCs have indirect bandgap. The bandgap of TMDCs lies in the visible and near-IR range.

TMDCs can be used in optoelectronic applications such as lasers, phototransistors, light-emitting diodes, photodetectors, and electroluminescent devices because they are semiconductor materials [3]. The important characteristics of TMDCs are atomic arrangement, chemical composition, dimensionality, and structure. A wide variety of methods are used to synthesize the 2D TMDCs because the methods provide the best results in fabrication of required nanosheets. The crystals of these TMDCs have hexagonal structures where metal atoms are sandwiched between chalcogen atoms; the bonding between them is weak due to Van der Waals forces [4]. GaAs and Si are typically used as solar absorbers, but by using TMDCs there is a tremendous increase in absorption of sunlight per unit volume as compared to GaAs and Si, and with less than 1nm thickness TMDCs can absorb 5-10% of incident sunlight. Atomic layered thickness materials can also be fabricated using TMDCs. The electronic properties of these TMDCs can be tuned by doping them with other molecules or atoms [5].

TMDCs also possess many other interesting features because of characteristics such as atomic thinness, high electron mobility, and flexibility. A broad range of electronic properties are shown by the TMDC compounds such as metallic, semiconducting and superconducting behaviors [6]. Of the TMDC compounds that have semiconducting behavior some are used in device applications because of their peculiar physical properties including the electronic and phonon structure which are dependent on thickness, symmetry, effects of quantum confinement, and crystal structure. Several techniques and methods have been proposed for the preparation and design of the TMDC heterostructures but they are still in their early stages. Due to miniaturization, silicon-based devices are being developed to keep up with state of the art progress, but the low power and ultra-small transistors which are made from the TMDCs are more efficient. TMDCs can be deposited onto flexible devices and have similarities to silicon with on/off ratio and high carrier mobility, and they also have the bandgap in the visible region to near IR region [7]. To develop TMDCs with defect free atomic layer thickness and in a large scale on the desirable substrates is a challenging task. For many optoelectronic applications, the direct bandgap of the TMDCs also results in photoluminescence. There are many layered related properties which means the properties depends on the layers of the material where features of the single layer TMDCs change with the properties of the multiple layer TMDCs [8].

Table 3.1 Characteristics of $MoSe_2$ material bandgap and type.

	Di-Sulphide (S_2)	Di-Telluride (Te_2)	Di-Selenide (Se_2)
Molybdenum (Mo)	Semiconductor Direct Bandgap (single layer) at 1.8 eV Indirect Bandgap (multiple layer) at 1.2 eV	Semiconductor Direct Bandgap (single layer) at 1.07 eV Indirect Bandgap (multiple layer) at 0.82 eV	Semiconductor Direct Bandgap (single layer) at 1.55 eV Indirect Bandgap (multiple layer) at 1.1 eV

3.2. TMDCs Growth Techniques

Recent growth of study in the field of TMDCs urged the research for the growth techniques. Here, we are going to discuss some of the techniques that are usually used to grow TMDCs. These include chemical vapor deposition (CVD), exfoliation, pulsed-laser deposition, molecular beam epitaxy, laser thinning, atomic layer deposition, and magnetron sputtering. Hence, we will discuss a few of the techniques mentioned above.

3.2.1. Chemical Vapor Deposition (CVD) Technique

Chemical vapor deposition (CVD) is one of the most prominent techniques that used to deposit TMDCs on the substrates. The target substrate and precursor powders are placed in the crucibles in a furnace. The transition metal used may be pure metal or the metal combined with oxides. The main goal in this process is maintaining temperatures and the distance between the sources and the substrates. In this method, the forerunner powders are placed beside the substrate, the transition metal powder is placed near the substrate and the chalcogen powder is placed near the transition metal powder. When the temperature is maintained at a level of 800 ^{o}C – 1100 ^{o}C, the powders of the transition metal and the chalcogen turn into vapors. The inert gas is allowed to pass through the furnace and carries the vapors of the transition metal and chalcogen to the substrate [9-11]. In this method the growth of the TMDCs can be maintained by changing the temperature, and monolayers of TMDCs are formed. To improve the layer quality of the TMDCs, seeding promoters are helpful. The selection of the substrate will also enhance the deposition, and depending on the substrate selected, the TMDCs deposition rate increases.

3.2.2. Pulsed Laser Deposition Technique (PLD)

This technique is not widely used to produce TMDCs but it is also one of the ways to create layers of TMDCs. In this process, under controlled conditions, the atomic species of the material is transferred atomically from bulk crystal to a thin film. Some of the variables used to determine the PLD film, chemical composition, and resultant stoichiometry are gas pressure, laser fluence, and deposition temperature. In this process, the interaction between the film and substrate is important; this interaction also changes the physical property of the deposited material. PLD offers better interaction between the film and substrate and also develops a better structure. The thermal evaporation during this PLD process helps atomic species to form films with high energy throughout the surface. Under controllable conditions, the film texture can be developed with the help of ion bombardment with the surface. The kinetic energy of the depositing atomic particle of the material can be tunable, providing possibilities for the structure formed as required. With this tunable kinetic energy, the dynamics of growth and nucleation can be controlled. In PLD, it is easy to control the stoichiometry due to use of a single target. In this process, the pure material powder is used as precursor without using the compound itself which constitutes a target as one of the contents. In this process, the material is placed on one holder and the substrate is placed on another holder. Under deployed conditions, the laser strikes the material, and then the material is vaporized and forms films on the substrate. Film thickness can be controlled by controlling the growth time [12-14].

3.2.3. Atomic Layer Deposition Technique

Atomic layer deposition (ALD) is a technique which is used to deposit TMDCs onto the substrates. In this process a single atomic layer is deposited onto the substrates at a time. The advantages of the ALD process are reproducibility, high conformity, high density, wafer-level thickness uniformity and controllability. High precision in the thickness of the TMDC layer can also obtained in this ALD process. ALD is one of the best techniques because its atomic composition and crystallinity can be changed. The thickness of the TMDCs can also be controlled by this process. The substrate that we use in this process is hydroxyl terminated such that it combines with the metal and forms metal oxide. In this process, gas-phase metal precursor is introduced onto the substrate, then a chemical reaction takes place and metal oxide is formed. Later, the surface reaches saturation level depending on the reactivity, temperature, and concentration, and then the chemistry between atoms stops. After the metal oxide gets deposited onto the substrate, the first precursor is removed and the second precursor is loaded to the system. The second reactive precursor starts reacting with the metal oxide, which is pre-formed on the substrate, and again the chemical reaction takes place. The final compound would be the TMDCs and the remaining would be removed. In this way monolayers of the TMDCs are deposited onto the substrates [15,16]. In this way, the ALD process can be used to control the thickness of the TMDCs with growth time and can achieve high film growth rates.

3.2.4. Exfoliation Technique

The major challenge in the preparation of TMDCs is preparing thin 2D atomic thickness TMDCs in the controllable production. Many methods have been used to successfully fabricate single layer TMDCs. One of the methods used in this fabrication is the exfoliation method. There are several types of exfoliation techniques such as mechanical cleavage, chemical exfoliation, ion intercalation, and liquid exfoliation.

TMDC materials are particularly successfully exfoliated using the mechanical exfoliation technique. which simply involves using Scotch Tape This process is simple and can be done in air. This method is used to exfoliate bulk materials to produce monolayers of the materials and is performed on the TMDCs [17,18]. This technique helped to fabricate TMDC layers onto the substrates, the layers that can be fabricated from single layer to few layers. In this method, the folding and unfolding of scotch tape several times onto the TMDCs, peels off the layers, which thin them down. These layers are then transferred to the substrates on which they need to be deposited. The ability to control the lateral size of the material and number of layers is unpredictable, making this method unsuitable for large-scale production.

The other method used for exfoliation is the chemical exfoliation process. In this process, the forces present in the TMDC material bonding are weakened by incorporating the small ions in to the interlayer spacing. When the sonication process takes place on this weakened TMDC solution, the layers are peeled, and the peeled layers remain in the solution. However, this process is difficult to maintain because it cannot form large single layers of the TMDC materials. By using this chemical exfoliation process, the TMDCs can be doped by adding the selected intentional impurities during this process. During this exfoliation process, there is a chance that the contaminants may attach to the surface layers of the TMDCs [19]. There is a process where the TMDCs with the help of water; it requires a water-soluble polymer which can help to promote the rate of exfoliation. By using this polymer, the exfoliation rate greatly increases, but this polymer may contaminate the layers of the TMDCs. Due to this contamination, the applications of the TMDCs becomes affected. The other method used in this exfoliation process is the ion intercalation method. In the ion-intercalation method the ions are driven into the layers of the TMDCs, which in turn form ion-intercalated compounds. The ions used most in this process are potassium, lithium and sodium ions. The next step is to sonicate the solution in which the ions are incorporated, this weakens the TMDC layers and produces the monolayers of the TMDCs. This process gives high yield and produces monolayers of required thickness [20].

3.2.5. Laser Thinning Technique

Laser thinning is a technique which is used to fabricate monolayer TMDCs. The controllability and reproducibility can be obtained during patterning the desired shapes of the TMDCs. It is a top-down approach in which the bulk crystal of TMDC is taken and brought down to a single layer with the help of a laser. A laser is used to thin down the layers of the TMDCs. The laser that is used in this process is a scanning laser used in Raman spectroscopy; the laser wavelength used in this method is around 500nm. Then the laser is focused onto the material to thin it down to individual monolayers. The laser is moved around the material to achieve the range of layers required. The last layer can withstand high powers of the laser because it is in direct contact with the substrate. The laser irradiation creates consecutive reduction in the thickness of the TMDC. During this process we can measure the thickness before and also in between the process with the Raman spectroscopy. The heat induced by the light absorption sublimates the upper layers and thins down the layer of the materials. By this method we can reduce the layers of bulk TMDCs. However, the layers may get damaged and leave some residue because of the high power used during this thinning process. The surface also remains rough due to this process [21-23].

3.3. Properties of TMDCs

The properties of TMDCs can be changed completely or become affected due to the processing steps of the standard semiconductor. Many techniques have been used to find the structural properties of the TMDCs, including Raman spectroscopy and Scanning Electron Microscopy. TMDCs with single layers or multilayers possess unique characteristics and have catalytic, electronic, optical, and photovoltaic [7] properties. The TMDCs are formed by sandwiching transition metal between two chalcogen layers. All transition metal combinations with chalcogens form over 40 different compounds. The position of the chalcogen with respect to the transition metal decides the structure of the TMDC. There are only two polytype structures for single layer TMDCs. Multiple polytype structures can be formed for multi-layer TMDCs depending on how the layers of the TMDCs are stacked. Different structures, such as trigonal prismatic or octahedral can be formed by TMDCs depending on the arrangement of atoms.

TMDCs provide a high scope of bandgap because they range from visible to infrared. Monolayer TMDCs have direct bandgap whereas multiple-layer TMDCs have indirect bandgap. TMDCs are very strong, mechanically flexible and efficient in electrical transport. TMDCs that are defect-free, high crystallinity, atomically thin and have no stacking faults [24]. The layers of the TMDCs are isolated because of the weak van der Waals forces of attraction. Quantum confinement and symmetry change also defines the electrical properties of TMDCs, so the electrical properties depend on the number of layers in the TMDCs. The electronic properties of the TMDCs are tunable, and there are many materials in the TMDCs which can be used in various applications [25].

Due to the reduced dimension size, there are extraordinary physical properties in the monolayer of TMDCs.

The spin-valley coupling between the atoms of TMDCs allows them to have different electronic structures which makes them useful in valleytronics applications. In single layer TMDCs, a strong photoemission is observed. The substrate selection also plays a significant role because extrinsic scattering may occur and the physical properties, electronic properties may become degraded [26]. TMDCs with single layers sustain more strain than the TMDCs with multiple-layers [27]. By improving the optoelectronic properties using uniaxial strain on TMDCs which increases the use of TMDCs in optoelectronic devices. Properties such as interlayer excitons and negative differential resistance in the hetero-structured TMDCs helps to design novel devices [28].

Enhanced photoluminescence is observed in single layer TMDCs because they have a direct bandgap. Carrier concentration, strain, number of layers and temperature can change the phonon structure of TMDCs. The properties of TMDCs such as lattice vibration and band structure make TMDCs useful in many device applications. The excitonic effects dominate the optical properties of TMDCs because of the reduced dielectric screening and charge confinement. TMDCs can possibly control the valley states and spin of single confined holes or electrons which makes them suitable for valleytronics applications [29]. The properties such as high current on-off ratio, valley-dependent physics, piezoelectric coupling and interaction with light are obtained from the monolayers of TMDCs. The properties of TMDCs can be modified or changed by straining, interfacing with other materials, doping, and surface adsorption. In the monolayer TMDCs if the interlayer interaction is absent, then the charge carrier distribution rises. The individual layers of the TMDCs cleave easily because the interlayers are bonded by the weak van der Waal forces. A single layer of TMDCs has a thickness of 0.6nm – 0.7nm in a hexagonal arrangement. From the changes in absorption spectra, photoluminescence, and photoconductivity, it can be observed that there is an increase in bandgap energy and change in bandgap from indirect to direct. The band structure feature of TMDCs can be shown with the help of photoconductivity. Atomic doping is the process in which the dopants are added to the TMDCs. The dopant and its concentration which is doped with TMDCs can change the properties and structure of TMDCs.

The benefit of doping TMDCs is the change in frictional characteristics, which is good for lighter transition metals. By doping with the transition metal, it improves the tribological properties and magnetic properties. The TMDCs can have electronic properties which are available in metallic, semiconducting and insulating. One of the unique properties of TMDCs is their stability in air. Consistency, high quality production, and reproducibility of TMDC layers allow them to integrate into very large size circuitry. In today's semiconductor industry, it is a requirement for methods that enable fabrication on specific locations of the substrates that the methods should be scalable and reliable. The impurities present in the material can also affect the properties of TMDCs. In hydrogen evolution reaction, TMDCs can be used as catalysts because of this properties [30].

3.4. Applications of TMDCs

TMDC materials have diversity in their band structure because they have a direct bandgap in monolayer structure and indirect bandgap in multiple layer structure. Due to this, TMDCs have unique physical and electronic properties, which increases the use of TMDCs in the applications of many devices such as photonics, energy and sensing devices. TMDCs also have other properties such as tunable electronic structure, high electrostatic efficiency, sensor sensitivity, mechanical strength, and optical transparency which makes them viable for many devices. TMDCs are used in flexible technologies such as sensors, solar cells, energy storage devices, displays and transducers. All these devices possess large areas. In digital electronic devices, there is a need for materials with high on/off current ratio. TMDCs are such devices with the high on/off current ratio property, which is highly desirable for digital electronic devices. As a result, TMDCs are used to produce transistors and many other devices. Monolayer TMDCs have high exciton binding energies, photoluminescence and electromagnetic spectrum which make them more useful for optoelectronic applications. The direct bandgap of monolayer TMDCs makes them more suitable for optoelectronic devices.

3.4.1. Field-Effect Transistors

The absence of short-channel effects in TMDCs [31] makes them suitable for use in transistor production. In electronic circuits, transistors are one of the basic devices. A field-effect transistor (FET) consists of a source, drain, gate and a channel that connects the source and drain. A transistor requires high on/off ratio because when a transistor is used as a switch, in the ON state it is highly conductive and in the OFF state it is in open position.

In transistors, a dielectric layer from a gate electrode is used to separate the source and drain electrodes. TMDCs are used as a semiconducting channel between the source and drain. The plane of the material where the scattering and transport of carriers are enclosed in TMDCs. The layer thickness determines the effect of the scattering mechanism on the carrier mobility. With an increase in temperature the phonon scattering also increasingly affected the carrier mobility [32]. Charged impurity limits the carrier mobility when there is low temperature. In the local environment, random perturbations affect the performance of single layer TMDCs. A semiconducting TMDC can achieve high mobility when it is encapsulated by an insulating material. Single layer TMDC conductance increases as temperature decreases. Flicker noise can arise in TMDC incorporated transistors when they are in the local environment, and they can limit the performance.

3.4.2. Photodetectors

Semiconductors have an exciton binding energy which creates free carriers when the incident photon energy exceeds the bandgap of the semiconductor. Depending on the number of TMDC layers, the wavelength of the photodetection sensitivity can vary. Photodetectors have many uses including biological diagnostics, metrology, imaging, and remote sensing [33]. The structure of a photodetector is metal-semiconductor-metal, where a single layer of TMDC semiconductor is used to design this photodetector. Photodetectors are used to convert the incident photons into light energy. Photocurrent can be generated by applying an electric field to separate the bound excitons. Phototransistors and photodiodes are some of the photodetectors designed using semiconductors. The presence of weaker bound excitons makes TMDC devices have faster responses. Single-layer MoS_2 has higher sensitivity towards green light and the double layers have greater sensitivity towards red light [34]. The photodetectors which are designed using hetero-structures of TMDCs have an excellent photoresponsivity. The photocurrent measured for a photodetector is designed using TMDC semiconductors; first the photons were made incident on the semiconductor and the photos contained the energy of more than the 1.9 eV bandgap [35].

For photocurrent generation, there is need of carrier separation and speculation of interband absorption, which is explained in the absorption spectrum, and photocurrent follows it. When the photo-generated carriers are collected at a shorter distance and also separated by an electric field, then the photodetector will show a short response time. To improve the performance in photodetectors, Schottky barrier modulation is required between the electrodes and TMDCs. The photo-resistivity and mobility can be improved by a self-assembled doping method [36]. To absorb different wavelengths of light, we can tune the phototransistors by changing the thickness of the TMDC semiconductor layer.

3.4.4. Supercapacitors

Supercapacitors are devices used to store high power densities. Supercapacitors must have an excellent term-to-term stability, high power intake and delivery, and high power density [37]. TMDCs can be used in the design of supercapacitor because of their high electrical conductivity and surface area. Due to the properties of TMDCs, they are used in phototransistors, sensors, electrochemical storage systems. So, the TMDC materials are used to make electrodes for the supercapacitor applications. Supercapacitors are also known as electrochemical capacitors where they are classified into two categories: electric double layer capacitors and pseudo capacitors.

Electric double layer capacitors are supercapacitors where the charge is stored at the layer of electrode and electrolyte interface. Pseudo-capacitors are supercapacitors where the charge transfer takes place very quickly through redox reactions. TMDCs have high ranges of specific capacitance [38] because ion diffusion takes place more quickly between the layers due to large interlayer space and surface area.

3.5. Characterization Techniques for TMDCs Deposition

TMDCs are nanoparticle materials; when they are deposited onto the substrates we need instruments that are capable of characterizing these deposited substrates. Some of the instruments used to characterize the nanoparticles are discussed here; they are Scanning Electron Microscopy (SEM) and Raman spectroscopy. SEM is used to image the nanoparticle material, so here it is used to image the nanoparticle TMDCs deposited on the substrates. Raman spectroscopy is used to provide the spectra of the material deposited on the substrates. These characterization techniques are explained in detail below.

3.5.1. Scanning Electron Microscopy (SEM)

A Scanning Electron Microscope(SEM) is a device used to get 3-D images of the sample. The 3-D images consist of morphology and topology of the sample. When the beam with high energy electrons hit the surface of the sample then the SEM records the reading and creates the images. When the beam hits the sample, it generates signals that the SEM captures to create images of the sample. The data captured consists of texture, structure and chemical composition of the sample. If a particular area is selected from the sample, it can be scanned, and data can be gathered about the area from the signals generated from the sample.

The depth of field is high when using SEM, and the images can be viewed clearly. In SEM they use a beam of electrons, and to focus the beam condenser lenses are used. The lenses are used to provide magnetic fields and electric fields that focus the electrons. The resolving power can be limited by the scattering of electrons and the beam size. The following components are used in the SEM: anode, condenser coils, electron detectors, electron gun, stage, magnetic lens, and scanner. SEM requires a power supply which is stable, high vacuum system, and cooling system. The scattering of electrons takes place when an electron beam touches the surface of the sample. The scattering of electrons which occurs can be elastic and inelastic. When the energy of electrons transfers to the atom of the sample it is called inelastic scattering and when the electrons scattered with angle greater than 90^o it is elastic scattering. The elastic scattering is also known as backscattering.

The SEM process starts with the generation of high-energy electrons from the electron gun. The electron beam passes through the condenser coils, electromagnetic field lenses and then hits the sample. When the beam passes through these coils, it accelerates and later hits the sample with high energy. Once the beam hits the sample it produces secondary and back scattered electrons, auger electrons and x-rays. These generated signals are captured by the detector. These generated signals are used to produce the image of the sample. Different kinds of information such as size of the sample, texture, shape and composition can be obtained from the scattered patterns of the beam.

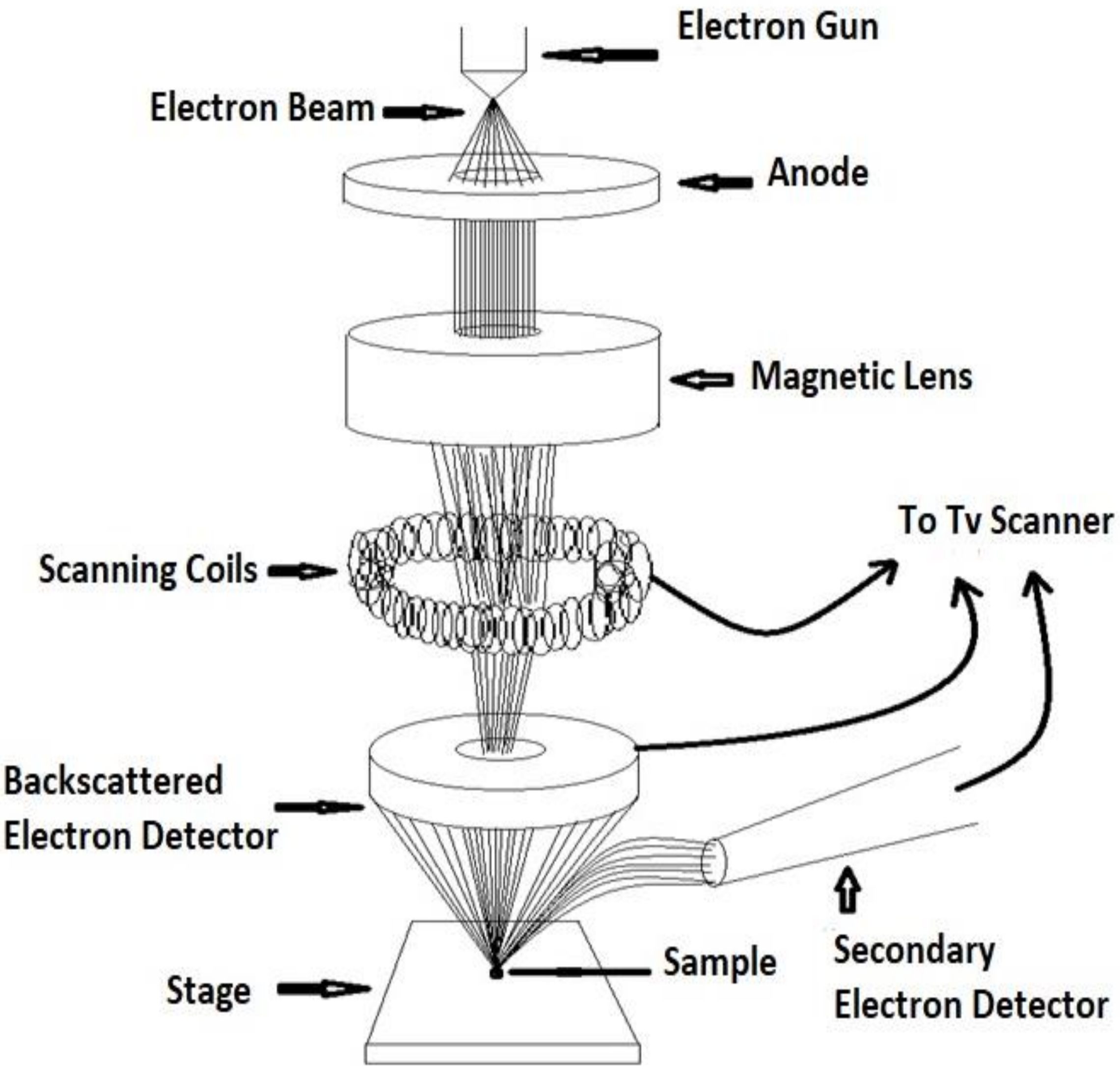

Figure 3.1. Scanning Electron Microscopy schematic diagram

SEM provides the most of the information related to the sample. Information such as mineral composition and element can be obtained from the x-rays that are produced during SEM process. Topographic information can be obtained from the back scattered electrons, and the contrast in the multilayer can be obtained from the secondary electrons. When all these signals are collected by the detector, these are converted into the electrical signals. Then these electrical signals are converted to the 3-D image of the sample.

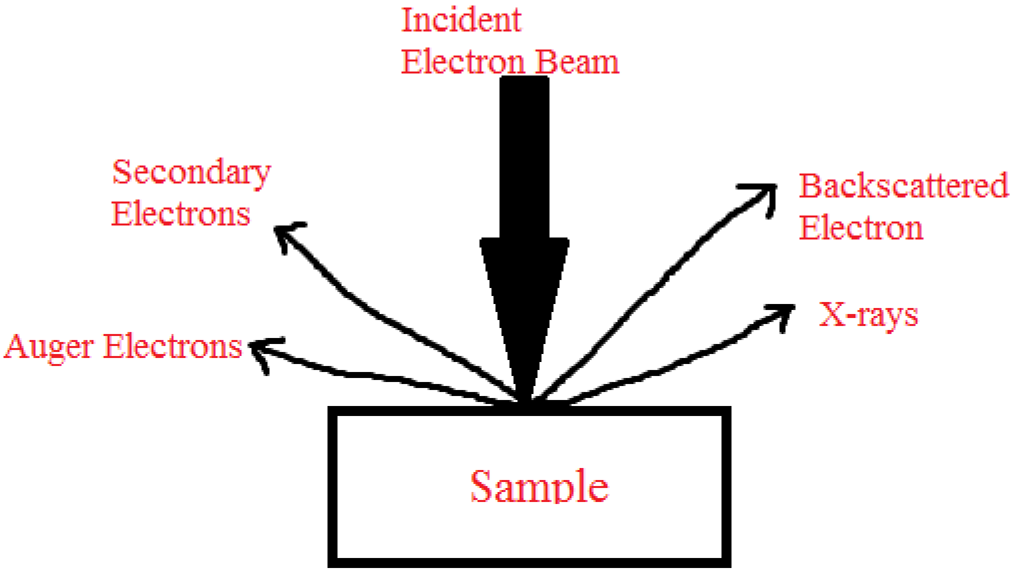

Figure 3.2. Generated signals when electron beam strikes the sample

By using SEM, samples can be imaged to different ranges of lengths and this process can be done extremely fast. It is easier to scan a conducting sample, but to scan a non-conducting sample it needs to be coated with a conducting layer before scanning. We can have a better resolution of image of sample by using SEM. SEM has some disadvantages, such as costly equipment, so operation requires experience. If the equipment gets damaged it is expensive to repair.

3.5.2. Raman Spectroscopy

Raman spectroscopy is a technique which is used find the unique structural finger print of the molecules. In this technique, a monochromatic laser beam is incident onto the material. When the incident light hits the molecules of the material, the energy of the incident light gets transferred to the scattered photons. The scattered photons can have the same, or higher, or lesser energy, wavelength and frequency when compared with incident photon. If the scattered photons have the same energy, wavelength, and frequency, then it is called as Raleigh scattering in the Raman spectroscopy. Raleigh scattering is known as elastic scattering. If the scattered photons have higher, or lesser energy, wavelength and frequency, then this type of scattering is known as Raman scattering. Raman scattering is known as inelastic scattering.

Raman Spectroscopy is mainly based on inelastic scattering. The change occurring in this scattering process is called the Raman effect. In the Raman spectroscopy techniques, when the laser beam is incident on the material it scatters photons. The scattered photons which have higher energy or lower energy then the incident photon are returned, and the scattered photons which have energy equal to incident photon is filtered out. In this inelastic scattering there is a difference in frequency which can be determined as the shift in energies that is helpful in identifying the materials with their unique wavelengths. In the inelastic scattering, if the energy of scattered photons is higher than that of the incident photon then it is called anti-stokes shift. In the inelastic scattering, if the energy of scattered photons is less than that of the incident photon then it is called stokes shift.

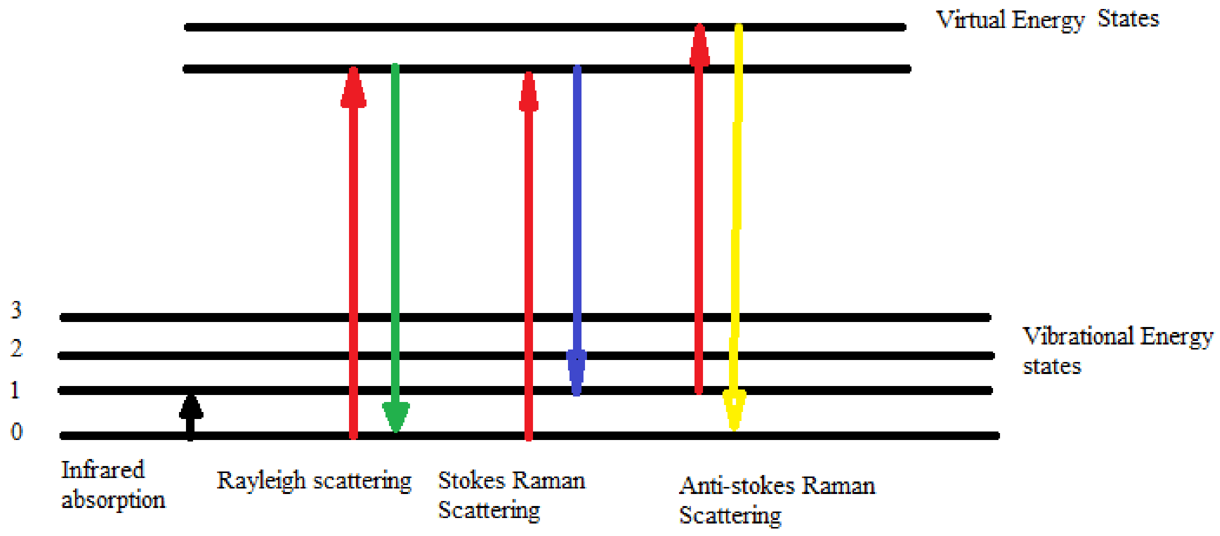

Figure 3.3. Raman Spectroscopy Energy level diagram

In Raman spectroscopy, the aim is to capture the photons that have similar energies than that of the incident photon. The main components in the Raman spectroscopy are laser source, beam splitter, wavelength selector, sample holder and charge coupled device. In this process the laser beam is allowed to illuminate the material by incident photons through beam on to the material; this may be in ultra violet, visible or near infrared range. Then the scattered photons from the material are allowed to pass through the lens and they are focused onto the wavelength selector such that the Rayleigh scattering photons are filtered out and the Raman scattering photons are allowed through. Then these Raman scattering photons are detected by the charge coupled devices.

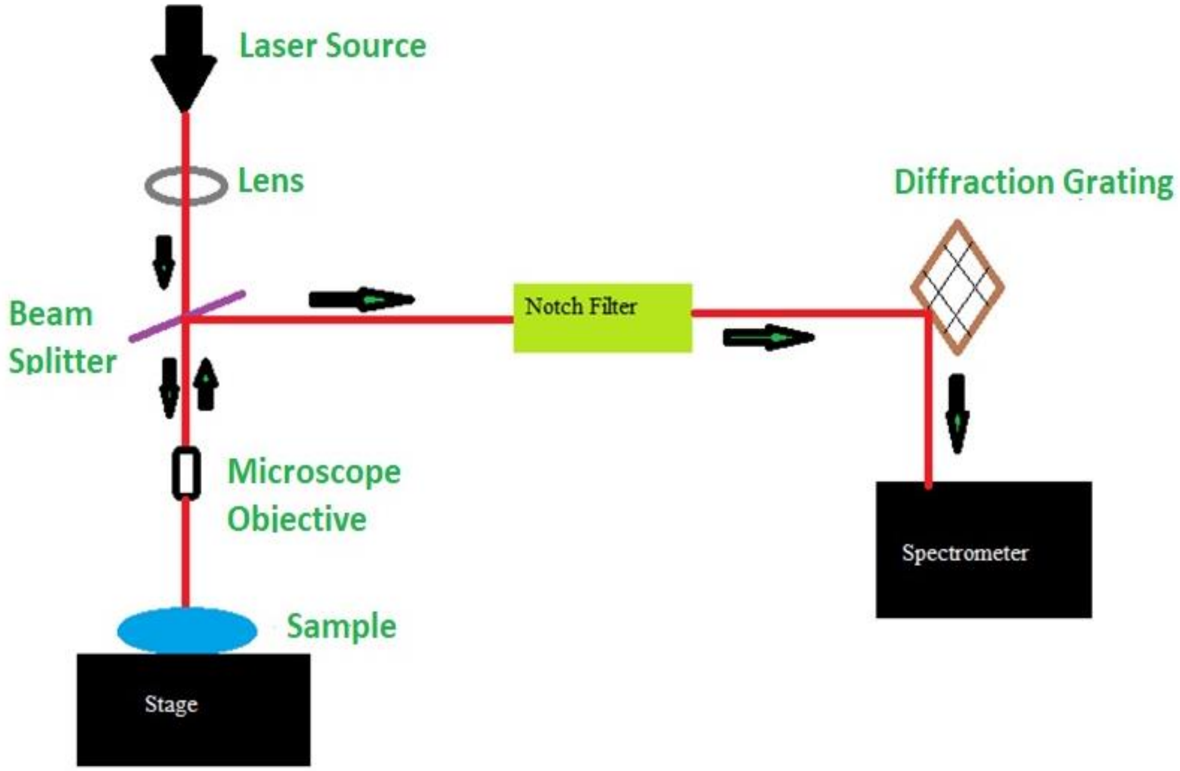

Figure 3.4. Raman spectrometer schematic diagram

This Raman spectroscopy technique helps to examine the wave number which is low, it helps to find the unique spectra of the individual material, it doesn't destroy the sample, using the spectrometer can also analyze polymers and glass. For these reasons, Raman spectroscopy has been used by many researchers to obtain the fingerprint of different molecules such as carbon nanotubes (CNTs), 2-D materials (TMDCs), and Rhodamine 6G (R6G) and test their application as Surface Enhanced Raman Spectroscopy(SERS) substrates [39-42]. There are a few disadvantages while using the technique: the sample gets heated because of the use of laser which has high energy, and this process cannot scan alloys.

CHAPTER 4. VOLTAGE CONTROLLED DEPOSITION OF DISPERSED TRANSITION METAL DICHALCOGENIDES (TMDCs)

In recent times, researchers have been attracted to the field of transition metal dichalcogenides due to the properties TMDCs possess. In our research work we have considered one of the TMDC materials, $MoSe_2$. $MoSe_2$ acts as both direct bandgap in single layer and indirect bandgap in multiple layer. In this research, we are initially going to disperse the TMDCs with the help of solvent, then continue the process by depositing dispersed TMDCs on to various substrates by voltage-controlled deposition technique, and then characterize the deposited substrates using SEM and Raman spectroscopy. The characterization is done to find the effectiveness of the deposition technique used to deposit the dispersed TMDCs on to the substrates.

The major work in this experiment is related to dispersing the TMDC and depositing it onto various substrates using voltage-controlled deposition technique. Initially the dispersion process takes place and is followed by the deposition technique. These processes are explained further in the below text.

4.1. Dispersion of $MoSe_2$ nanoparticles

A quantity of 350mgs of powdered $MoSe_2$ was mixed in 250ml of N methyl Pyrrolidone. The resultant solution was stirred with help of tip-sonicator (model-VCX500, Sonics Materials Inc.) for 30 minutes (24 seconds on, 6 seconds off) in a bed of ice to minimize the temperature rise due to sonication. After tip sonication the resultant $MoSe_2$-NMP solution produced a grey suspension. The solution was kept in the hood untouched for 48 hours to create a stable dispersion as shown in **Figure 4.1.** below.

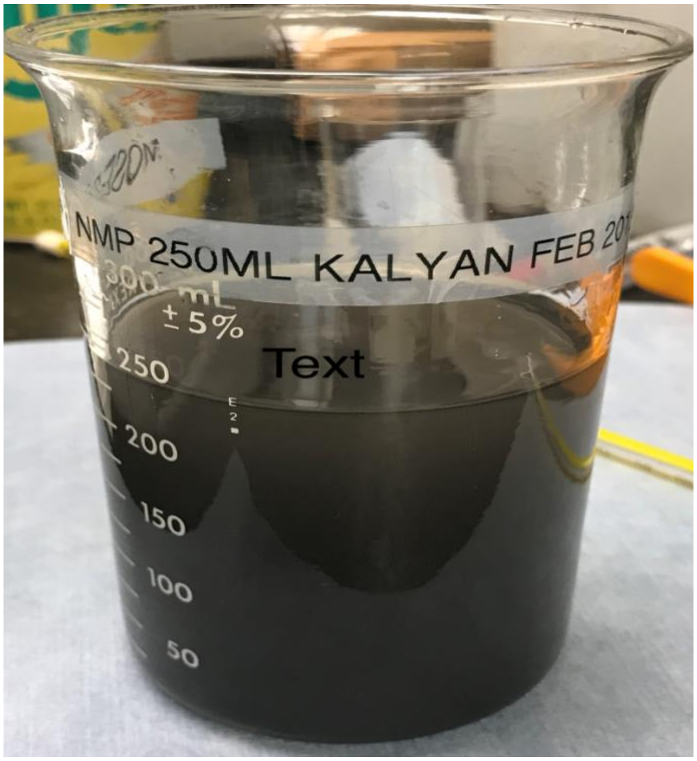

Figure 4.1. Dispersed solution of $MoSe_2$ in NMP

4.2. Deposition of Dispersed $MoSe_2$ Onto Various Substrates Using Voltage-controlled Deposition Technique

The schematic diagram of voltage-controlled deposition of $MoSe_2$ is shown in the **Figure 4.2.**. A 30ml capacity syringe was filled with dispersed $MoSe_2$ in NMP solution and it was mounted on to the Harvard Apparatus 22 dual syringe pump, which faces the substrate setup. The substrate was cleaned before deposition by rinsing it in acetone, isopropyl alcohol (IPA), and De-Ionised water, and was finally dried with the help of nitrogen gas. The target substrate was placed on to the rotating holder which is grounded, and was spun at a constant velocity (7000 rpm) to ensure uniformity in deposition throughout the substrate. A uniform electric field is obtained for spraying by blunting the needle of the syringe. A DC voltage supply (varying from 0-30 kV) was connected to the tip of the blunted needle, a voltage supply of 15 kV was applied to the tip to obtain a uniform electric field. A distance of 5cm is maintained between the tip of the needle and substrate, so that the solution gets sprayed on the whole substrate. The flow rate of $MoSe_2$ suspension through the syringe is kept at a constant 1.5 μL / min throughout the deposition process. Different substrates were deposited (Al, Si, glass, SiO_2, and PMMA) using the same experiment procedure. The deposited substrates were dried under the chemical hood for several hours. The same setup has been previously used by our group to deposit CNTs and TMDCs on different types of substrates [43-48].

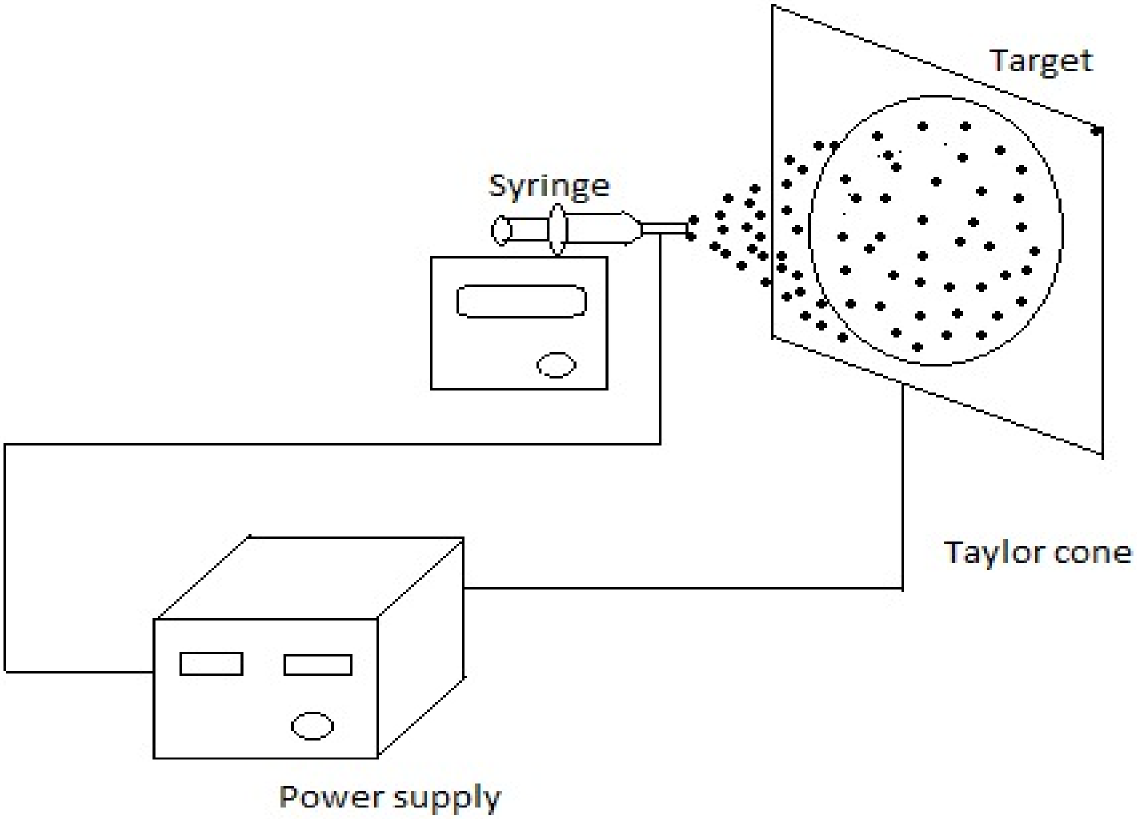

Figure 4.2. Schematic diagram of voltage-controlled deposition technique

4.3. Characterization of $MoSe_2$ Deposited Substrates

By using this voltage-controlled deposition technique, the next step is to confirm that the dispersed solution of $MoSe_2$ was deposited onto the substrates. The substrates that are used in the experiment are conducting substrate (aluminum foil), semi-conducting substrate (bare silicon), and three insulating substrates are considered (silicon dioxide, glass sample, poly methyl methacrylate).

When these deposited substrates are characterized by scanning electron microscopy (SEM) and Raman spectroscopy, it is found that this technique succeeded in depositing the dispersed $MoSe_2$ due to the packing density of the material. This is explained in the characterization techniques which are described below.

4.3.1. Characterization using Scanning Electron Microscopy (SEM)

To obtain the images of the $MoSe_2$ deposited substrates, we used SEM topographical and morphological images. The $MoSe_2$ deposited substrates were examined using a JPEL JSM 6610 SEM system, which is operated at 20-30 kV of acceleration voltage for micro-structural surface imaging of the substrates. **Figure 4(3-7)** depicts the deposition of $MoSe_2$ onto different substrates. The deposition for each of the substrates was verified by the homogeneity and density of the $MoSe_2$. Multilayered $MoSe_2$ nanosheets were observed in **Figure 4(3-7)**, where the images are taken on a scale of 500 nm as shown. Plate-like structures are observed in the form of 3-7 layers which confirm deposition of 2D material onto the substrates. On the other hand, these layers of the $MoSe_2$ exhibit a random displacement, thus calling for the future work of improving uniformity in the deposition technique. Nevertheless, as per the goal of the research for this thesis, the images confirm that the deposition was completed successfully using this technique.

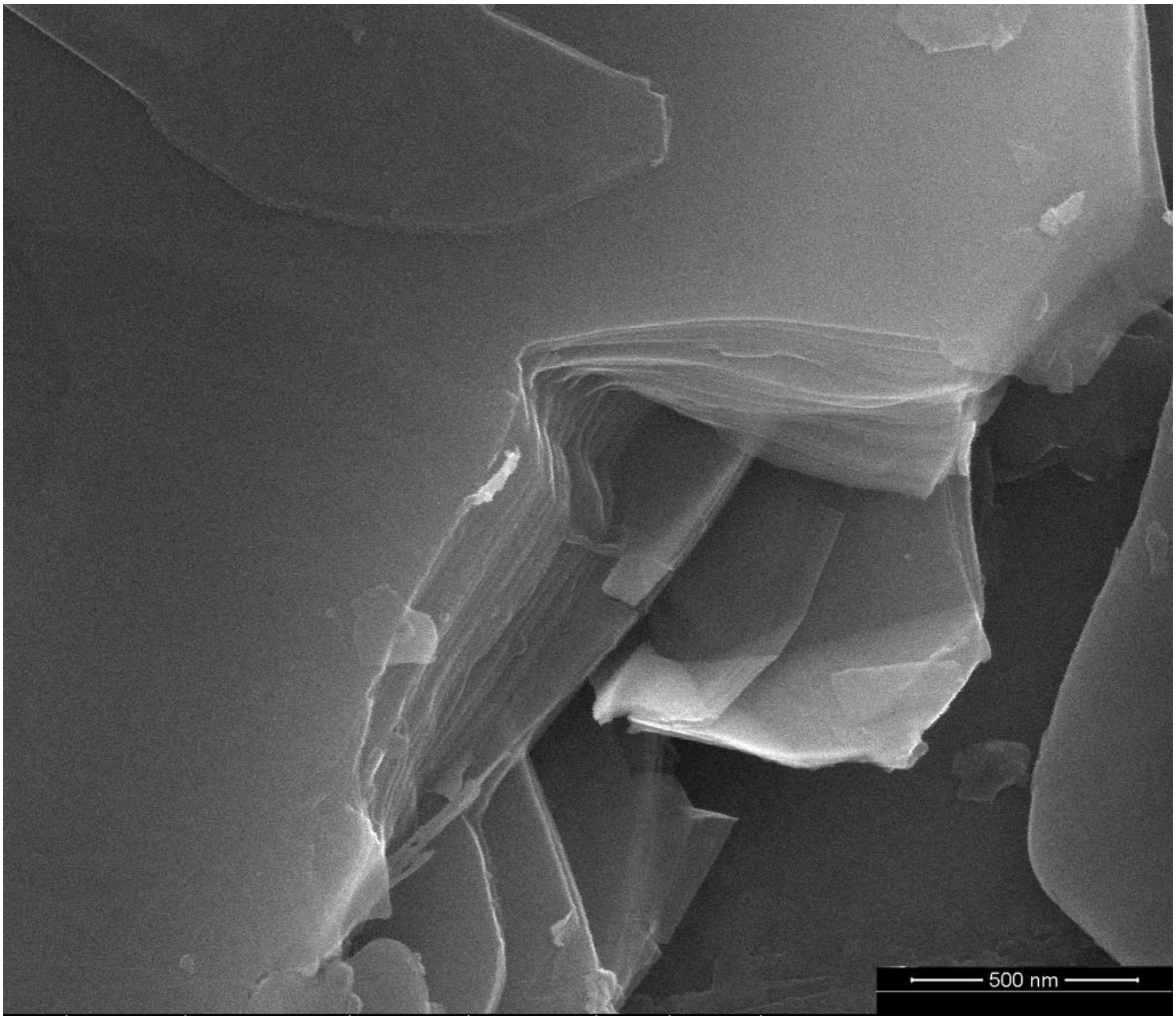

Figure 4.3. SEM image of $MoSe_2$ deposited on Aluminum foil

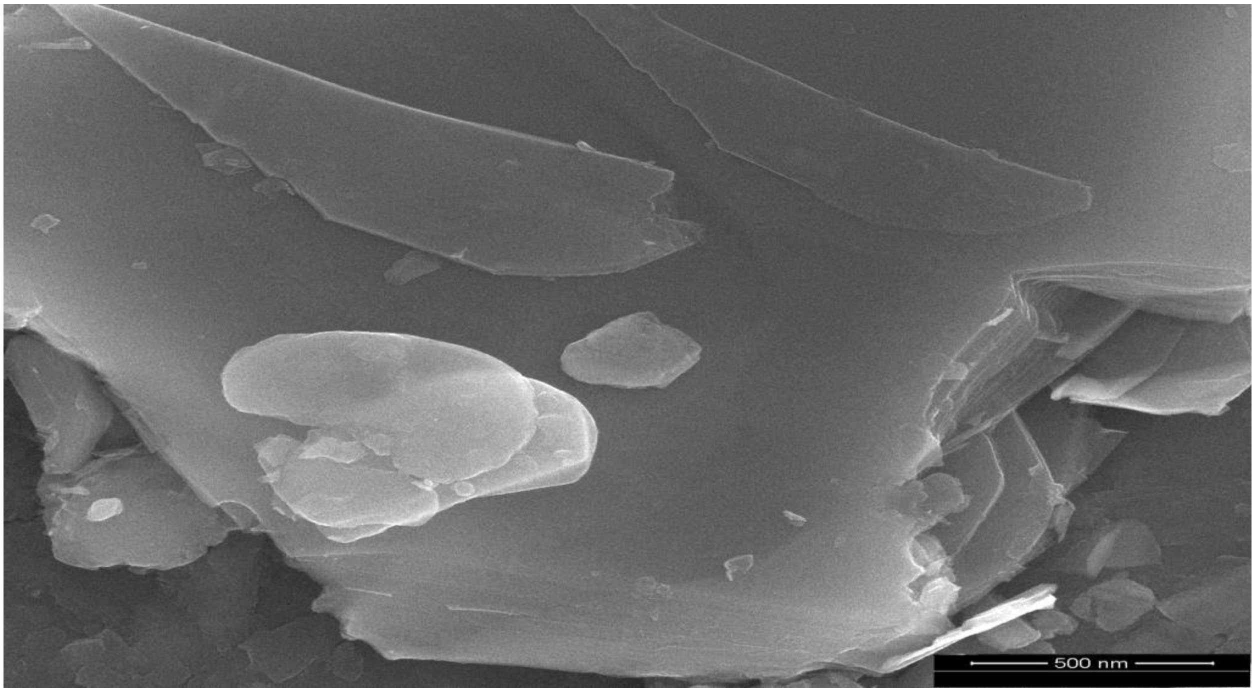

Figure 4.4. SEM image of $MoSe_2$ deposited on Si wafer

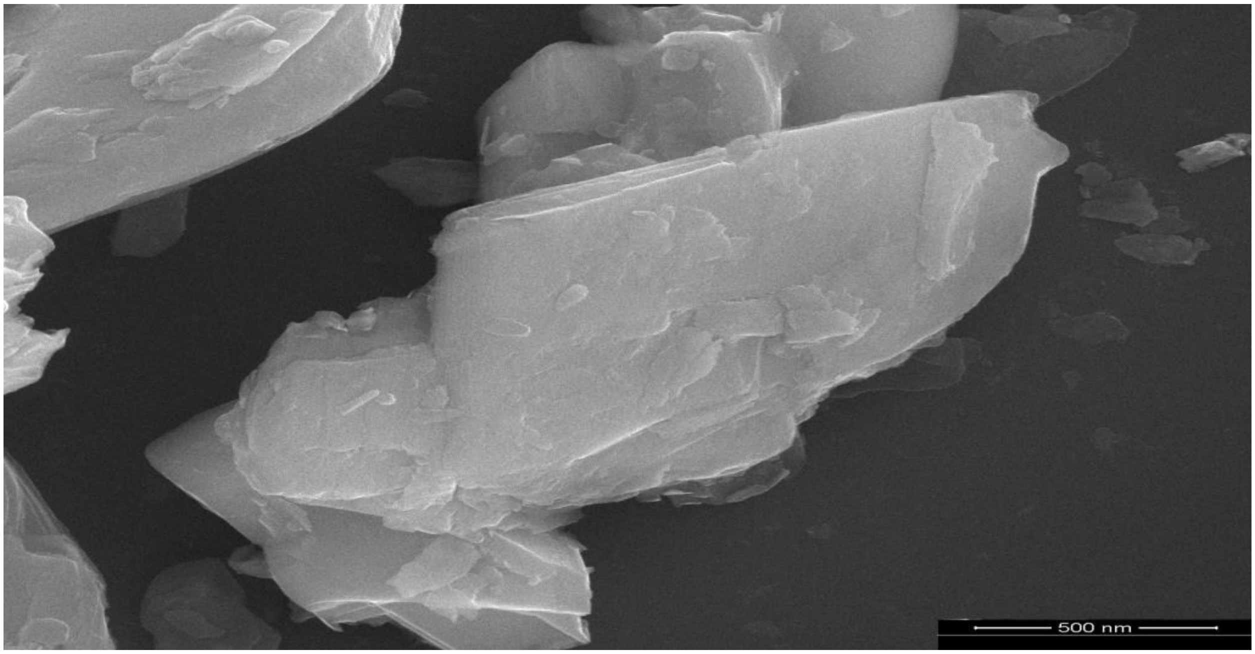

Figure 4.5. SEM image of $MoSe_2$ deposited on PMMA

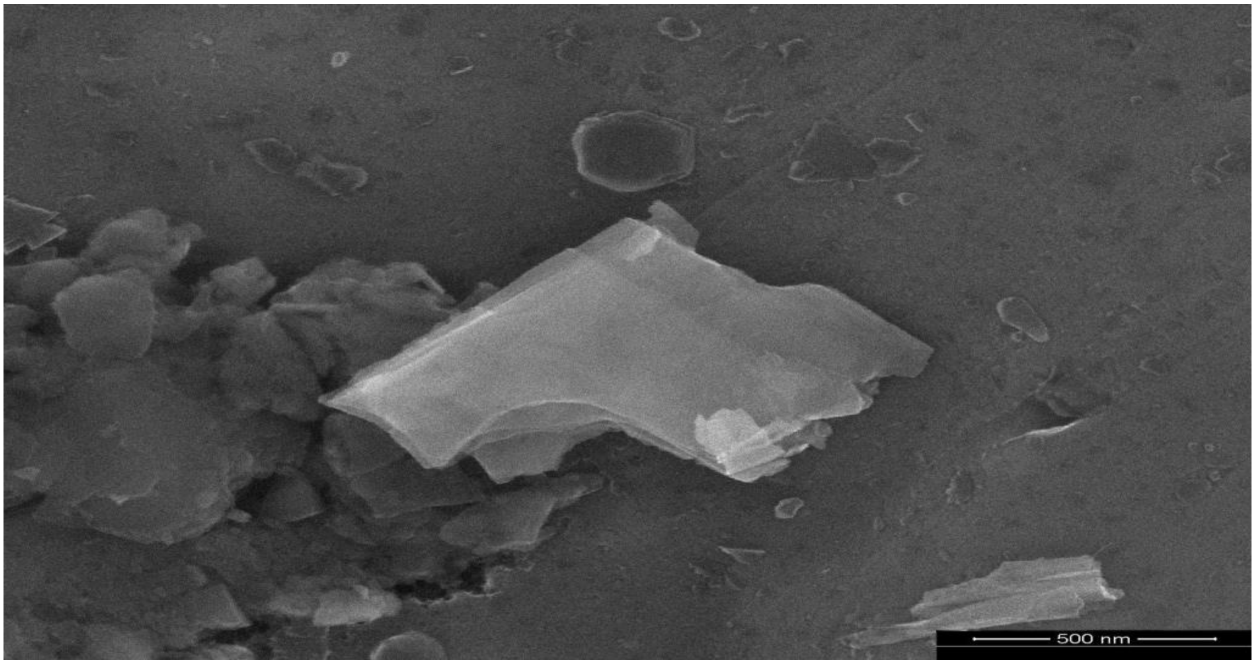

Figure 4.6. SEM image of $MoSe_2$ deposited on SiO_2

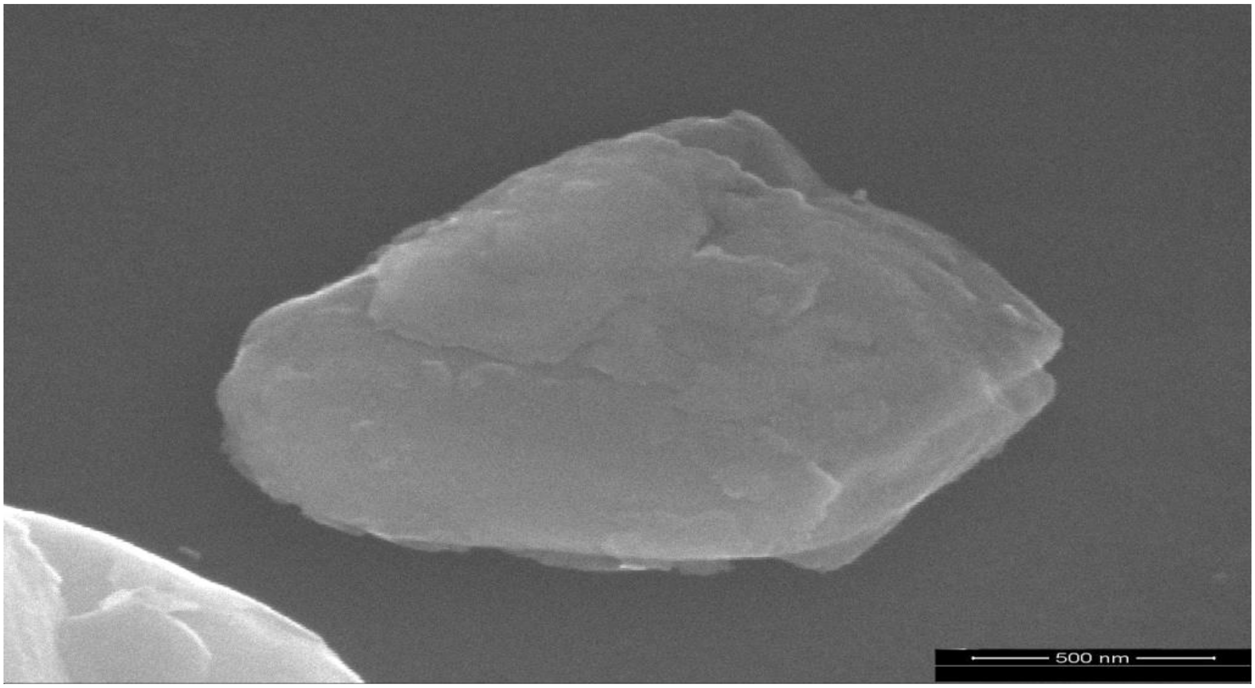

Figure 4.7. SEM image of $MoSe_2$ deposited on Glass

4.3.2. Characterization using Raman Spectroscopy

Raman spectroscopy is another technique we used to characterize the materials which were deposited onto the substrates. Raman spectroscopy provides unique spectra for individual material, so we used this technique to further verify that $MoSe_2$ was deposited onto the substrates. As per the previous experiments conducted by different researchers on $MoSe_2$, the Raman spectra for $MoSe_2$ is obtained between 100 – 400 cm^{-1} [49-51].

A Jobin Yvon Horiba Labram Raman spectrometer was used to characterize the deposited $MoSe_2$ on the substrates, and the spectrometer was equipped with a 17 mW, 632.81 nm wavelength HeNe laser. The aperture of the confocal hole was selected at 200 µm, and the grating of 1800 lines/mm was selected for the scanning. The peaks were measured at the range of 100 to 400 cm^{-1} wavenumber. **Figure. 4(8-12)** shows the Raman spectroscopy of $MoSe_2$ on different substrates. From the results below it is clearly depicted that the peaks are observed between 240-342 cm^{-1}. From these peaks we can confirm the presence of $MoSe_2$ on all the substrates.

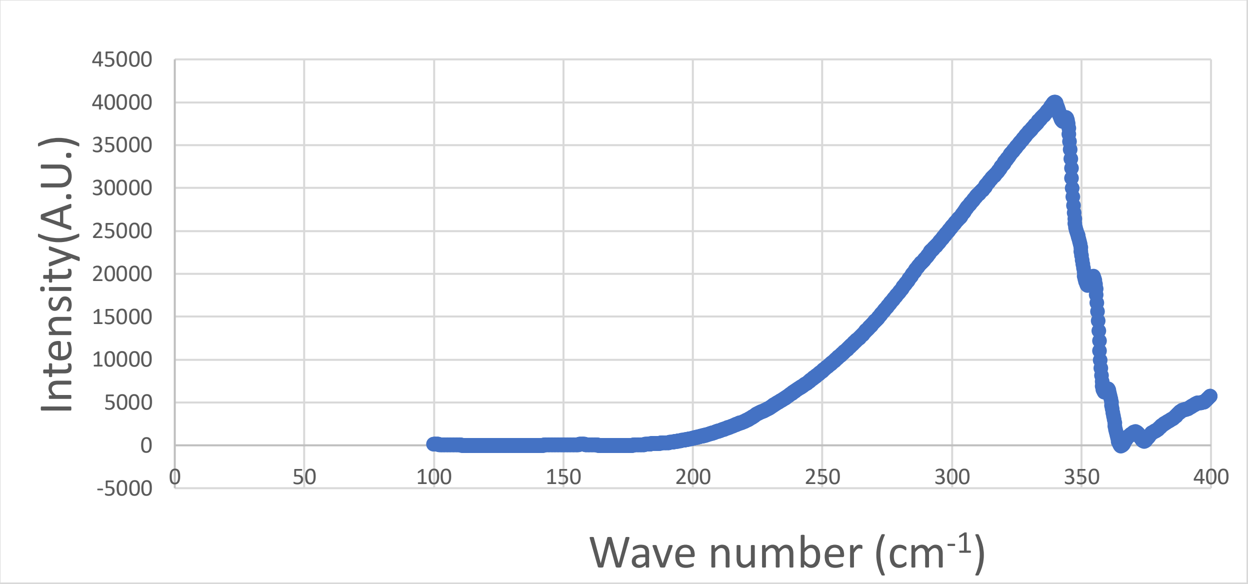

Figure 4.8. Raman spectra of $MoSe_2$ deposited on Al foil

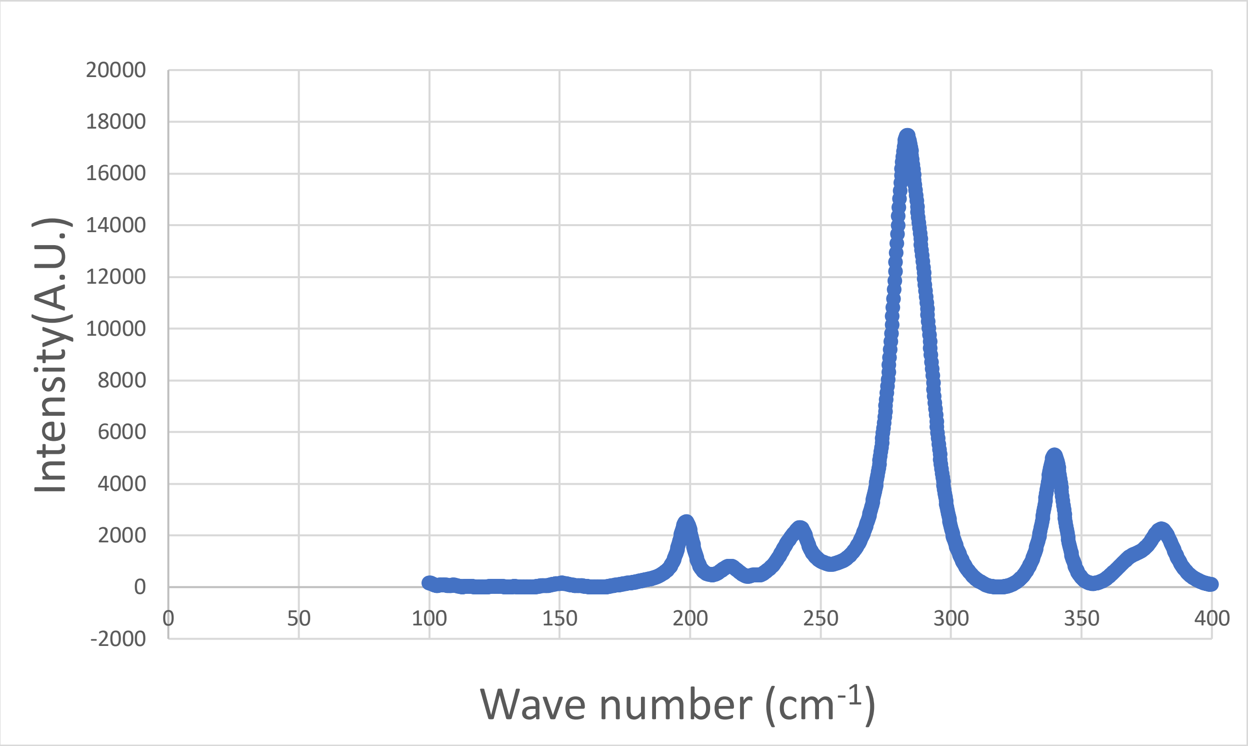

Figure 4.9. Raman spectra of $MoSe_2$ deposited on Si wafer

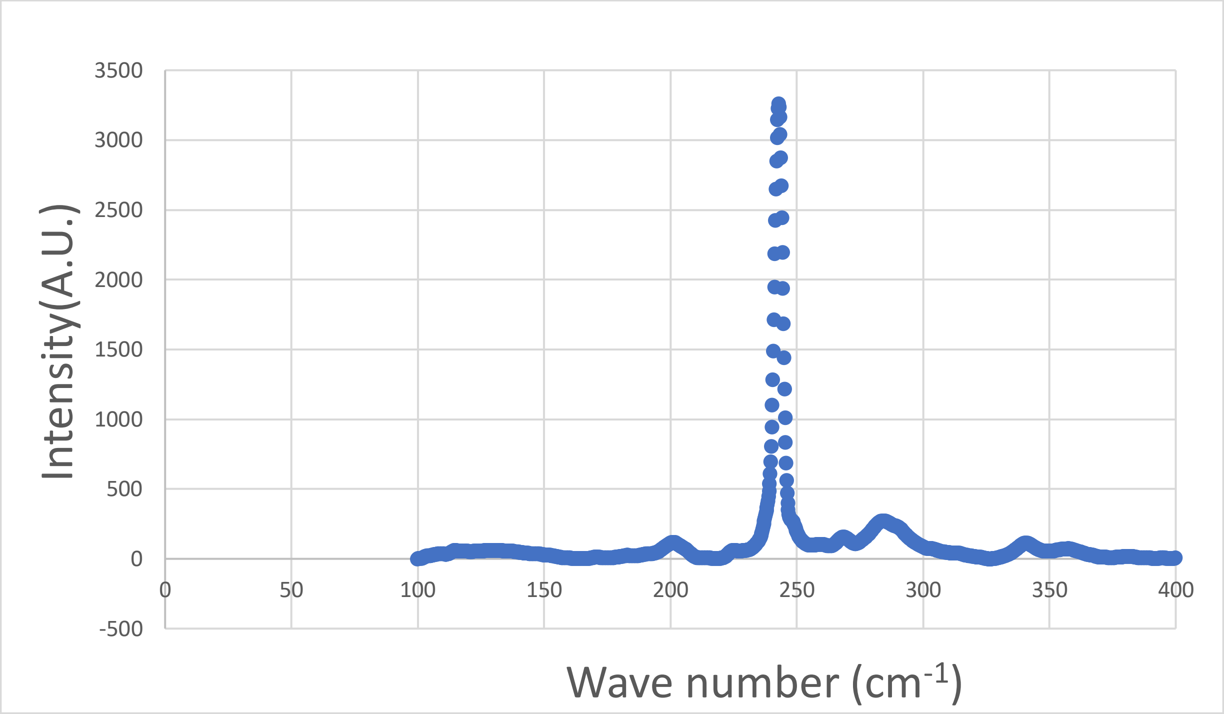

Figure 4.10. Raman spectra of $MoSe_2$ deposited on PMMA

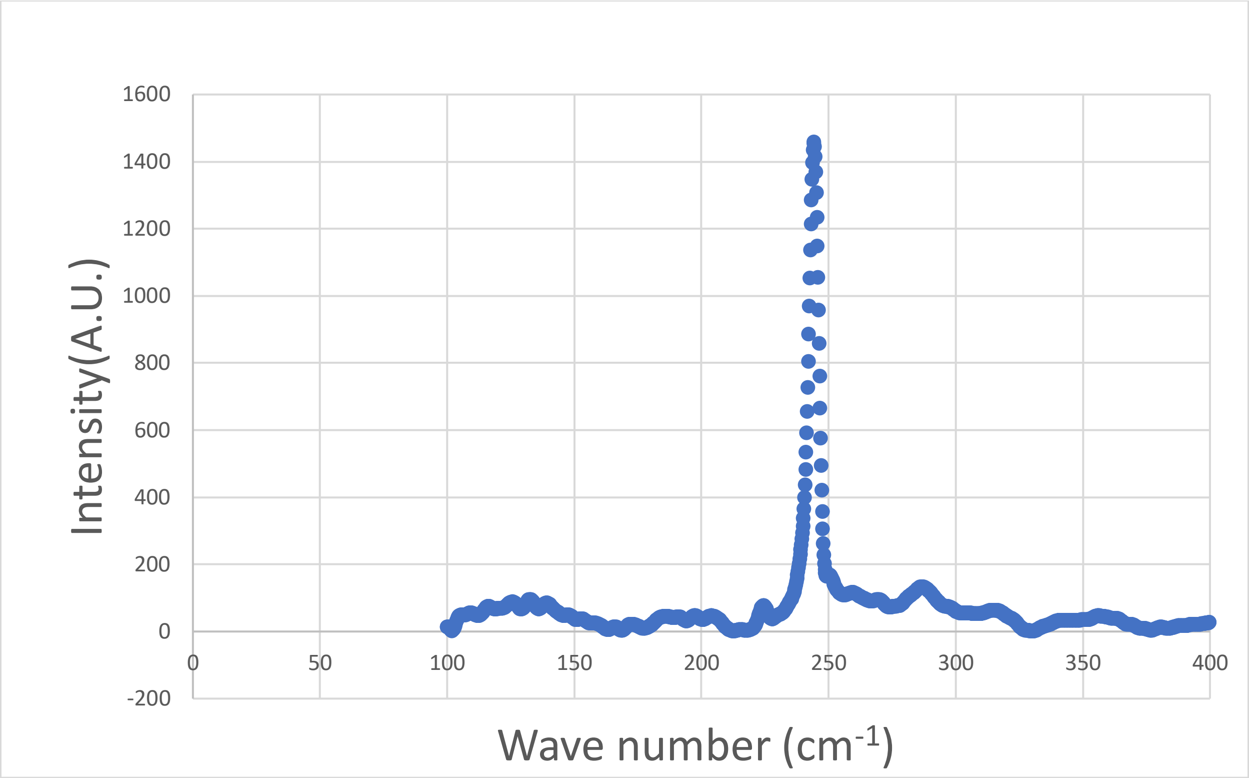

Figure 4.11. Raman spectra of MoSe$_2$ deposited on SiO$_2$

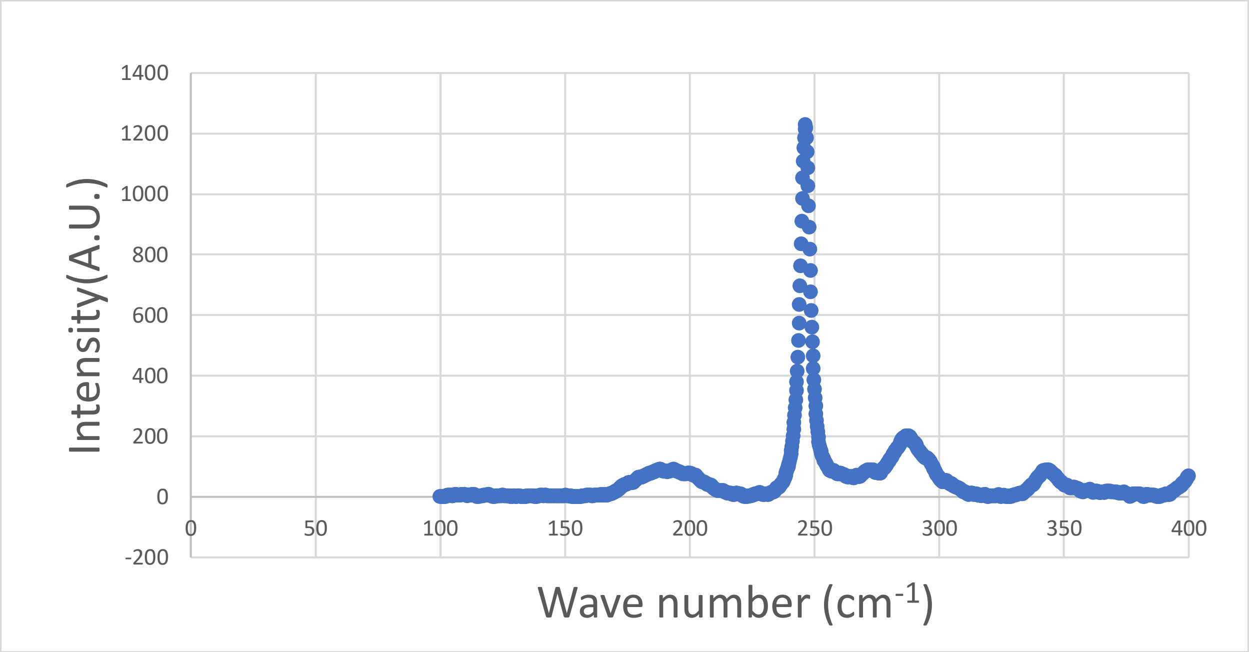

Figure 4.12. Raman spectra of MoSe$_2$ deposited on glass

4.4. Etching of Substrates:

Etching is a process where the top most layers of the substrate are removed. In this research, thus far, we have performed experiments on the substrates without pretreatment. On further we would like to etch the substrates and conduct the same experiments to compare the etched and non-etched results. The substrates used in this research are Al foil as conducting substrate, Si wafer as semi-conducting substrate, Glass, PMMA and SiO_2 as insulating substrates. The etching agents used here are 30% KOH (Potassium Hydroxide) for etching Al foil, to etch Si wafer, SiO_2, glass buffered oxide etchant is used. To etch PMMA, sand paper is used.

The Al foil is placed in the 30% of KOH solution for 30sec and then it is cleaned using DI water and dried using Nitrogen gas. The Si wafer, SiO_2, and Glass are placed in the buffered oxide etchant for 3 mins and then they are cleaned using DI water and dried using Nitrogen gas. The PMMA is etched using sand paper; it is cleaned using DI water and dried using nitrogen gas.

4.4.1. Characterization of $MoSe_2$ Deposited Substrates

Once the etched substrates are deposited, they are characterized by the scanning electron microscopy (SEM) and Raman spectroscopy, it is found that this technique succeeded in depositing the dispersed $MoSe_2$ because it is deposited on the surface with a great packing density. This is explained in the characterization techniques which are described below.

4.4.2. Characterization using Scanning Electron Microscopy (SEM)

To obtain the images of the $MoSe_2$ deposited etched substrates, we used SEM topographical and morphological images. The $MoSe_2$ deposited substrates were examined using a JPEL JSM 6610 SEM system, which is operated at 20-30 kV of acceleration voltage to get micro-structural surface imaging of the substrates. **Figure 4(13-17)** depicts the deposition of $MoSe_2$ onto different substrates. The deposition for each of the substrates was verified by the homogeneity and density of the $MoSe_2$. Multilayered $MoSe_2$ nanosheets were observed in **Figure 4(13-17)**, where the images are taken on a scale of 500 nm as shown. The $MoSe_2$ nanosheets can been seen in the plate-like structure, it also has 3-7 layers. On the other hand, these layers of the $MoSe_2$ exhibit a random displacement. The images confirm that the deposition was completed successfully using this technique as per the goal the research.

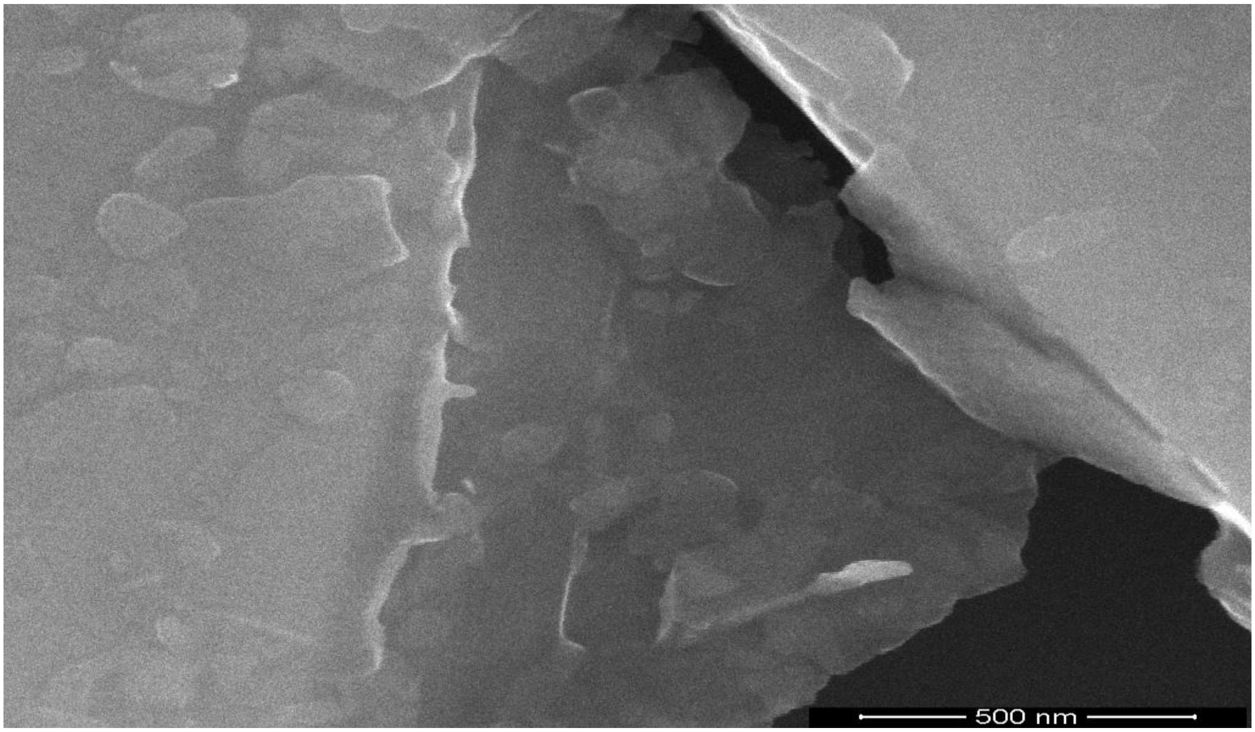

Figure 4.13. SEM image of $MoSe_2$ deposited on Etched Aluminum foil

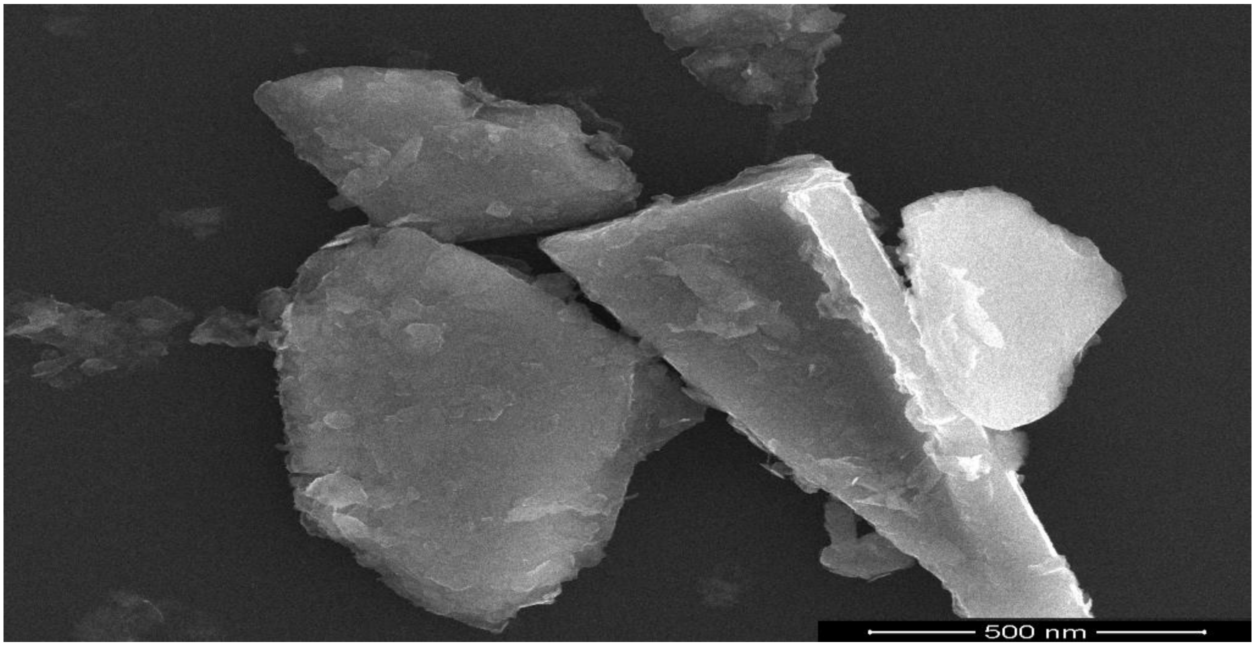

Figure 4.14. SEM image of $MoSe_2$ deposited on Etched Si wafer

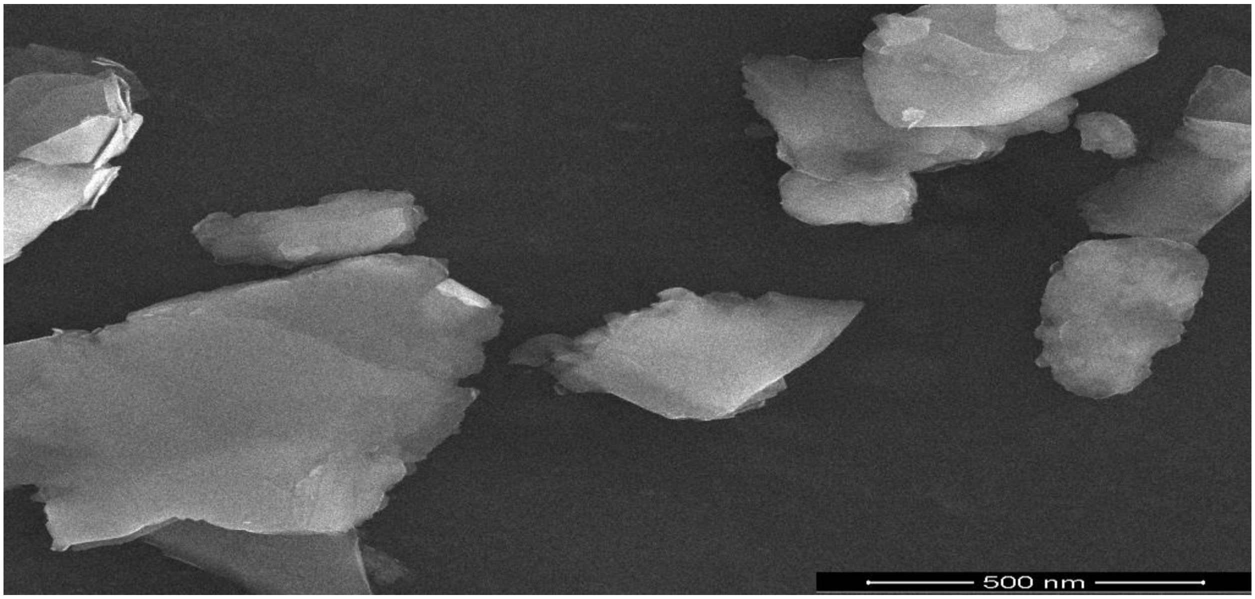

Figure 4.15. SEM image of $MoSe_2$ deposited on Etched PMMA

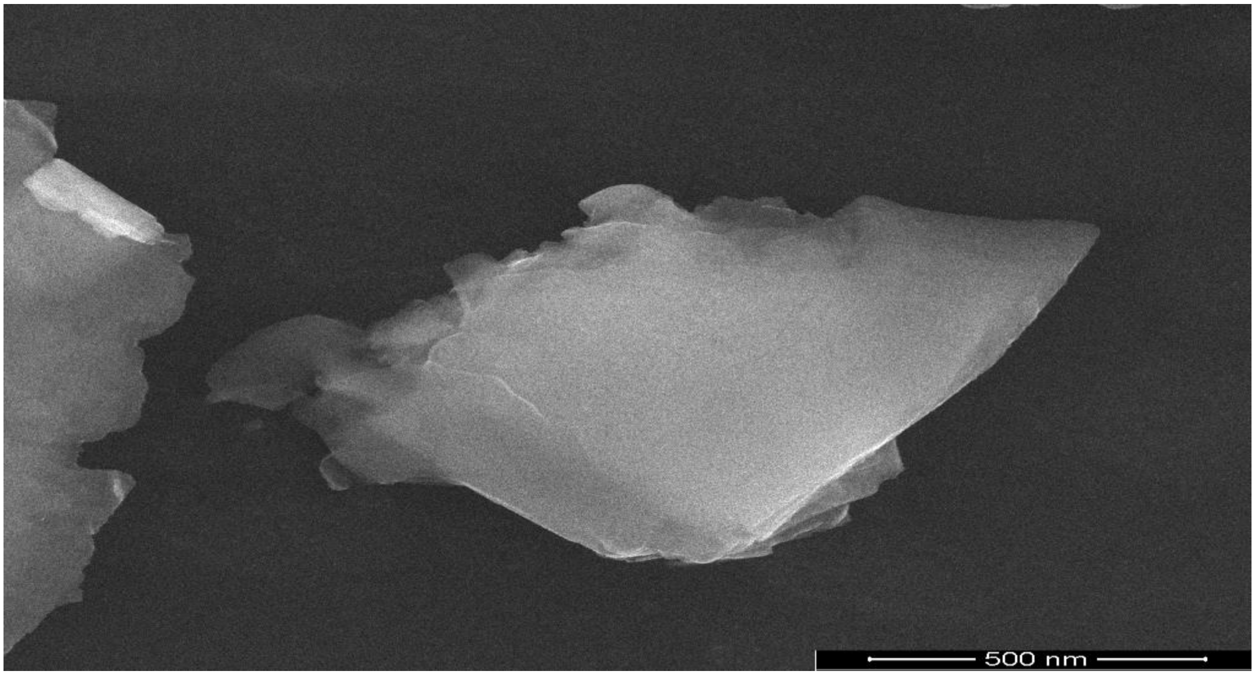

Figure 4.16. SEM image of $MoSe_2$ deposited on Etched SiO_2

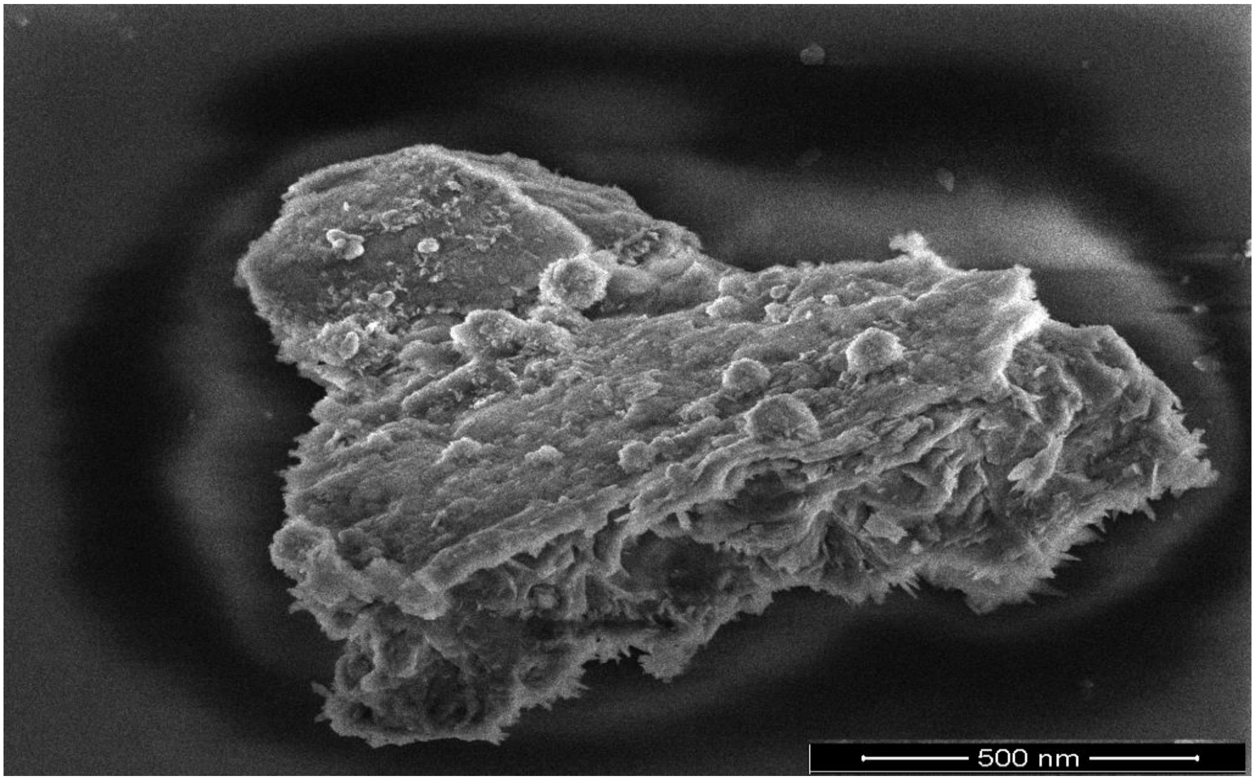

Figure 4.17. SEM image of $MoSe_2$ deposited on Etched Glass

4.4.2. Characterization using Raman Spectroscopy

A Renishaw inVia Reflex Raman Microscope was used to characterize the deposited $MoSe_2$ on the etched substrates, and the spectrometer was equipped with 633 nm wavelength of Ne laser. The aperture of the confocal hole was selected at 50 L. As per the previous experiments conducted by different researchers on $MoSe_2$, the Raman spectra for $MoSe_2$ is obtained at 100 – 400 cm^{-1} [65-67]. The peaks were measured in the range of 100 to 400 cm^{-1} wavenumber. **Figure. 4(18-22)** shows the Raman spectroscopy of $MoSe_2$ on different etched substrates. The results below clearly depict that the peaks are observed between 146-305 cm^{-1}. From these peaks we can confirm the presence of $MoSe_2$ on all the substrates.

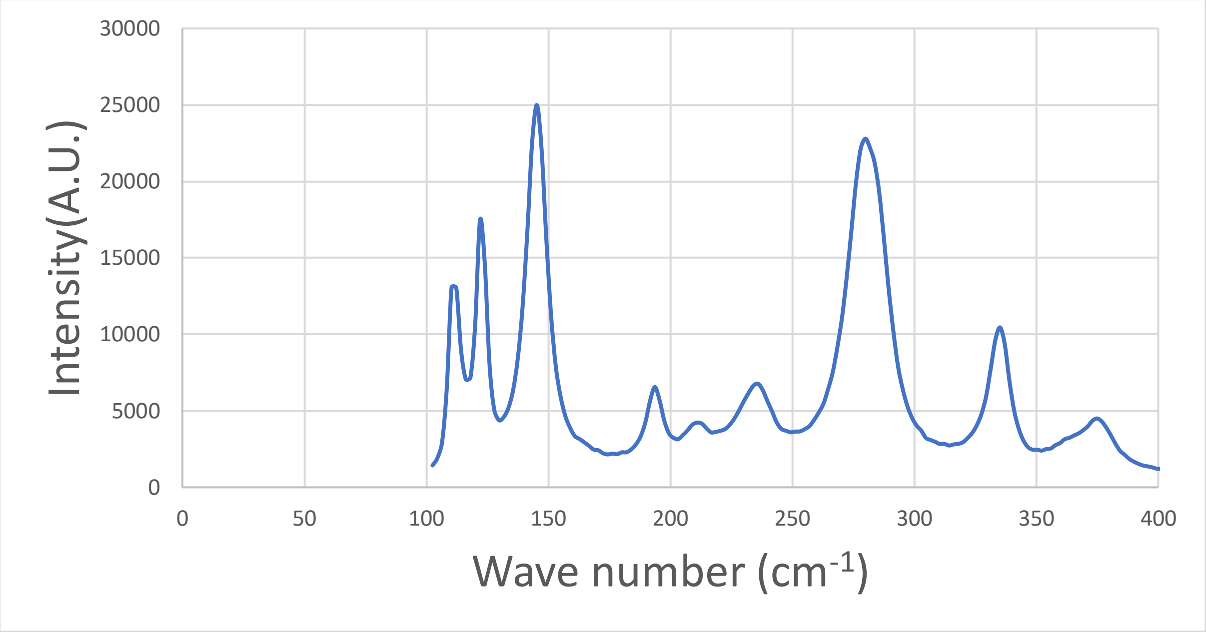

Figure 4.18. Raman spectra of $MoSe_2$ deposited on Etched Al foil

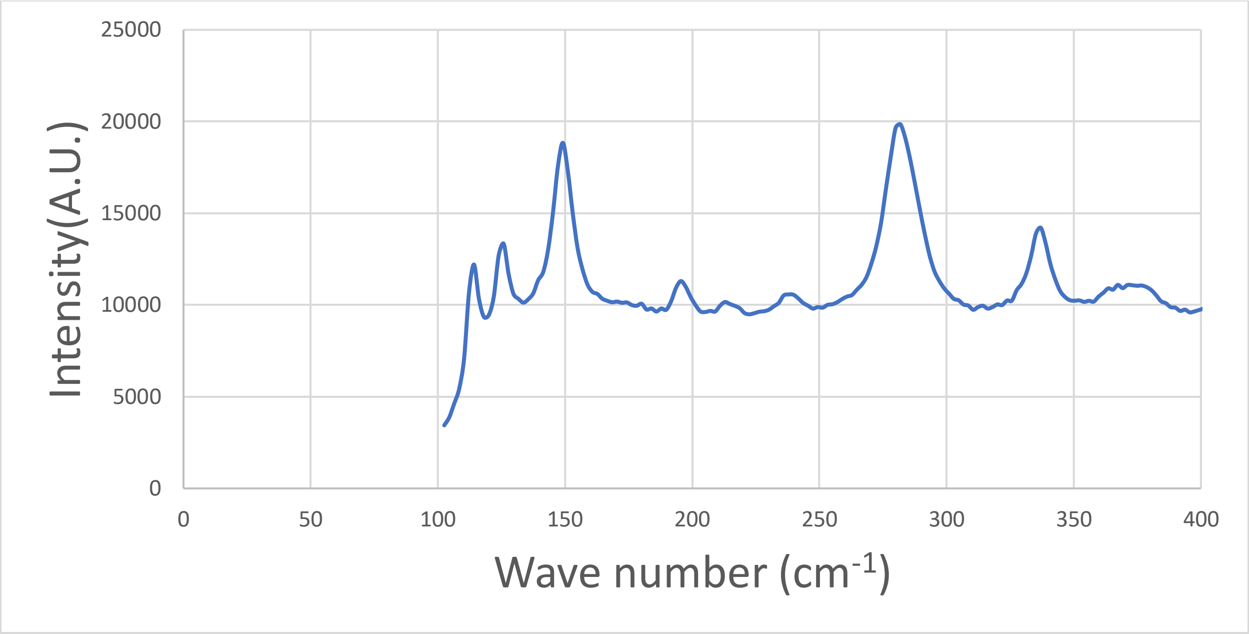

Figure 4.19. Raman spectra of $MoSe_2$ deposited on Si wafer

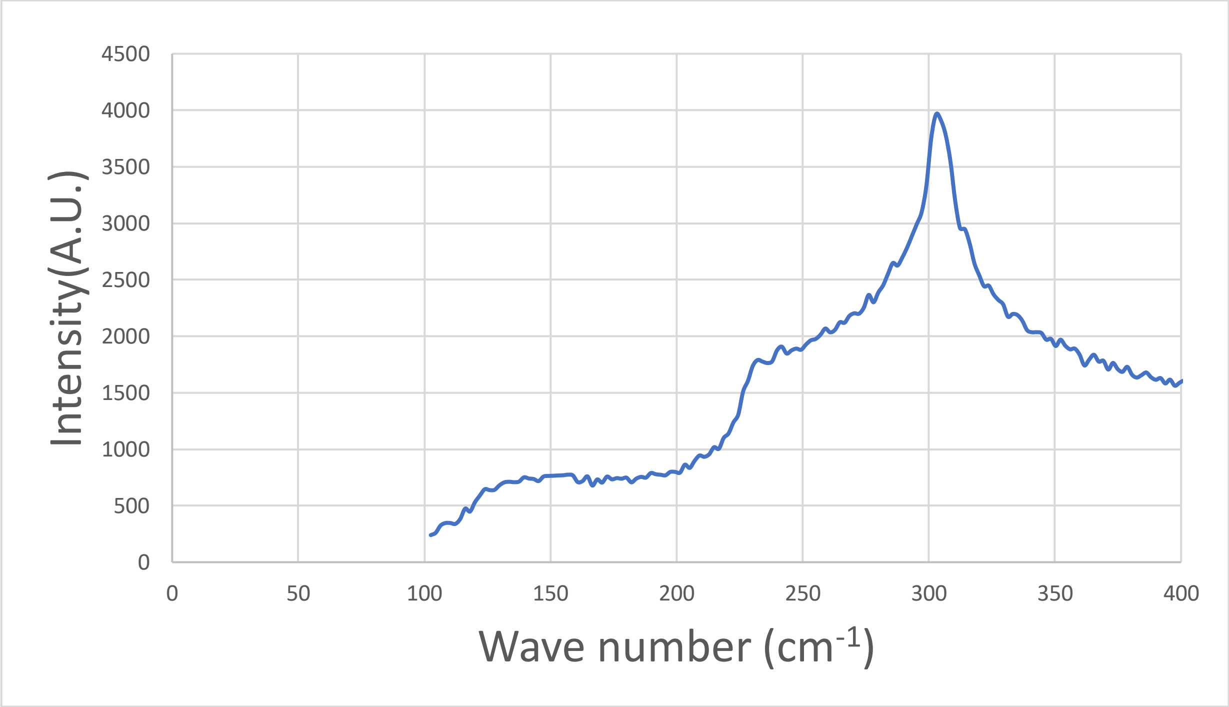

Figure 4.20. Raman spectra of $MoSe_2$ deposited on Etched PMMA

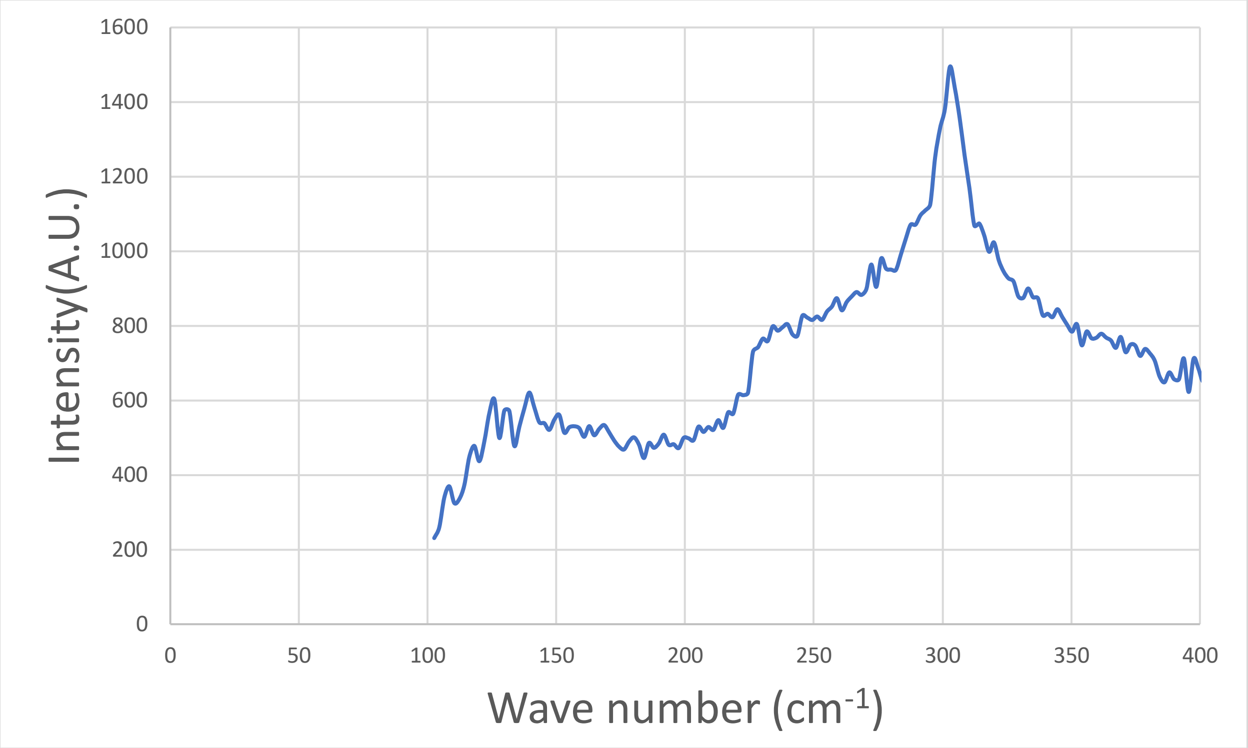

Figure 4.21. Raman spectra of $MoSe_2$ deposited on Etched SiO_2

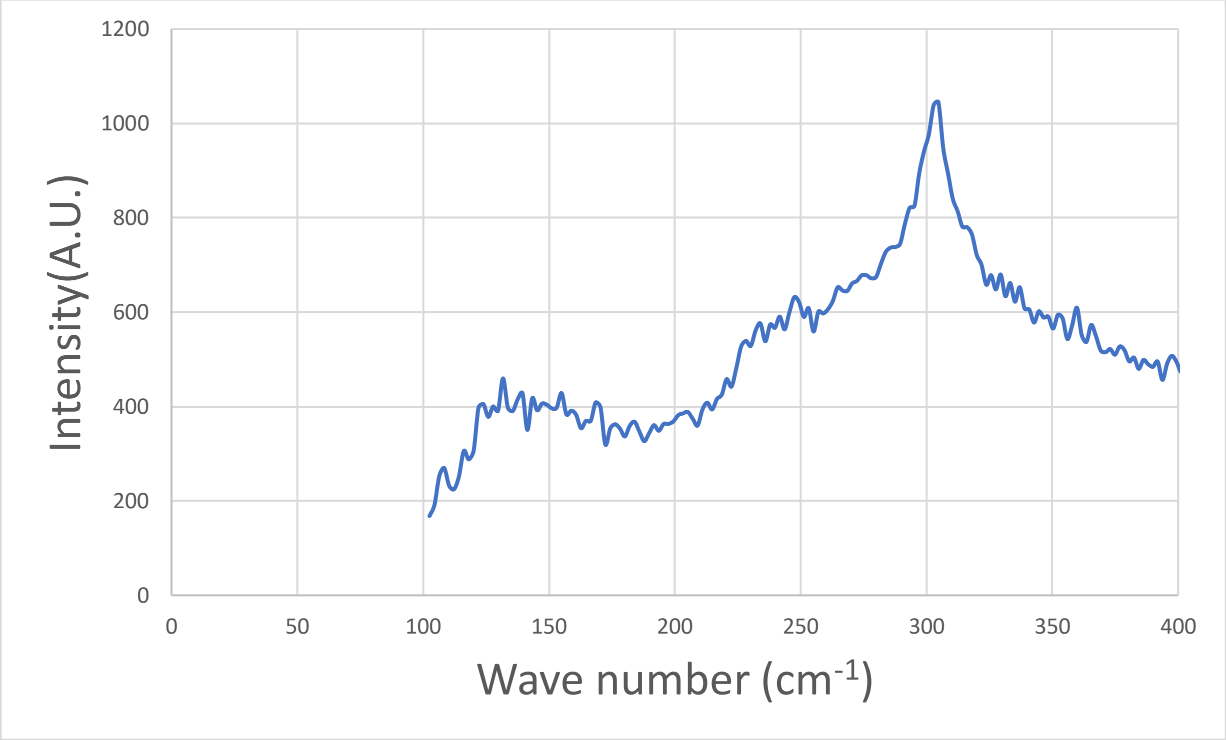

Figure 4.22. Raman spectra of $MoSe_2$ deposited on Etched Glass

4.5 Thickness measurement of $MoSe_2$ films deposited on etched and non-etched substrates

Alpha Step Surface Profiler is an instrument which is used to find the thickness of the material deposited onto the substrates. The thickness of the $MoSe_2$ on the substrates varies with deposition time. **Table 4.1.** below give details of the thickness of the deposited $MoSe_2$.

Table 4.1. Thickness of $MoSe_2$ on various substrates

SUBSTRATE	Thickness of $MoSe_2$ Film		
	Thickness of Avg. deposition time of 5 min.	Thickness of Avg. deposition time of 10 min.	Thickness of Avg. deposition time of 20 min.
Al Foil	650 nm	1.9 μm	3.3 μm
Si Wafer	580 nm	1.7 μm	3.1 μm
PMMA	565 nm	1.6 μm	2.8 μm
SiO_2	494 nm	1.4 μm	2.5 μm
Glass	420 nm	1.1 μm	2.1 μm

Table 4.2. Thickness of $MoSe_2$ on various etched substrates

SUBSTRATE	Thickness of $MoSe_2$ Film		
	Thickness of Avg. deposition time of 5 min.	Thickness of Avg. deposition time of 10 min.	Thickness of Avg. deposition time of 20 min.
Al Foil	590 nm	1.82 μm	3.1 μm
Si Wafer	560 nm	1.7 μm	2.9 μm
PMMA	525 nm	1.5 μm	3.0 μm
SiO_2	460 nm	1.2 μm	2.6 μm
Glass	400 nm	0.95 μm	1.9 μm

The thickness of the $MoSe_2$ on the etched substrates varies depending on the deposition time. The **Table 4.2.** above give the details of the thickness of the deposited $MoSe_2$.

4.6 Conclusions

In this work, we have discussed the results that we obtained from the dispersion, deposition and characterization of $MoSe_2$ with respect to various species of substrates. It is clearly depicted in the results, that the dispersed $MoSe_2$ solution is well deposited on to the various substrates and also onto the etched substrates. The voltage-controlled deposition technique is proven to successfully deposit on to the substrates without any pretreatment and also onto the etched surface of substrates. The characterization proves that the substrates are coated with the $MoSe_2$. From the results we can conclude that $MoSe_2$ can be deposited effectively and efficiently using voltage-controlled deposition technique with low cost and high yield of deposition.

This deposition technique is economical, low-cost, fast, facile and consistent. These results are promising toward the use of the steps and methods explored in this thesis for other TMDCs and nanoparticle material.

Chapter 5. SUMMARY AND RECOMMENDATIONS FOR FUTURE WORK

5.1. Summary

In this research, we have developed a prominent technique that is voltage-controlled to deposit dispersed solutions onto substrates without pretreatment. This work has been successful in depositing dispersed $MoSe_2$ on to various substrates which are etched and non-etched. The substrates used in this research are conducting substrate (aluminum foil), semi-conducting substrate (bare silicon), three insulating substrates (silicon dioxide, glass sample, poly methyl methacrylate). This technique eliminated the need for extensive labor, long processing times, exorbitantly high costs, and substrate pretreatment which are necessary in some other methods. Verification techniques such as SEM and Raman spectroscopy are used to determine the deposition of $MoSe_2$ on to various substrates.

5.2. Future work

In the future work, we can focus to use $MoSe_2$ as channel, possibly for transistors. This voltage-controlled deposition technique can be used to deposit other 2D materials onto various other substrates. We can also use Atomic Force Microscopy and other equipment for further characterization.

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VITA

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