

# **Electrical and Optical Characterization of Thin Film**

**A thesis submitted to the Electronics and Communication Department of East West University-  
Bangladesh in partial fulfillment of the requirements for the degree of Masters of Science in  
Applied Physics and Electronics.**

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## Declaration

This is to certify that this thesis is my original work. No part of this work has been submitted elsewhere partially or fully for the award of any other degree or diploma. Any material reproduced in this thesis has been properly acknowledged.

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## Approval

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## **Table of Contents**

Page No

<b>Chapter 1 Introduction.....</b>	<b>(1-2)</b>
1.1 Introduction	1
1.2 Characterization of Semiconductor Materials and Device	2
1.3 Objective of the Project	2
<b>Chapter 2 Deposition of Thin Flim.....</b>	<b>(3-5)</b>
2.1 Definition of Thin Film Deposition	3
2.2 Classification of Thin Film	3
2.2.1 Chemical Deposition	3
2.2.2 Physical Deposition	4
2.3 Basics thermal Evaporation	5
<b>Chapter 3 Vacuum Technology.....</b>	<b>(6-7)</b>
3.1 Brief History of Vacuum Technology	6
3.2 Types of Vacuum Techniques	7
3.3 Applications of Vacuum Technology	7
<b>Chapter 4 Substrate Cleaning and Experimental Deposition of Materials.....</b>	<b>(11-17)</b>
4.1 Experimental Procedure	
4.1.2 Substrate Cleaning	
4.2 RCA Cleaning	11
4.1.1. Preparation	11
4.2.2 Procedure	12
4.2.3 HF Etching	12
4.2.4 Preparation	12
4.2.5 Procedure	12
4.3. Experimental Procedure	13
4.3.1 Principle of Thermal Evaporation	13
4.3.2 Preparation for Deposition	13
4.3.3 Deposition for Aluminum and Zinc Oxide	14
4.4 Equipment Used in Experiment	15
4.4.1 Mechanical Pump	15
4.4.2 Diffusion Pump	16
4.4.3 Baffle (Water-Cooled)	17

<b>4.4.4 Cold Trap</b>	17
<b>4.4.5 Roughing Line and Valve</b>	17
<b>Chapter 5 Measurement of Vacuum.....</b>	<b>19-22)</b>
<b>5.1. Pirani Gauge</b>	19
<b>5.2 Penning Gauge</b>	20
<b>5.3. Leak Detection</b>	20
<b>5.4 Techniques for Leak Detections</b>	22
<b>Chapter 6 Optical and Electrical Characteristic of Thin Film.....</b>	<b>(24-30)</b>
<b>6.1. Measurement of Thickness of Thin Film</b>	24
<b>6.2. Profilometer Analysis of Thin Film Structure</b>	24
<b>6.3. Measurement of Resistance of Thin Film</b>	28
<b>6.4. Findings</b>	30
<b>Chapter 7 Development of Protocols for Dektak-150 Autoprofilometer and RV-126 Auto scraper.....</b>	<b>(31-37)</b>
<b>7.1. Switch On Steps for Dektak-150 Autoprofilometer</b>	31
<b>7.2. Switch Off Steps for Dektak-150 Autoprofilometer</b>	31
<b>7.3. Loading the Sample</b>	31
<b>7.4. Hardware leveling of the stage</b>	31
<b>7.4.1. Mirror Leveling</b>	31
<b>7.4.2. Stage Leveling</b>	32
<b>7.5. Single Line Scan- Standard Scan</b>	32
<b>7.5.1. Single Scan Setup Steps</b>	33
<b>7.5.2. Programming a Single Line Scan Routine</b>	33
<b>7.5.3. Running a Single Line Scans Routine</b>	33
<b>7.5.4. Setting Cursor Locations and Bandwidths</b>	34
<b>7.6. Performing Software Leveling</b>	34
<b>7.7. RV 126 Autoscriber</b>	35
<b>7.7.1. Protocol Development of the Autoscriber</b>	35
<b>7.7.2. Principle of Cutting</b>	35
<b>7.7.3. Starting the Scriber</b>	36
<b>7.7.4. Working with the Autoscriber Scribing a Silicon Wafer</b>	37

**Chapter 8 Studying the Geometry of Samples with Dektak-150 Autoprofilometer (38-41)**

<b>8.1. Observing Non- Uniformity of a Glass Slide</b>	38
<b>8.2. Observing Film Layer on a Silicon Wafer</b>	39
<b>8.3. Detecting the Step at the Al-Silicon Interface</b>	40
<b>8.4. Measuring Width of a Stripe of the Mask</b>	41
<b>8.5. Measuring Depth of a Stripe of a Section of the Mask</b>	41

**Chapter 9 Scanning Electron Microscope.....(43-46)**

<b>9.1. What is a SEM</b>	43
<b>9.2. How Does a SEM Work</b>	43
<b>9.3. How is a Sample Prepared</b>	44
<b>9.4. Strengths and Limitations of Scanning Electron Microscopy (SEM)</b>	45
<b>9.5. Result and Discussion</b>	46

**Chapter 10 Conclusion.....(47-48)**

10.1 Summary	47
10.2 Future Work	48

<b>Reference</b>	49
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## **Abstract**

**In this work, thin film of Al and ZnO has been developed on microscopic glass substrate using ~~SHWARD~~ thermal vacuum evaporator. The thickness of the deposit films was measured using surface profilometer Dektak-150. The deposition of the films was found in between 600 and 650 nm. Over a total scan length 1000 $\mu$ m. The obtained thickness could be assessed precise for the present research and the uniform deposition within the obtained thickness may be attributed to strong adhesion between the materials of film and the microscopic glass substrate.**

**Resistivity of the deposition thin films was measured by four point probe collinear technique using 4200 model semiconductor characterization system (SCS). The morphology of thin films was studied using Scanning Electron Microscope. Absorption was assessed by UV-VIS spectrometer SEM.**



# CHAPTER ONE

## Introduction

### 1.1 Introduction

Semiconductor materials have electrical conductivity between a conductor and an insulator. It can vary over a wide range between a conductor and an insulator either permanently or dynamically. Semiconductor materials behave as insulators at absolute zero temperature, but show some electrical conductivity at room temperature. Two general classifications of semiconductors are the elemental semiconductors and the compound semiconductor materials. The elemental semiconductor materials are found on group IV and the compound semiconductor materials are formed from special combinations of group III and group V elements. Again the elemental and the compound semiconductor materials are combined known as intrinsic semiconductor. Intrinsic semiconductor can be doped with impurities that alter its electronic properties in a controlled way and then it becomes extrinsic semiconductor. Doping with a trivalent impurity turns it into a p-type extrinsic semiconductor. The electronic and optical properties of semiconductor materials are strongly affected by impurities such a way.

Semiconductor materials are important in electronic technology. Semiconductor devices, electronic components made of semiconductor materials are essential in modern consumer electronics, including computers, mobile phone, and various other applications. Silicon is to create most semiconductors commercially, but dozens of other materials are used as well. Because of their application in devices like transistors and therefore computers and lasers, the search for new semiconductor materials and the improvement of existing materials is an important field of study in materials and the improvement of existing materials and the improvement of existing materials science.

So in today's world more and more research is being conducted in the field of semiconductor technology. In recent few years saw a rapid development of high performance modern electronic devices, both in terms of new materials as well as well as new technologies. Primary semiconductors the dominated the last decade were silicon (Si) and group III-V materials such as Gallium Arsenide (GaAs) and Aluminum arsenide. Although these materials had extensive application, their narrow band gap (1.1eV for Si and 1.4eV for GaAs) limited their usage in most of today's high temperature and high power applications.

Semiconductor devices are usually fabricated in clean rooms, an ultra pure environment.

## **1.2 Characterization of Semiconductor Materials and Device**

Both the semiconductor materials and the fabricated devices are routinely characterized by the following three basic techniques:

- Electrical characterization
- Optical characterization
- Physical/ Chemical characterization

In our project a metal semiconductor contact and a metal sandwich structure were fabricated and then characterized electrically and physically. For electrical characterization there are two methods: two probe method and four probe method. The metal semiconductor contact characterized using the four probe method and the metal dye metal sandwich structure contact characterized using the four probe method. In the electrical characterization of the metal semiconductor contact four probe methods was used, since this method excludes the effect of stray capacitance and inductance of the copper wire. In case of the metal hue metal sandwich structure, the two probe method was used for its constructional structure.

The physical characterization of the contact was done very precisely using a highly sophisticated Profilometer named the dektak-150 Auto Profilometer, which has a resolution of 4 Å. Thus, any structure of few thousand atomic layers was detected and measured successfully using this Profilometer.

## **1.3 Objective of the Project**

- a. Study the fabrication process of thin film deposition using a thermal evaporator.
- b. Protocol development for the Dektak-150, Auto profilometer, Auto scriber and thermal evaporator.
- c. Characterization of thin film deposition
  - Electrical characterization using four probe method and
  - Physical characterization using dektak-150 auto profilometer.

## Chapter Two

# Deposition of Thin Film

### 2.1 Definition of Thin Film Deposition

The act of applying a thin film to a surface is thin-film deposition- any technique for depositing a thin film of material onto a substrate or onto previously deposited layers. "Thin" is a relative term, but most deposition techniques control layer thickness within a few tens of nanometers.

### 2.2 Classification of Thin Film

The act of applying a thin film to a surface is thin-film deposition- any technique for depositing a thin- film of material onto a substrate or onto previously deposited layers. Deposition techniques fall into two broad categories, depending on whether the process is primarily chemical or physical. They are:

#### 2.2.1 Chemical Deposition:

There are various types of chemical evaporation technique available. Some of them are mentioned below.

- **Chemical Vapor Deposition (CVD):** CVD is the process of chemically reacting a volatile compound of a material to be deposited with other gases to produce a non-volatile solid that deposits atomistic ally on a suitably placed substrate. In cases of metalorganic chemical vapor deposition (MOCVD) an organometallic gas is used.
- **Plasma Enhanced CVD (PECVD):** The introduction of plasma results in much enhanced deposition rates thus permits the growth of films at relatively low substrate temperature. It uses an ionized vapor, or plasma, as a precursor. Commercial PECVD relies on electromagnetic means (electric current, microwave excitation), rather than a chemical reaction, to produce a plasma.
- **Plating:** Often a solution of water with a salt of the metal needs to be deposited. Plating process may be used in this case. Some plating processes are driven entirely by reagents in the solution (usually for noble metals) but by far the most commercially important processes is electroplating.

### 2.2.2 Physical Deposition

Physical deposition uses mechanical, electromechanical or thermodynamic means to produce a thin film of solid. An everyday example is the formation of frost. Since most engineering materials are held together by relatively high energies, and chemical reactions are not used to store these energies, commercial physical deposition systems tend to require a low-pressure vapor environment to function properly; most can be classified as physical vapor deposition (PVD).

A thermal evaporator uses an electric resistance heater to melt the material and raise its vapor pressure to a useful range. This is done in a high vacuum, both to allow the vapor to reach the substrate without reacting with or scattering against other gas-phase atoms in the chamber, and reduce the incorporation of impurities from the residual gas in the vacuum chamber. Obviously, only materials with a much higher vapor pressure than the heating element can be deposited without contamination of the film. Molecular beam epitaxy is a particularly sophisticated form of thermal evaporation.

An electron beam evaporator fires a high-energy beam from an electron gun to boil a small spot of material; since the heating is not uniform, lower vapor pressure materials can be deposited. The beam is usually bent through an angle of  $270^\circ$  in order to ensure that the gun filament is not directly exposed to the evaporant flux. Typical deposition rates for electron beam evaporation range from 1 to 10 nanometres per second.

In molecular beam epitaxy (MBE), slow streams of an element can be directed at the substrate, so that material deposits one atomic layer at a time. Compounds such as gallium arsenide are usually deposited by repeatedly applying a layer of one element (i.e., gallium), then a layer of the other (i.e., arsenic), so that the process is chemical, as well as physical. The beam of material can be generated by either physical means (that is, by a furnace) or by a chemical reaction (chemical beam epitaxy).

Sputtering relies on a plasma (usually a noble gas, such as argon) to knock material from a "target" a few atoms at a time. The target can be kept at a relatively low temperature, since the process is not one of evaporation, making this one of the most flexible deposition techniques. It is especially useful for compounds or mixtures, where different components would otherwise tend to evaporate at different rates. Note, sputtering's step coverage is more or less conformal. It is also widely used in the optical media. The manufacturing of all formats of CD, DVD, and BD are done with the help of this technique. It is a fast technique and also it provides a good thickness control. Presently, nitrogen and oxygen gases are also being used in sputtering.

Pulsed laser deposition systems work by an ablation process. Pulses of focused laser light vaporize the surface of the target material and convert it to plasma; this plasma usually reverts to a gas before it reaches the substrate.

Cathodic Arc Deposition (arc-PVD) which is a kind of ion beam deposition where an electrical arc is created that literally blasts ions from the cathode. The arc has an extremely high power density resulting in a high level of ionization (30–100%), multiply charged ions,

neutral particles, clusters and macro-particles (droplets). If a reactive gas is introduced during the evaporation process, dissociation, ionization and excitation can occur during interaction with the ion flux and a compound film will be deposited.

**Electrohydrodynamic Deposition (electrospray deposition)** is a relatively new process of thin film deposition. The liquid to be deposited, either in the form of nano-particle solution or simply a solution, is fed to a small capillary nozzle (usually metallic) which is connected to a high voltage. The substrate on which the film has to be deposited is connected to ground. Through the influence of electric field, the liquid coming out of the nozzle takes a conical shape (Taylor cone) and at the apex of the cone a thin jet emanates which disintegrates into very fine and small positively charged droplets under the influence of Rayleigh charge limit. The droplets keep getting smaller and smaller and ultimately get deposited on the substrate as a uniform thin layer.

### **2.3 Basics of Thermal Evaporation**

A thermal evaporation uses an electric resistance heater to melt the material and raise its vapor pressure to a useful range. This is done in a high vacuum, both to allow the vapor to reach the substrate without reacting with or scattering against other gas phase atoms in the chamber, and reduce the incorporation of impurities from the residual gas in the vacuum chamber. Obviously, only materials with a much higher vapor pressure than the heating element can be deposited without contamination of the film. Molecular beam epitaxy (MBE) is a particular sophisticated form of thermal evaporation.

## Chapter Three

### Vacuum Technology

Any given space having molecular density less than this is said to be under "Vacuum" conditions. The technology dealing with the production of such reduced pressure environments using different scientific concept is known as vacuum technology.

It is interesting to know that we human beings continuously create vacuum during respiration and suction. Another natural Phenomenon is the decrease in Atmospheric Pressure with an increase in altitude.

Vacuum Technology is fundamental to a range of scientific explorations and technological Processes, extending from analyzing atomically clean surfaces at extremely low pressure of the order of  $10^{-11}$  Torr to freeze drying of food stuffs at relatively high pressure in the range of  $10^{-1}$  vacuum conditions are required for TV tube Productions, Vacuum furnaces, Vacuum coating, Semi conduction processing, Particle accelerators, space simulations etc.

In recent scientific literature, Pressure is expressed in torr and mbar which are non-coherent pressure units but widely used.

#### 3.1 Brief History of Vacuum Technology

The history of vacuum technology begins with Galileo (1564-1642) who was the first to conduct experiments to produce vacuum with a piston in cylinder for removal of water from mines later in 1643, Torricelli, an associate of Galileo, discovered the vacuum which is produced at the top of column of mercury when a long tube with mercury is inverted in a trough containing mercury. During the same period Pascal carried out several (1623-62) Vacuum physics experiments and was among the first to invent a barometer. In honor of these two great scientists, we now have two units of vacuum Torr and Pascal.

Guericke (1654) was the first to show the practically useful mechanical effects associated with the forces due to the pressure difference between vacuum & atmosphere. The inventions of the vacuum diode (1902) & triode (1907) and of the tungsten filament (1909), initiated the development of electron & x-ray tubes.

McLeod (1874) developed his primary gauge to measure pressures accurately with the subsequent development of Pirani's (1906) thermal conductivity gauge, Gaede's (1915) and Langmuir's (1916) diffusion Pumps, Buckley's (1916) hot cathode ionization gauges vacuum technology became matured.

After 1940 Vacuum Technology went through new development for nuclear research, metallurgy, thin film coating, freeze-drying etc. Further invention of the Bayard-Alpert gauge (1950) was an important step in pressure measurement in an ultra-high vacuum range. More recently, with the development of reliable closed cycle helium gas refrigerations, cry pumps with huge capacity are being developed by vacuum industries for space simulations and particle accelerator applications.

### 3.2 Types of Vacuum Techniques

There are lots of thin film deposition Techniques Where we have chosen Evaporation methods. Types of Evaporation methods are:

- Vacuum Evaporation
- Conventional Evaporation
- Electron beam Evaporation
- Molecular beam
- Reactive Evaporation

Classification of Vacuum ranges:-

➤ Low vacuum -----	760-25 Torr
➤ Medium vacuum -----	$25 \cdot 10^{-3}$ Torr
➤ High vacuum -----	$10^{-3} - 10^{-6}$ Torr
➤ Very high vacuum -----	$10^{-6} - 10^{-9}$ Torr
➤ Ultrahigh -----	Below $10^{-9}$ Torr

### 3.3 Applications of Vacuum Technology

Based on different physical characteristics of vacuum, we can achieve different industrial/scientific applications. For example, vacuum in a chamber allows us to have controlled sub atmospheric pressures, useful for several mechanical operations and controlled flow of gases in specified directions. The heat transfer rate is greatly reduced under vacuum conditions. The clean environment obtained by vacuum is very valuable in surface analysis experiments. The low molecular density in high vacuum conditions is utilized in several electrical and electronic devices. Indeed the range of applications of vacuum is now very wide, and is briefly covered in Tables 1.3 to 1.9 under different categories.

**Table 3.1 Evacuation/maintaining the Vacuum**

Industry	Product or scope
Electrical/ Electronics Scientific instruments Mechanical operations Refrigeration, cryogenic engineering Automobile Research and development	Gas-filled tubes, electron tubes, TV picture tubes. X-ray tubes, capacitors and transistors, rectifier tubes, cathode ray tubes, fluorescent tubes. Switching tubes, interrupters, photocells, gas discharge lamps, incandescent lamps. Maintaining the vacuum in electron microscopes. Mass spectrometers. Surface analysis systems, oscilloscopes, thermo flasks. Railway braking, industrial filtering, vacuum sniffers, vacuum cleaning, holding/lifting/transporting solids and liquids. Insulation of vessels and tanks for liquid petroleum gases. cryogenic liquids. Filling of air-conditioning, cooling and servo systems. Space simulation chambers, bubble chambers, fusion experiments, fusion reactors, particle accelerators, ion implantation systems, thermal insulation. Cryogenic experimentation.

**Table 3.2 Vacuum Impregnation**

Industry	Product or scope
Electrical Utility goodsPlastics	Cables, casting resin and oil insulated measuring transformers, motors, coils. Paper (in rolls), fibre boards and materials for pipe manufacture, fishing nets, wood, packaging materials, asbestos objects, crayon leads. Coils and windings.

**Table 3.3 Vacuum Degassing/Drying/Filing/Annealing/Brazing**

Industry	Product or scope
Electrical Chemical Refrigeration Automobiles Plastics Metallurgical	Waxed paper capacitors Regulators and thermometers with liquid-filled capillary tubes, oil circuit breakers. Spinning dopes or solutions, polymers like cable insulating compounds. Oils for capacitors and transformers. Plastics and susceptible chemicals. Refrigeration systems. Refrigeration oils. Brake fluid systems. Primary synthetic products. Casting resins.



	<p>High vacuum treatment of metals and their alloys susceptible to oxidation or gas.</p> <p>Vacuum cast forging ingots. Vacuum degassed alloyed steels and vacuum cast steel.</p>
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**Table 3.4 Vacuum Melting/Alloying/Casting/ Cleaning/Ageing/Sintering**

Industry	Product or scope
Electrical Plastics	Semiconductor production.
Metallurgical	Moulded parts (e.g.insulators) of casting resins, compound m moulding.
Nuclear engineering	Metals with minimized gas content and free of cavities, e.g. for hail bearings, wires, Hard (refractory) metals, magnets, and other powder metals susceptible to oxidation/or gas. Nuclear metals, ceramics, carbides.

**Table 3.5 Vacuum Coating (Evaporation/Sputtering)**

Industry	Product or scope
Scientific instruments Mechanical Automobile Utility goods and commodities Toys and jewellery Textiles Electronics	Thin film technology, molecular beam epitaxial systems, metal film resistors, metal coating layers, contacts, microcircuits, selenium rectifiers, thin-film deposition on oscillator crystals, photo-sensitive layers. Preparation of samples and specimens for electron microscopy. Hard and wear-resistant layers on tools, protective and decorative coatings on parts of precise mechanical apparatus. Electrically conductive front mirror coatings for fast defrosting headlamp and rear-light reflectors, horn push buttons. Decorative metal coatings on plastics, nameplates, ornamental signs, boxes, cups, vases, screw caps, buttons, etc. Plastic toys. jewellery like bracelets, ear clips, combs, medals, badges. Imitation gem-stones, fancy articles of wood. Protective metal coatings and iridescent decorative layers on textiles.

**Table 3.6 Vacuum Distillation/Sublirnatjon/Elimination of Solvents/ Concentration of Dissolved products**

Industry	Product or scope
Chemical Metallurgical Food processing	Waxes, high boiling chemicals, and intermediate products, plasticizers, mineral oils, vitamins. Explosives, metal powders, organic chemicals, of pure such as bismuth, selenium, zinc, antimony, magnesium, zirconium and Titanium.

Fruit Juices and luxury foods. The preserving vitamin contents and flavors.
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**Table 3.7 Vacuum Freeze Drying**

Industry	Product or scope
Chemical instruments research Scientific Medical Food processing	Yeasts, proteins, pharmaceutical products. Preparation of samples and specimens for electron microscopy. Laboratory research and studies, blood anatomy (macroscopic preparation for demonstration), histochemistryforpreparation location of tissue constituents), cytology of cell Strictures, surgery (storable grafting material), botany (cell nuclei andstudies),enzymebacteriology (bacteria, viruses and vaccines). Luxury foods with reduction in volume and weight well Conservation and packaging under inert gas atmosphere, preserving thenutritional values and tastes.

## Chapter Four

### Substrate Cleaning and Experimental Deposition

There are lots of technique for thermal deposition we have used thermal evaporations technique.

#### 4.1 Experimental Procedure

##### 4.1.2 Substrate Cleaning

Thermal Evaporation Technique is used for the thin film deposition in this experiment. A High Vacuum Thermal Evaporator system is used to produce a thin film on a glass substrate. Before proceeding with the technique, the surface must be cleaned clearly, as foreign materials may contaminate the coating process. We will use RCA cleaning in this experiment.

#### 4.2 RCA (Silicon Wafer) Cleaning

The famous RCA-2 clean (sometimes called "standard clean-2", SC-2), Developed by Werner Kern at RCA laboratories in the late 1960's, is a procedure for removing metal ions from silicon wafers. The decontamination works based on sequential oxidative desorption and complexation with  $H_2O_2-NH_4OH-H_2O$  (RCA-2). Typically, this is preceded by an RCA-1 clean (SC-1,  $H_2O_2-NH_4OH-H_2O$ ) to remove organic residues. In the process, it oxidizes the silicon and leaves a thin oxide on the surface of the wafer. This is a level-1 process and requires basic INRF safety certification. The use of dangerous chemicals requires that the user may not perform the process alone.

This process takes 30 minutes to complete in total.

Materials needed

- Hydrogen chloride
- Hydrogen peroxide
- Pyrex bath containers
- Hot plate

##### 4.2.1 Preparation

Setup time for this process is about 5 minutes. This process takes about 20 minutes to complete. The general recipe for RCA-2 cleanser is 6 parts water ( $H_2O$ ), 1 part 27% hydrogen chloride ( $HCl$ ), 1 part 30% hydrogen peroxide ( $H_2O_2$ ).

72 ml DI water  
12ml HCl  
12 ml H<sub>2</sub>O<sub>2</sub> (30%)

#### 4.2.2 Procedure [RCA-2]

Put 300 ml DI water in a Pyrex beaker, carefully add 50 ml HCl and then heat to  $70\pm 5^\circ\text{C}$  on hot plate. Remove from hot plate and add 50 ml H<sub>2</sub>O<sub>2</sub> (30%). Solution will bubble vigorously after 1—2 minutes, indicating that it is ready for use. Soak the silicon wafer in the solution for 10 minutes. When finished, remove the wafer and rinse with clean DI water.

#### 4.2.3 HF Etching

Hydrofluoric acid (HF) is used to remove native silicon dioxide from wafers. Since it acts quickly, one needs to only expose the wafer from a short time (“dip”). HF is a dangerous chemical and protective gear must be worn when using it, in particular, neoprene or thick nitrile gloves and eye protection must be worn. This process takes about 5 minutes to complete.

#### 4.2.4 Preparation

We need 2% solution of HF for this procedure. How to prepare a 2%  
Solution explain below-

480 ml water  
20 ml HF (49%)

Wear heavy protective gloves and protective eye gear. Add 480 ml water to polypropylene beaker then, add 20 ml HF. Never use a glass beaker with HF since HF attacks glass.

#### 4.2.5 Procedure

Soak the wafer for 2 minute in this solution. Remove the wafer and rinse in running DI water. Check the hydrophobicity by performing a wetting test. Pour a little DI water on the surface. If the water beads up and rolls off, the surface is hydrophobic and water will not wet. Since oxide is hydrophilic and pure silicon is hydrophobic, a non-wetting surface is clean of oxides. Blow dry with nitrogen and store in a clean, dry environment.

## 4.3 Experimental Procedure

Thermal Evaporation Technique is used for the thin film deposition in this experiment. A High Vacuum Thermal Evaporator system is used to produce a thin film on a glass substrate. The machine used is manufactured by Edwards. The model is Edwards 306.

### 4.3.1 Principle of Thermal Evaporation

Evaporation involves two basic processes: a hot source material evaporates and condenses on the substrate. It resembles the familiar process by which liquid water appears on the lid of a boiling pot. However, the gaseous environment and heat source (see "Equipment" below) are different.

Evaporation takes place in a vacuum, i.e. vapors other than the source material are almost entirely removed before the process begins. In high vacuum (with a long mean free path), evaporated particles can travel directly to the deposition target without colliding with the background gas. (By contrast, in the boiling pot example, the water vapor pushes the air out of the pot before it can reach the lid.) At a typical pressure of  $10^{-4}$  Pa, an 0.4-nm particle has a mean free path of 60 m. Hot objects in the evaporation chamber, such as heating filaments, produce unwanted vapors that limit the quality of the vacuum.

Evaporated atoms that collide with foreign particles may react with them; for instance, if aluminum is deposited in the presence of oxygen, it will form aluminum oxide. They also reduce the amount of vapor that reaches the substrate, which makes the thickness difficult to control.

Evaporated materials deposit non-uniformly if the substrate has a rough surface (as integrated circuits often do). Because the evaporated material attacks the substrate mostly from a single direction, protruding features block the evaporated material from some areas. This phenomenon is called "shadowing" or "step coverage."

When evaporation is performed in poor vacuum or close to atmospheric pressure, the resulting deposition is generally non-uniform and tends not to be a continuous or smooth film. Rather, the deposition will appear fuzzy.

### 4.3.2 Preparation of Deposition

In evaporative deposition, the source and substrate will be placed inside a vacuum chamber. With the pump turned off, vent the vacuum chamber by opening the vent-valve, and open the door of the chamber. Remove the sample holder and the bolts securing the evaporator flange to the vacuum chamber. Remove the evaporator, and place it upside down on the desk-holder. Close the door of the chamber and cover the flange with aluminum foil to keep dust out of the chamber. Cut a Si wafer on the slides and place a Si slide under the shadow mask and secure the mask and sample with the vacuum compatible screws. This slide will be the substrate on which you will grow a thin film of silver.

Check to see if the evaporation crucible is loaded; it should contain a bead of silver. If it does not, use the insertion tool to load a new pellet of silver into the evaporator.

Remove the old copper gasket from the opened flange of the vacuum chamber. Clean the conflate knife-edges on both the flanges with isopropanol. Connect the evaporator flange with the chamber. It is important at this stage to tighten the bolts evenly. First, insert all 6 bolts and tighten them by hand. Second, tighten the bolts, do this gently, turning each of the bolts by no more than one-eighth of a turn after you first feel resistance.

The system contains the essential elements typically required to obtain high vacuum. The most common and reliable systems utilize three pumping devices:

- The rotating mechanical pump
- The diffusion pump and
- The cold trap.

Other system components, such as valves and baffles, aid or control the action of these pumps. A graphic display of the pumping process is given below:

### **4.3.3 Deposition for Aluminum and Zinc Oxide**

When the pressure gets below about  $1 \times 10^{-5}$  tor, turn on the evaporation source current by switching the power supply and slowly 0.2 A/mm increase the current. The source will be heated until the aluminum (Al) and zinc oxide (ZnO) melts and begins to evaporate. The resulting vapor will then condense on all surfaces inside the vacuum chamber, including our substrate. Open the QSM shutter and record the deposition rate vs. time at regular intervals until the evaporation rate gets to the 3A/min range. Control the evaporation rate at constant current (10min) and record it in the lab-journal. Open the main shutter and deposit the film until the sensor indicates a film thickness 300 Å. Close the main shutter and slowly (0.2 A/mm) decrease the current down to 1 A and after that turn off the power Wait 20 min. with the pump turned off, vent the vacuum chamber by opening the vent-valve, and open the door of the chamber. Remove the sampleholder.

## 4.4 Equipment Used in Experiments

### 4.4.1 Mechanical Pump

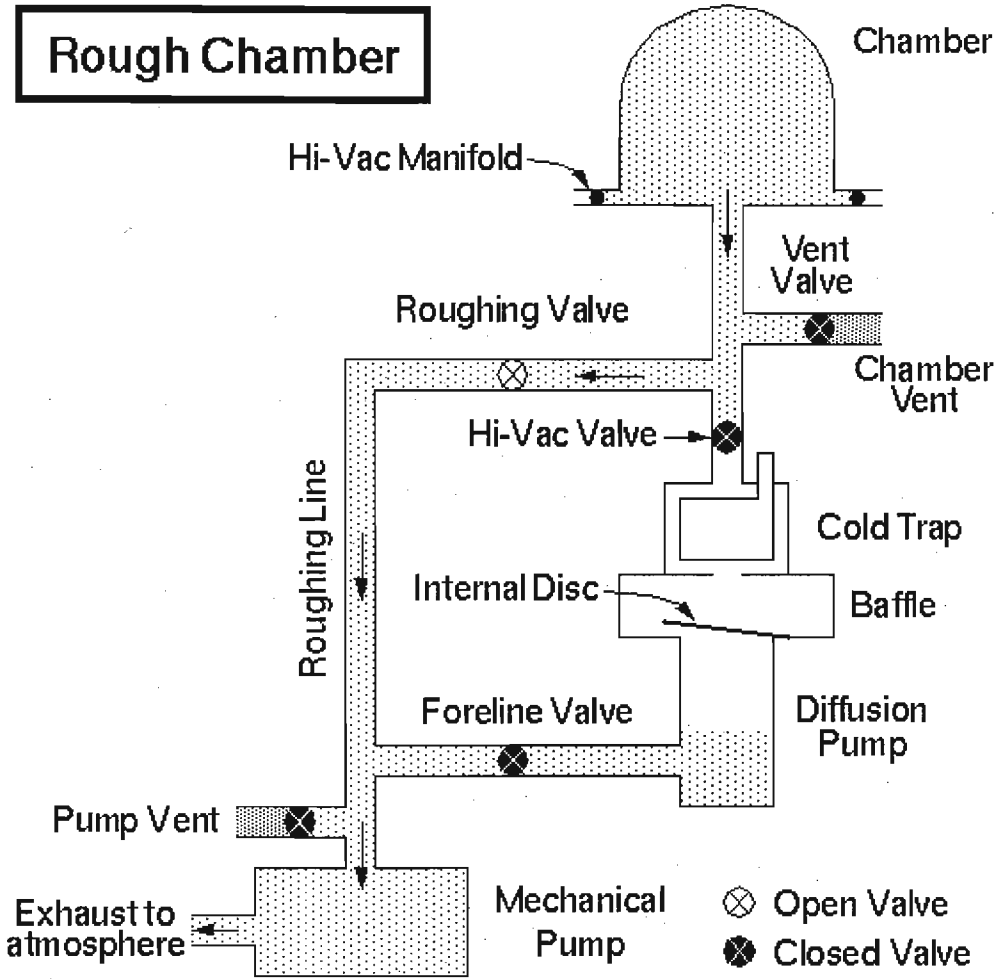


Figure 4.1 :Mechanical Pump.

A typical mechanical pump is shown schematically in Figure 2. Mechanical pumps physically "sweep" the air from the system, usually with a rotary device as shown. The rotor is eccentric to the pump cavity. The rotating vane (or sweep) is kept in contact with the walls of the pump cavity by means of a compression spring. Rotating vane, positive displacement pumps have large gas handling capacities, but cannot achieve high vacuum. They are used for two purposes: to remove ("rough") the bulk of the air from a system which is initially at atmospheric pressure, and, once this is accomplished, to "back" the diffusion pump, (see below), since a diffusion pump cannot exhaust against atmospheric pressure. Hence, mechanical pumps are often called roughing pumps, backing pumps or . In our illustration

(Figure 4.1) a single pump serves for both roughing and backing. In some applications, two pumps may be used.

#### 4.4.2 Diffusion Pump

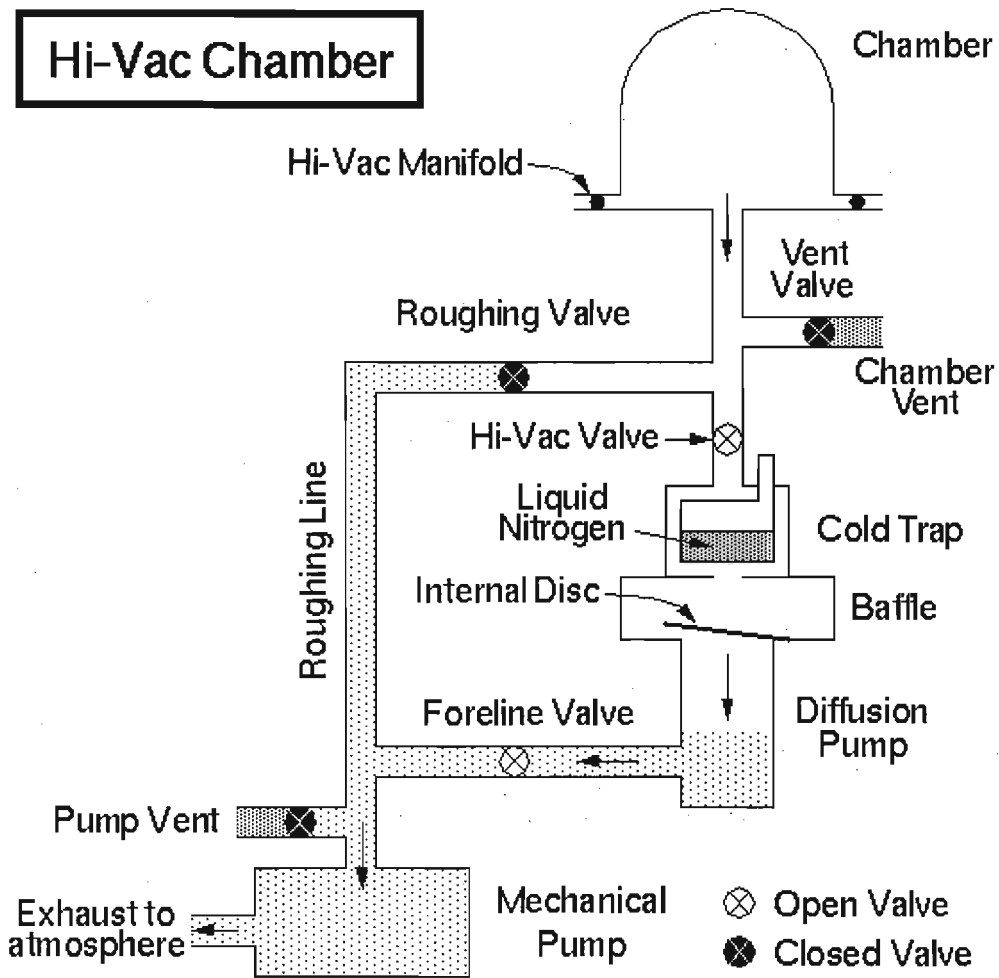


Figure 4.2 :Diffusion Pump

(Capable of reducing system pressure in the region of  $10^{-7}$  Torr). A diffusion pump has a maximum pressure against which it can exhaust; this is usually in the mtorr region. (The maximum exhaust pressure is also known as the "tolerable forepressure".) The mechanical pump noted above provides and maintains this exhaust pressure for the diffusion pump. Fast



pumping action is achieved through the use of high speed jets of oil vapor which collide with gas molecules and compress them in the direction of the mechanical pump (see Figure 3). (The term "jet" is used to refer to both the vapor stream and to the nozzles from which the vapor issues.) The oil pool at the bottom of the pump is heated, causing oil vapor to be forced up the jet stack. The vapor strikes the umbrellas, and is projected downward and outward through the nozzles of the jet stack. In passing through the narrow jets, the oil vapor flows at a velocity near that of sound. The high-speed vapor jet collides with gas molecules giving them a downward direction toward the foreline. The oil molecules condense on the walls of the pump, which are cooled either by an air stream or by water, and flow back to the bottom pool. Thus, a continuous cycle of vaporization, condensation, and revaporization takes place. Oil of very low vapor pressure is used in these pumps.

1 torr = 1 mm Hg

#### 4.4.3 Baffle (Water-Cooled)

Not all oil is contained and condensed by the diffusion pump. A small amount can escape toward the HiVac area of the system. This "backstreamed" oil is detrimental to the system. To contain it, a water-cooled baffle, shown in Figure 6, is located between the diffusion pump and the cold trap. Most back-streamed diffusion pump oil molecules are condensed on the internal Water-cooled baffle disc and returned to the diffusion pump in the form of liquid oil. The baffle helps keep the refrigerated surface of the cold trap operating at maximum efficiency.

#### 4.4.4 Cold Trap

(Shown with liquid nitrogen as refrigerant in Figure 6 ). This device reduces pressure by condensing, or freezing out, onto its cold surfaces, condensable vapors that may exist in the system. It also prevents oil vapor from the diffusion pump from diffusing back, or "backstreaming", into the system. By removing "condensables" such as water vapor, a trap actually serves as a pump. The trap is filled after the system has been evacuated to a pressure of less than one mtorr, when most of the condensable vapors have been pumped out of the system by the mechanical and diffusion pumps.

#### 4.4.5 Roughing Line and Valve

Exposure to atmosphere when at operating temperature will result in decomposition of diffusion pump oil. It is therefore necessary to employ a bypass line around a heated diffusion pump when evacuating a chamber from atmospheric pressure to a "rough" vacuum prior to connecting the chamber to the diffusion pump; hence, the terms "roughing line" and "roughing valve". The foreline valve and the hi-vac valve serve to isolate the diffusion pump,

the baffle, and cold trap from the object being roughed. During roughing the roughing valve is open. When roughing has been completed (at ~20 mtorr), the roughing valve is closed before the foreline valve and the hi-vac valve are opened. The manifold vent valve admits air to the port manifold to "break" the vacuum and make possible the removal of objects after they have undergone vacuum processing. The roughing valve and the hi-vac valve must be closed during this operation if the pumps on the vacuum system are still in operation. The mechanical pump vent valve serves to admit atmosphere to the roughing line, thus bringing the mechanical pump to atmospheric pressure. It is used when shutting down the system; the diffusion pump is turned off and allowed to cool, and then the mechanical pump is turned off. Bringing the stopped mechanical pump to atmospheric pressure prevents mechanical pump oil from being drawn back into the foreline. The hi-vac valve and the foreline are usually closed during the operation, thus maintaining the diffusion pump, cold trap and baffle under high vacuum. This assures a clean system when starting up at some later date.

## Chapter Five

### Measurement of Vacuum

#### 5.1 Pirani Gauge

In its simplest form, the Pirani Gauge is made up of a glass or metal tube enclosing a wire stretched along the tube's axis. This wire is heated by sending current through it.

At low pressure the energy balance may be expressed as

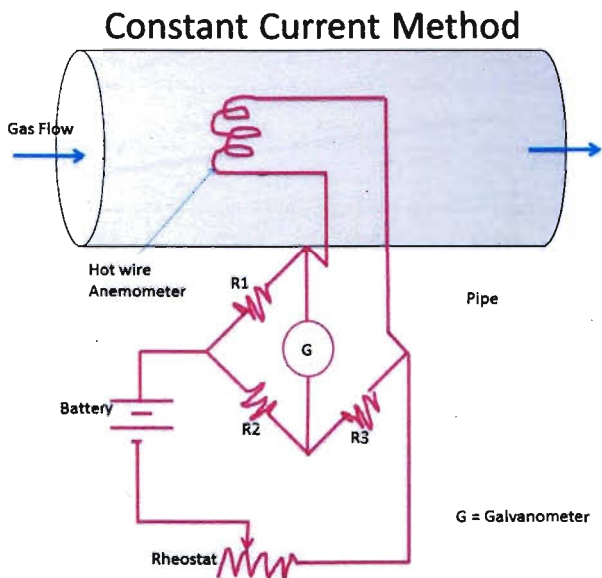
$$I^2 R (1 + \alpha \Delta T) = c P_x \Delta T + \sigma (T^4 - T_a^4) + b \Delta T \quad \text{-----(5.1)}$$

Where  $\Delta T$  is the difference in the filament temperature ( $T$ ) and the ambient temperature ( $T_a$ ).  $P_x$  is the pressure within the enclosure,  $c$ ,  $\sigma$  and  $b$  are proportionality constants,  $I$  is the current through the filament and  $R$  is the resistance of the filament at room temperature &  $\alpha$  is temperature coefficient.

The unknown pressure ( $P_x$ ) is obtained from

$$I^2 R (1 + \alpha \Delta T) = c P_x \Delta T + \sigma (T^4 - T_a^4) + b \Delta T \quad P_x = C \Delta T \dots \dots \dots (5.2)$$

The working of a Pirani gauge is demonstrated in fig (5.1)



**Fig 5.1:** Schematic diagram of Pirani Gauge

Most commercial gauge heads are made of steel tube or a glass enclosure and are provided with a tungsten filament. The measuring circuit is a simple Wheatstone bridge network. The network is formed by the four resistances  $R_x$ ,  $R_1$ ,  $R_2$  and  $R_3$ .  $R_x$  is made up of a material having a high temperature coefficient of resistance so that a small change in temperature will bring about a marked change in  $R_x$ , whereas  $R_1$  is fabricated from a material having a low

value of  $\alpha$  R2 & R3 are made of materials having low  $\alpha$  value and have the same value of resistance. These resistors are made of manganese or constantan wires.

Pressure is measured by keeping the filament Rx at constant temperature. As the unknown pressure changes, the amount of heat withdrawn from the filament changes and so does its temperature. Now the filament current is readjusted till the filament temperature is brought back to its

original value by nulling the out of balance current is measured with an ammeter (M) The filament current once calibrated ( $M_2$ ), gives the measured values of Pressure. Many commercial gauges omit  $M_2$  to monitor constant bridge current and prefer using electrical circuitry to provide a constant current source.

The pressures covered by these gauges are in the range  $1-10^{-4}$  Torr gives the calibrated Pressure versus micrometer reading for various vapors gases.

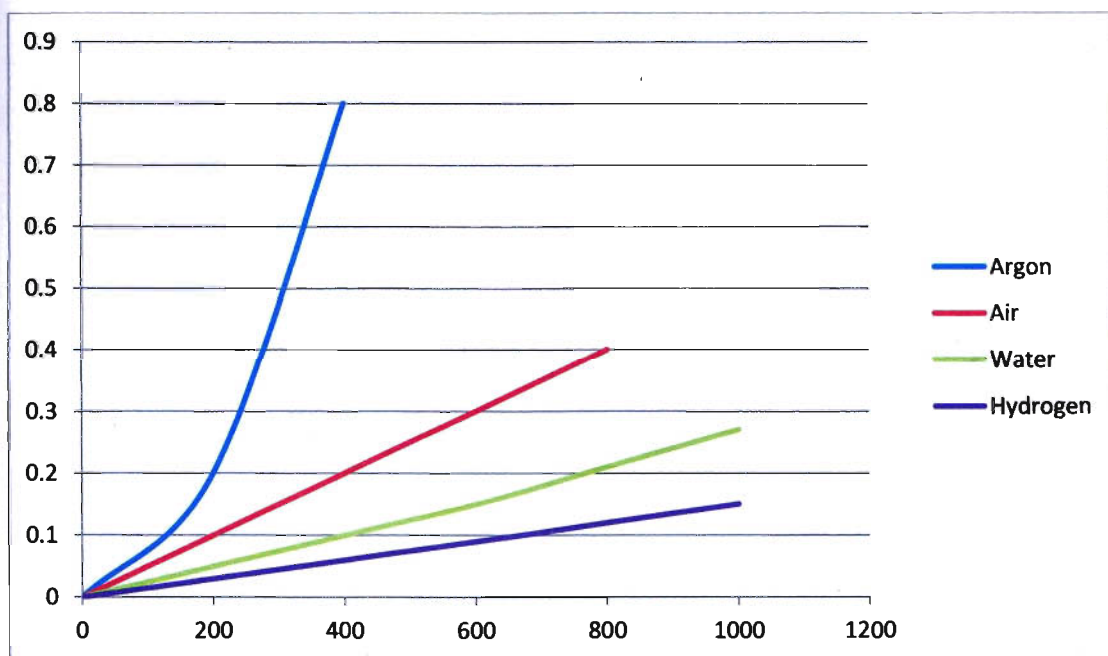


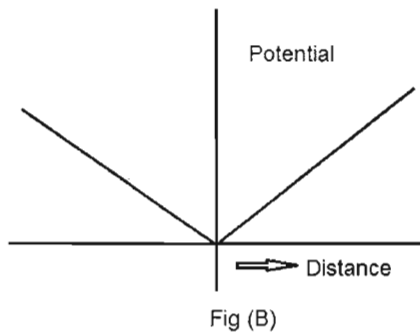
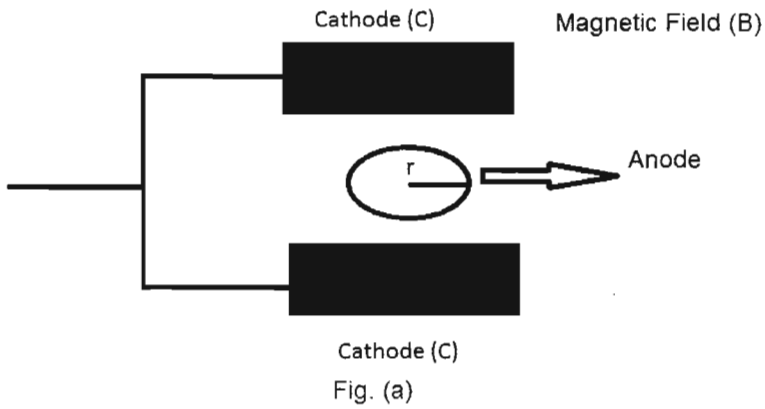
Fig 5.2 Pressure versus micrometer reading for various vapors gases

## 5.2 Penning Gauge

The basic geometry of the penning gauge with a tubular anode has been widely applied to the design of UHV pressure measurement. The manner in which the gauge operates can be seen from fig (5.3)

The anode A, is between two cathodes (c) and a magnetic field is produced by a horse shoe magnet. The magnetic field is perpendicular to the planes of the cathodes and the anode. It might appear at first glance, that the electrical and magnetic field in a penning gauge is parallel and not crossed. However, the potential on the axis of an operating penning discharge is depressed to near cathode potential by the electronic space charge.

Fig 2 (b) & (c) explain the potential and field configuration inside the Penning gauge tube. The general form of the calibration curve for a cold cathode discharge is shown in fig 5.3. the ion-currentpressure relation is non linear and is given by  $I^+ = kp^n$



**Fig 5.3** (a) penning gauge & electric and magnetic field configuration within the gauge.  
 5.3 (b) Variation of electric potential along the radius of the anode ring

Convection Gauge Sensor Voltage for a Constant Temperature Controller

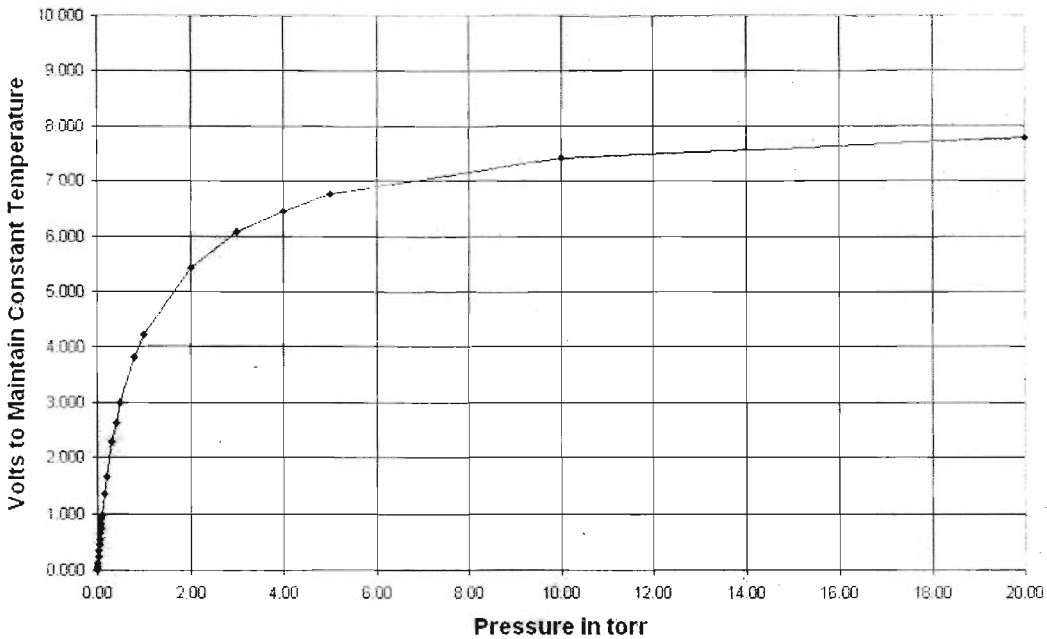


Fig 5.4 General form of the calibration curve of a Penning gauge

### 5.3 Leak Detection

In any vacuum system, however perfect it may be, there are points where a gas leak occurs. The vacuum system designer aims, therefore to keep the total leakage within acceptable limits. This is why leak detection and its quantitative estimation is so important in vacuum technology.

To detect a leak it is essential that the system be fitted with a gauge and valve with which the system can be isolated from the Pump.

### 5.4 Techniques for Leak Detections

Probe Test	Description	Minimum detectable Leak rate (torrliter/sec)
1. Pressure Test	Search gas pressurize greater than atmospheric	$1 \times 10^{-5}$
2. Vacuum Tests: Spark coil tests	A low power spark is made to find its way across a leak	1 to $10^{-3}$
3. Pressure gauge	Leaks are located by a change in output signal as the search gas passes through a	$10^{-5}$ to $10^{-10}$

	leak	
4. Halogen test	Leaks are located by a change in electron emission current from a hot surface exposed to the search gas containing halide molecules.	$1 \times 10^{-5}$
5. Mass spectrometric test	By mass spectrometric analyzing of search gas	$10^{-9}$ to $10^{-12}$

## Chapter 6

### Optical and Electrical Characteristic of Thin Film

#### 6.1 Measurement of Thickness of Thin Film

The Al film from the sources is coated on the glass substrate within a short period. The thickness of deposited Al thin film is recorded from the screen panel. The glass substrate is removed from the vacuum chamber. A uniform, silvery, mirror – like, fine coating of Al thin film is obtained on the glass substrate.

The Al coated substrate is then placed on the manual x-y sample positioning stage in Dektak-150 Surface Stylus profiler.(FIG) .This Surface Profiler profiles the surface topography, Surface waviness and Surface roughness in the nanometer range. The model, Dektak-150 is capable of measuring samples up to 6 inches in dimensions, 4 inches thick, steps below 100Å and provides a step height repeatability of 0.6 nm(6Å). Thus, it enables us to study the surface of roughness and waviness of Al film deposited on the substrate.

#### 6.2 Profilometer Analysis of Thin Film Structure

Since the Al thin film is developed therefore the thickness of layers can be measured easily using the Autoprofilometer .A single line scan program if the Profiometer makes it able to measure thickness.

Scan Parameters for Al thin film on Si wafer (VLSI lab at BAEC in Savar)

Scan type\ Standard scan

Stylus radius\12.5

Measuring range for first Al layer/1000um

Scan duration\10 sec

Resolution\0.333um/sample

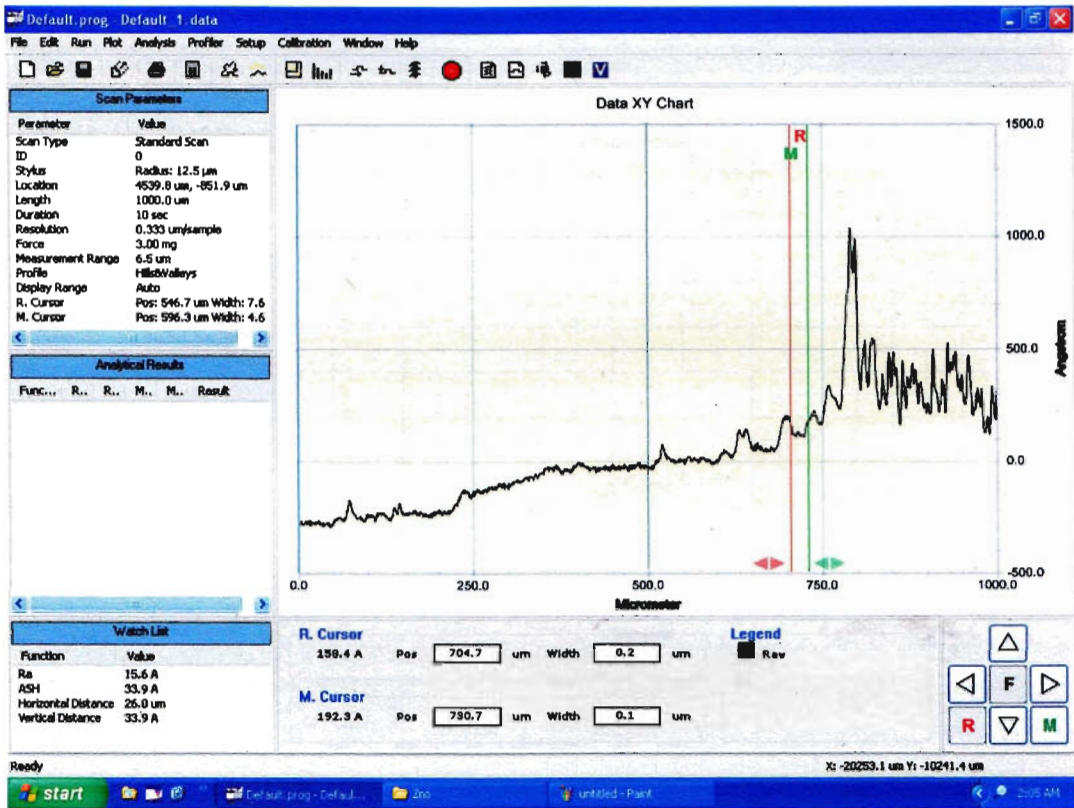
Stylus force\3.00mg

Display range\Auto



## Scan profiles\ Hills & Valleys

Using this parameter values a single line scan program in the autoprofilometer was run and the obtained profile reading is shown in the figure:



**Figure 6.1:** Dektak – 150 autoprofilometer reading for 1<sup>st</sup> Al thin film on Si wafer .  
The thickness of the thin film is obtained as : 33.9A

Scan Parameters for 2<sup>nd</sup> Al thin film on Si wafer (VLSI lab,Savar):

Scan type\ Standard scan

Stylus radius\12.5

Measuring range for first Al layer\1000 $\mu\text{m}$

Scan duration\15 sec

Resolution\0.222 $\mu\text{m}/\text{sample}$

Stylus force\3.00mg

Display range\ Auto

Scan profiles: Hills & Valleys

Using this parameter values a single line scan program in the autoprofilometer was run and the obtained profile reading is shown in the figure:

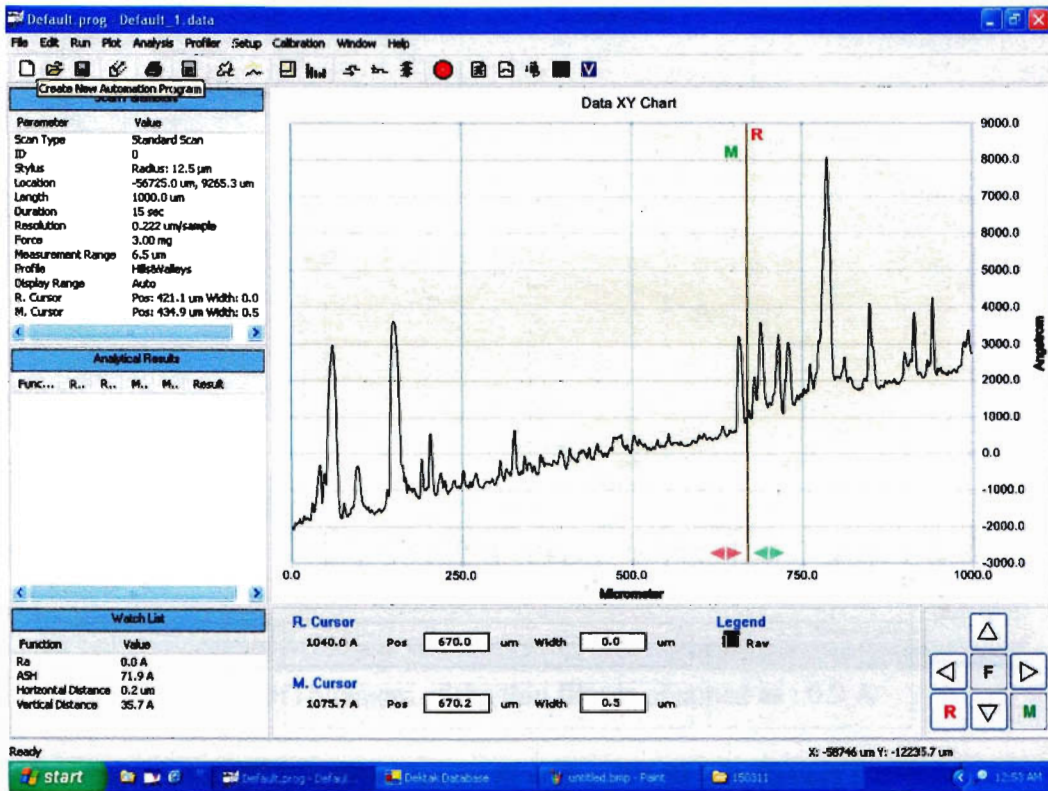


Fig 6.2: The Thickness of the thin film is obtained as : 35.7 A

Scan Parameters for 1<sup>st</sup>Zno Thin Film on Si Wafer (VLSI lab, Savar):

Scan type: Standard scan

Stylus radius\12.5

Measuring range for first Al layer\1000um

Scan duration\10 sec

Resolution\0.333um/sample

Stylus force\3.00mg

Display range\Auto

## Scan profiles\Hills & Valleys

Using this parameter values a single line scan program in the autoprofilometer was run and the obtained profile reading is shown in the figure:

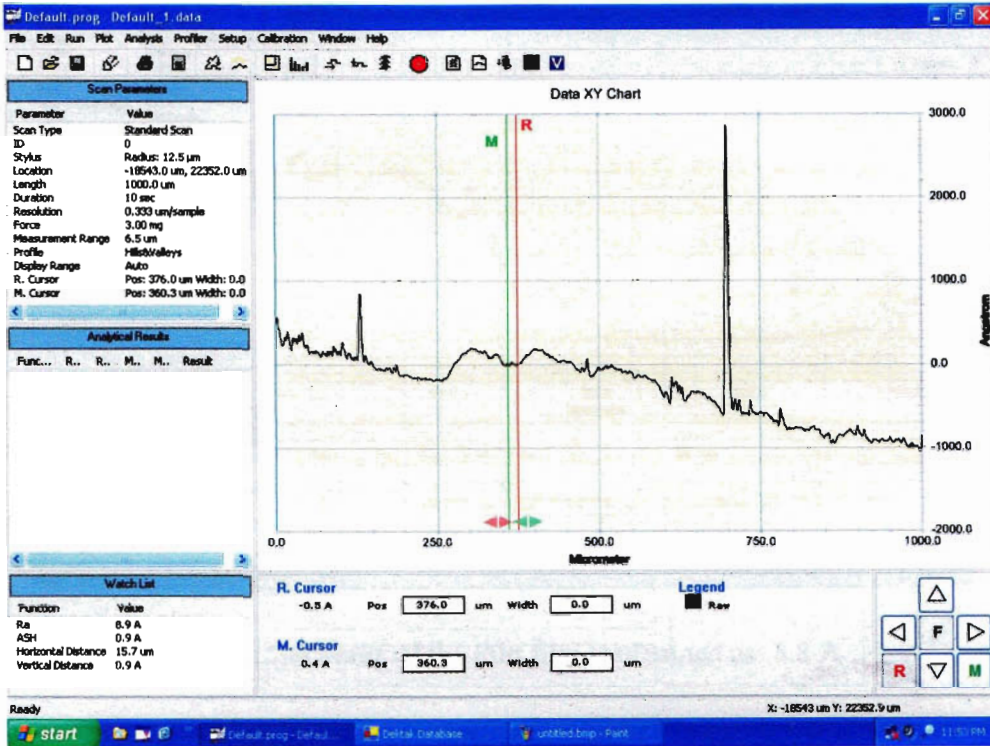


Fig 6.3: Thickness of the thin film is obtained as : 0.9 A

## Scan Parameters for 2<sup>nd</sup> ZnO thin film on Si wafer (VLSI lab, Savar) :

Scan type: Standard scan

Stylus radius\12.5

Measuring range for first Al layer\1000 $\mu\text{m}$

Scan duration\10 sec

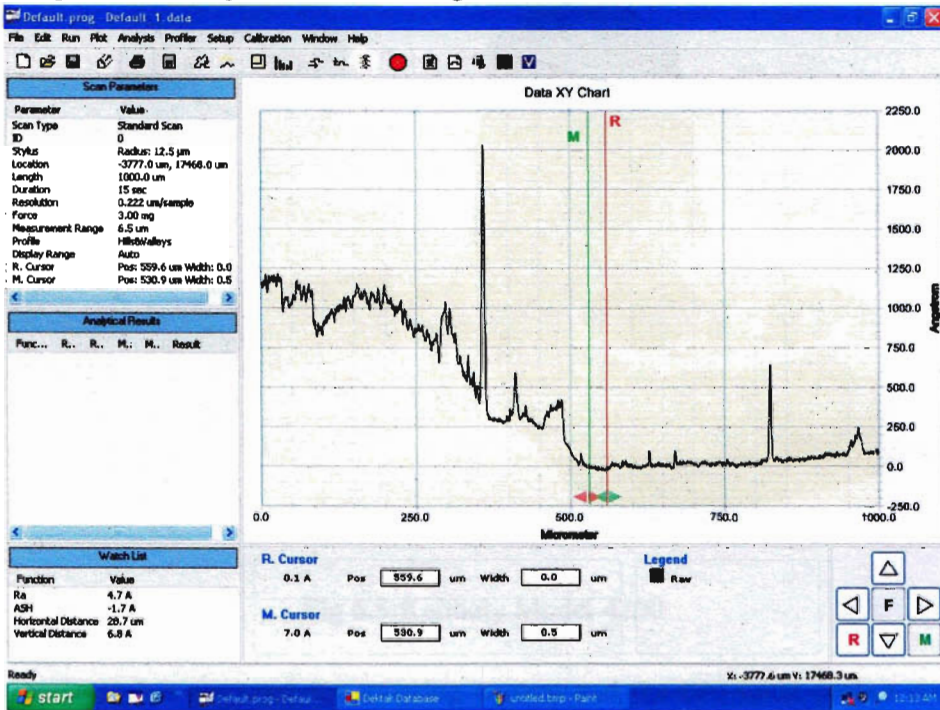
Resolution\0.333 $\mu\text{m}/\text{sample}$

Stylus force\3.00mg

Display range\ Auto

Scan profiles\ Hills & Valleys

Using this parameter values a single line scan program in the autoprofilometer was run and the obtained profile reading is shown in the figure:



**Fig6.4:** Thickness of the thin film is obtained as: 6.8 A

### 6.3 Measurement of Resistance of Thin Film

The electrical properties of the deposited Al film are then obtained through Four point probe Resistivity method using Keithley Model 4200 Semiconductor Characterization system, SCS (fig6.5) In four point colliner probe method, four equally spaced probes are placed in contact with the surface of materials of unknown resistance. In this case, the deposited thin film of Al. The probe array is placed in the center of the material under test.



**Fig 6.5:Keithely Model 4200**

The two outer probes are used for sourcing current and the two inner probes are used for measuring the resulting voltage drop across the surface of the sample. The volume resistivity of the film is calculated using

$$P = \frac{\pi}{\ln 2} \times \frac{V}{I} \times t \times k \dots \dots \dots (6.1)$$

Where,

p= Volume resistivity ( $\Omega\text{m}$ )

V= The measured voltage (volts)

I= The source current (Amperes)

t= The sample thickness (cm)

k\*= A correction factor based on the ratio of the probe to wafer diameter and on the ratio of wafer thickness to probe separation.

### 6.3 Findings

A piece of silicon wafer and glass substrate with thin film was taken as the sample. It is seen that the left portion on the graph (VLSI, LAB, AERC, SAVAR) is almost on the zero line, which represents the smoothness of the wafer surface except the presence of some dust particles. The spike on the glass substrate (Semiconductor TRC, University of Dhaka) represents the non-uniformity of the thin film and the presence of dust particles. The resistivity of the thin film is  $1.183 \times 10^{-6} \Omega\text{cm}$  and conductivity  $8.54 \times 10^{+5} \Omega\text{cm}$ .

Development of Protocols for Dektak-150 Autoprofilometer and RV-126 Auto scribe

## Chapter 7

# Development of Protocols for Dektak-150 Autoprofilometer and RV-126 Auto Scriber

### 7.1 Switch on steps for Dektak-150 Autoprofilometer

1. To switch on the computer,
2. To switch on the machine and
3. To switch on the Dektak software

This will take few minutes to show the sample-positioning window on the screen. During this period, the computer system will communicate with the Dektak.

### 7.2 Switch off steps for dektak-150 Autoprofilometer

1. To switch off the Dektak software,
2. To switch off the machine and
3. To switch off the computer.

### 7.3 Loading the Sample

First, a ceramic tweezers is used to hold the sample. Then carefully the sample is taken into the inside of the outer cover of the machine through the door and the sample is places at a suitable position on the stage. During placing the sample on the stage, one should be very careful so that the stage is not touched by one's finger or hand. After placing the sample, the stage should be leveled as described in the following section. The leveling of the stage involves two types of leveling:

### 7.4 Hardware Leveling of the Stage

#### 7.4.1 Mirror Leveling:

This is done to bring the stylus tip to the null position. The steps are :

- a. Tower down
- b. Stylus down
- c. Check whether the tip is on the null position or not
- d. If not, rotate the screws on the mirror of the instrument (there are to screws, one for moving the stylus along X-axis another for moving the stylus along Y-axis).

- e. During rotating the screws the position of the stylus tip on the screen is noticed and the screws are rotated so that the stylus tip is brought to the sample positioning window.
- f. Once the stylus tip is brought at the null position then stylus is moved up by the stylus up command.

#### 7.4.2 Stage Leveling:

The purpose of stage leveling is to make the tilt of the sample zero. To perform it is needed to take a single line scan (procedure for taking single line scan in section 4.1.5) Now the working steps are:

- a. An optically flat surface on the sample is chosen (this may be as small as a few hundred micron).
- b. Single line scan on this location is performed (scan length should be less than the length of optically flat surface).
- c. While the scan is running, the wheel below the stage is rotated clockwise for positive tilt and anticlockwise for negative tilt. Here, positive tilt means the upward movement of the scanning trace and negative tilt means the vice versa.
- d. While wheel is rotated one should be very much careful so that the stage is not touched in anyway.
- e. Taking a single line scan, the slope is to be computed and noticed so that it approaches a value close to zero during the next scan. The acceptable value for the slope should contain at least four zeros after the decimal point.

Similarly, a number of scans are taken and the slope is computed for each scan. This way for certain scan the slope becomes almost zero and then the stage is said leveled and the sample is now tilted to zero.

#### 7.5 Single Line Scan- Standard Scan

A Single scan routine is an automation program that runs only a single scan routine containing a single set of parameters. Analytical functions, software leveling, and other functions can be included in a single scan routine.

Typical Uses of Single Scan Routine:

If the system includes the X-Y auto stage, a single scan routine is created whose automation program is copied and used as the basis for setting up an automation program that runs multiple scans with different parameters. If the system includes the X-Y auto stage, a single scan routine is created whose automation program is copied and used as the basis for setting up



an automation program for a map scan. During a map scan, the sample moves under the stylus in two horizontal axes while also tracking the vertical differences in height.

### 7.5.1 Single Scan Setup Steps

To set up and run a single scan routine:

1. Including any desired analytical functions, an automation program is created.
2. The scan location and length are defined.
3. The scan is Run and the following adjustments are made:
  - \* Set the cursor location and bandwidths for leveling and analytical functions.
  - \* If necessary, manually level the stage.
  - \* If necessary, run additional test scans and perform manual or software leveling.

### 7.5.2 Programming a Single Line Scan Routine:

Scan type: standard

Stylus radius: 12.5  $\mu\text{m}$

Measuring range; desired measuring range (1000 $\mu\text{m}$ , 2000 $\mu\text{m}$ , 5000 $\mu\text{m}$  etc)

Scan duration: may be 60 sec, 120 sec, 180 sec etc.

Resolution: it is set automatically

Stylus force: 10-15 mg for hard material and 0.03-0.5mg for soft material.

Display range: auto

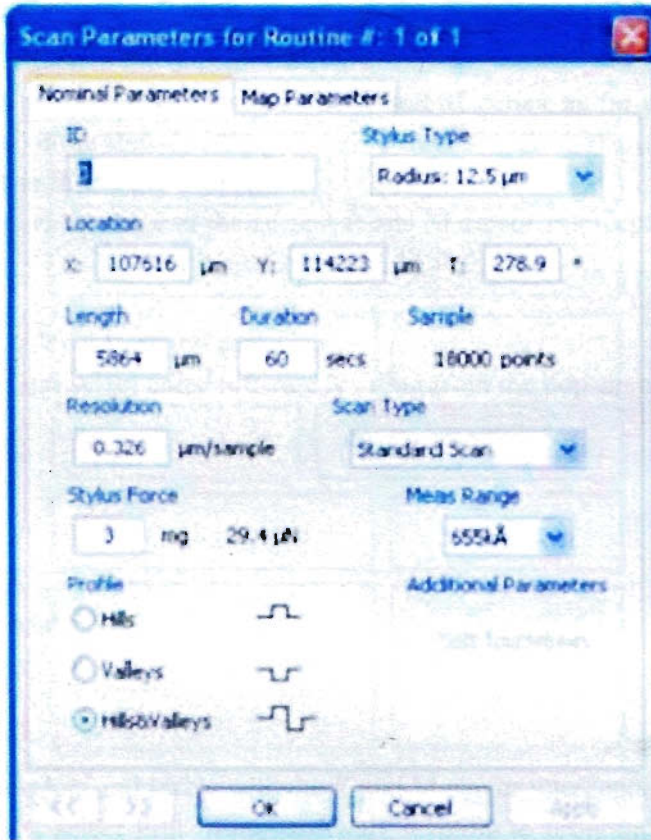
Scan profiles: Hills, valleys or Hills & valleys.

### 7.5.3 Running a Single Line Scans Routine :

To run a single scan routine, click the run currently active scan routine icon. Alternatively, select run> scan from the menu bar or the F4 key on the keyboard. The following sequence of events occurs after you initiate a single scan:

- The stylus lowers onto the sample surface and nulls. After a brief pause, the scan begins by doing a short backlash (to relieve the system backlash). As the stylus scans across the sample, the full scale profile trace plots on the scaled grid in real time over live video of the stylus.
- Next, the Data plot window appears with the scaled grid superimposed over the camera view pane of the stylus and sample.
- After the scan is complete, the stylus lifts off the surface, and the stage returns to the location where the scan originated. The image displayed on the monitor resembles the one shown in figured 4.5.3 which includes the following elements :
  - The scan profile
  - Reference/ measurement cursor functions
  - List of user set scan parameters
  - List of analytical results (if any analytical functions were selected for this scan)

- A watch list, which provides real time monitoring of ASH (delta average step height) and Ra, as well as horizontal distance and vertical distance.



**Fig7.1.** Scan Parameter dialog box

#### 7.5.4 Setting Cursor Locations and Bandwidths

The red reference (R) cursor and green measurement (M0 cursor in the Data plot window define the portion of the profile trace for leveling of performing analytical functions. You can adjust the bandwidth at each cursor to average the data points within the cursor's bandwidth. This is useful for software leveling and average step height measurements.

#### 7.6 Performing Software Leveling

Software leveling allows the system to quickly and automatically level the profile trace. You must software level the stage in order to obtain accurate step height measurements and accurate readings from analytical functions. Software leveling sets the reference and measurement cursors at zero to establish a reference for measurements.

Software leveling is performed between the M cursor and R cursor by means of common sense, that is, the M cursor and R cursor are placed at the same level considering the sample and the two ends of the trace being at the same real level. This is shown in figure.

To software level a trace in the data plot window:

1. In the Data plot window, position the R and M cursor as far apart as possible along the baseline of the step.
2. Do one of the following :
  - \* Click the level the trace at the current R and M cursor Intercepts along the baseline of the step.
  - \* Press F7
  - \* Select plot > level from the menu bar
  - \* Right click and select enter software leveling from the pop-up menu.
    - Replot F6
    - Level F7
    - Zero F8
    - Data type
    - Clear Bands
    - Default Bands
    - Band widths..
    - Boundaries

The profile trace re-plots and levels with the R and M cursor intercepts at zero.

3. Alternatively, select Edit> Enter software leveling to program software leveling into the scan routine to level the trace automatically at the conclusion of each scan.

## **7.7 RV 126 Autoscriber**

### **7.7.1. Protocol Development of the Autoscriber:**

An Autoscriber is a machine that is used to scribe a glass, ceramic plate of semiconductor wafer and that has the ability to control the applied force at a fixed value. Generally, an autoscriber contains a diamond cutter whose scribe force is uniform across the total sample.

### **7.7.2 Principle of Cutting**

Cutting a glass or ceramic plate means interrupting the normal surface tension of the material creating a grooved zone of high tension at the line where the break is required. This can be achieved by a very shallow but even and continuous groove.

The most important cutting parameters are:

- Operating angle of tool,
- Scribing force
- Cutting speed.

The operating angle of the scribe is of primary importance for good result. A proper adjustment is normally achieved when the diamond holder is in-line with the scribe arm.

### 7.7.3 Starting the Scriber :

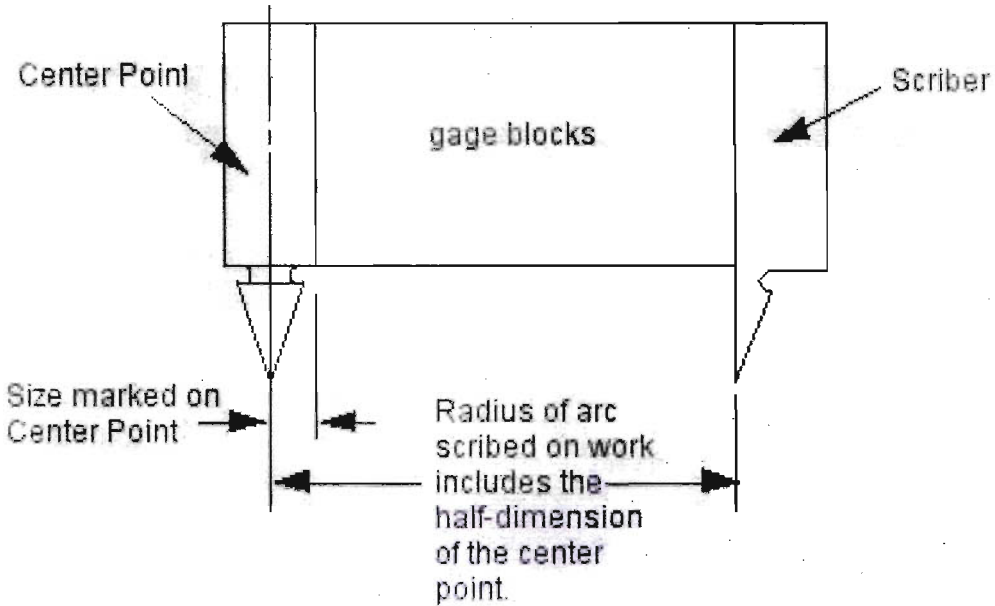


Fig 7.1: Schematic diagram of a scriber

- Filing the alcohol container
- Adjustment of the diamond tool
- Setting the contact point

Lay a trial substrate on the ruler. Pull scriber head forward against the stop and press down. The diamond point must exactly touch the edge of the plate, but never drop in front of it. Adjust with screw "A".

\* Setting the scriber force

The force of the diamond tool depends on the hardness and composition of the surface to be cut. It is very important for the quality of the groove and the life of the tool. According to the wear and tear of the diamond, the scribing force can be set between 0.25 N and 8 N by screw "K".

- Setup the zero line

This setup is necessary after every release of the diamond tool.

Set the right stop (7) at about 10mm and fasten it.

Cut piece of trial substrate.

Release the zero-stop (8) with the attached Allen-wrench. Don't shift the right stop (7) by doing so.

Tighten the screw of the zero-stop (8).

By shifting of the right stop (7) every chosen piece cut width can be set up exactly with a caliper square.

#### **7.7.4. Working with the Autoscriber Scribing a Silicon Wafer:**

We worked with an autoscriber at BAEC, Savar

Following was placed below the diamond tip and a large piece of silicon wafer was scribed successfully, which is shown in figure 4.6. The value of force was set to 8N. After scribing the wafer successfully, it was placed between two glass slides in such a way that the scribe on the wafer was just at the edge of the glass slides. Then the scribed segment of the wafer was press softly and then at a small pressure the wafer was broken along the scribbling line. The wafer which was scribed and cleaved is shown in fig. along with cleaved portion.

## Chapter 8

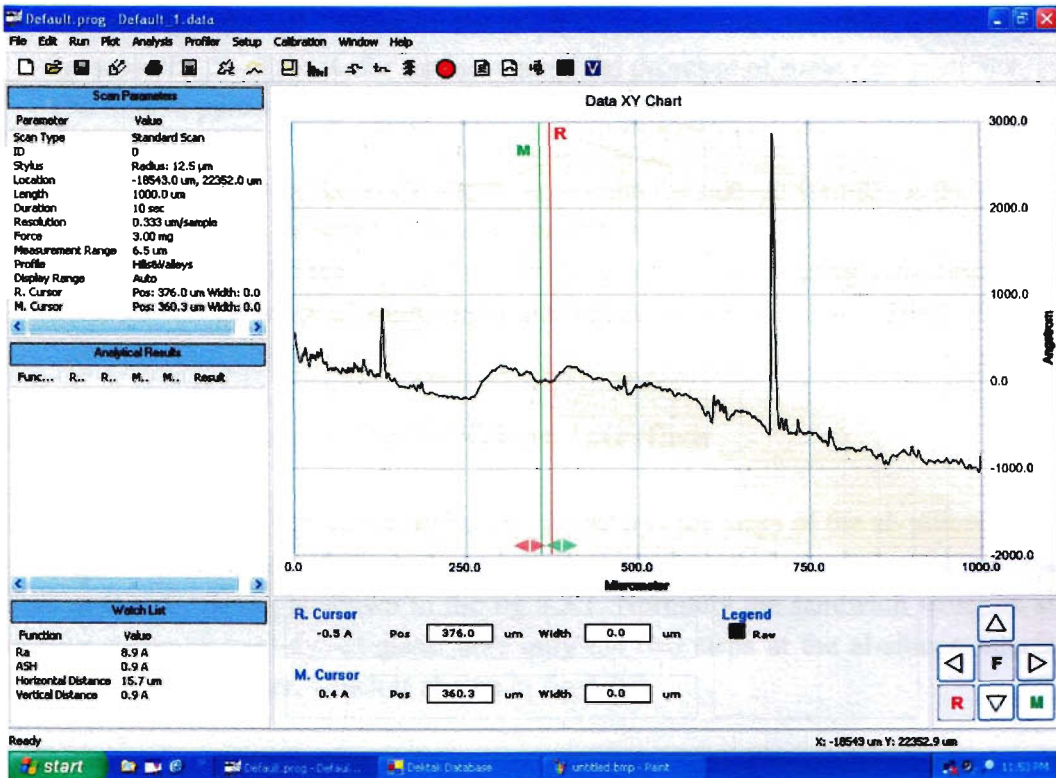
# Studying the Geometry of Samples with Dektak-150 Autoprofilometer

### 8.1 Observing Non-Uniformity of a Glass Slide

Normally a glass slide is smooth to look at. But it might be a non-uniform structure with a non-smooth or rough surface. The surface might be a rough one with roughness at least at angstrom and even at micron level. Here the surface roughness of a glass slide was observed using dektak-150 auto profiler and the reading as obtains is shown in fig. 8.1.



Fig8.1: Observing non-uniformity of a glass slide for aluminum.



**Fig8.2:** Observing non uniformity of a glass slide for zinc oxide.

In the Autoprofilomete reading in the figure:

- It is seen that the glass slide surface is not smooth at angstrom level at all.
- Rather it is seen to have an average roughness of 12.2A for aluminum and 20.8A for zinc oxide.
- It is also observed that the molecules in the glass slide are not uniformly distributed. The curvy structure of the graph results from this non- uniformity of molecules distribution in the glass slide.

As seen on the graph, there are some spikes in both upward and downward direction. The upward spike may have been resulted from the presence of dust particles on surface and the downward spike on the right portion of the graph might have been resulted from the absence of some molecules first few layers of the surface.

## 8.2 Observing Film Layer on a Silicon Wafer

A piece of silicon wafer with an oxide layer on the surface was taken as the sample. The purpose of this reading was to observe the surface roughness of the silicon wafer along with the oxide layer. The surface might be a rough one with roughness at least at angstrom and even at micron level. Here the surface roughness of a silicon wafer with oxide layer on it was observed using dektak-150 auto profiometer reading in the figure.

- It is seen that the left portion on the graph is almost on the zero line, which represents the smoothness of the wafer surface except the presence of some dust particles.
- The middle portion of the graph represents the presence of an oxide layer on the wafer surface.
- The smaller spike in the oxide region represents the non-uniformity of the oxide layer formation and the presence of smaller dust particles.
- The largest spike on the right portion of the graph, upon crossing the oxide layer may be due to the presence of some larger dust particles on the wafer surface.

### 8.3 Detecting the Step at the Al-Silicon Interface

The purpose of this Profilometer analysis was to observe the steps of the al- silicon interface. The image of Al-Silicon and Zno silicon interface as taken with the help of a microscopic camera at BAEC, Savar, is shown in the fig 5.3.1. Normally the sandwich structure should have four steps: glass-al-dye-al-glass. here only the two steps at the al-silicon interface at both sides of the dye layer, which is shown in fig-5.3.2.

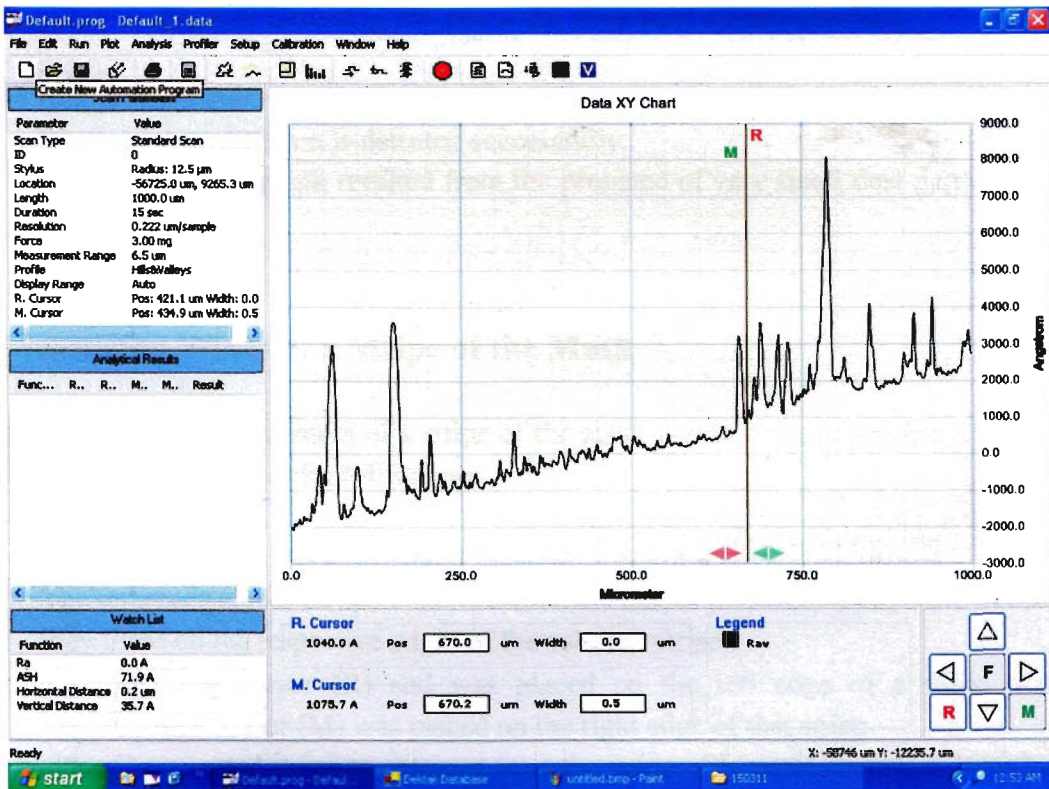


Fig 8.3: Detecting the step at the aluminum silicon interface



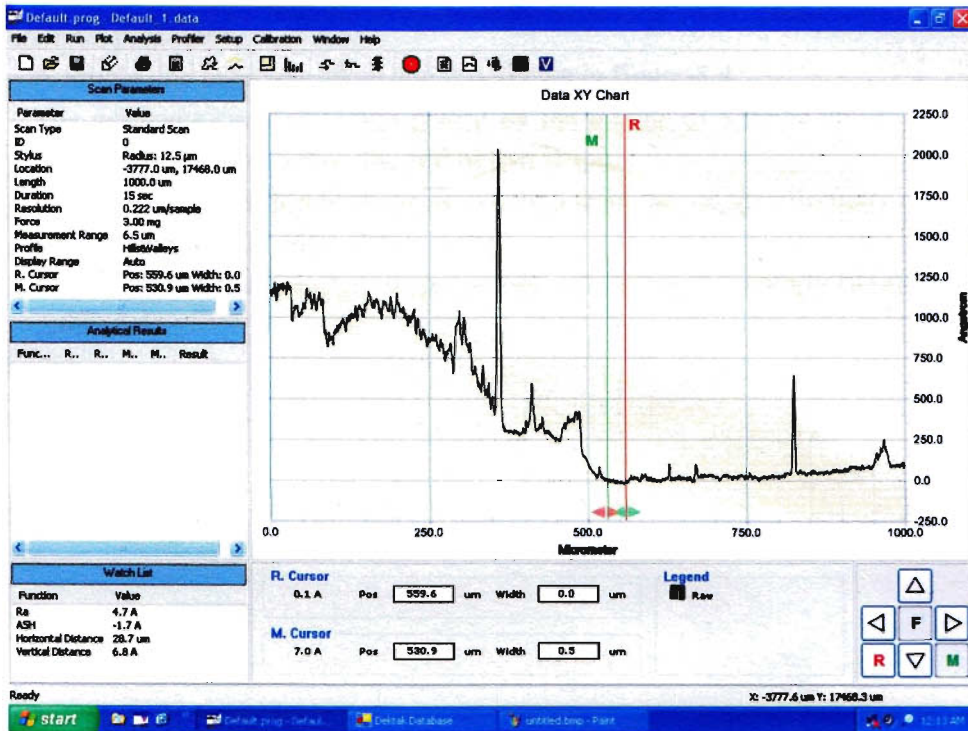


Fig 8.4: Detecting the step at the aluminum silicon interface

In the Autoprofilometer reading in the figure-

- It is seen that al-silicon is detected successfully.
- The spike on the graph resulted from the presence of very small dust particles on the structure.

## 8.4 Measuring Width of a Stripe of the Mask

The purpose of the measurement of a stripe of the mask was to observe a stripe and measure its width with the help of Autoprofilometer.

Measurement procedure-

- A single line scan was taken and it is shown figure 8.4
- The reference cursor (R) and the measurement cursor (M) were made narrower so that they fitted on the respective edges of the selected stripes.
- The reference cursor (R) and was placed on the left edge of a stripe and the measurement cursor (M) was placed on the right edge of that stripe.

The width of the selected stripe as obtained from the autoprofilometer reading is 2689.2  $\text{\AA}$ .

## 8.5 Measuring Depth of a Stripe of a Section of the Mask

The purpose of the measurement of a stripe of the mask was to observe a stripe and measure its depth with the help of Autoprofilometer.

Measurement procedure-

- A Single line scan was taken and it is shown in figure-8.4
- The reference cursor (R) was placed on the middle of a stripe and the measurement cursor (M) was placed on the end of that stripe.
- The depth of the selected stripe as obtained from the autoprofilometer cursor (M) was placed on the right edge of that stripe.
- The width of the selected stripe as obtained from the autoprofilometer reading is 2689.2 Å.

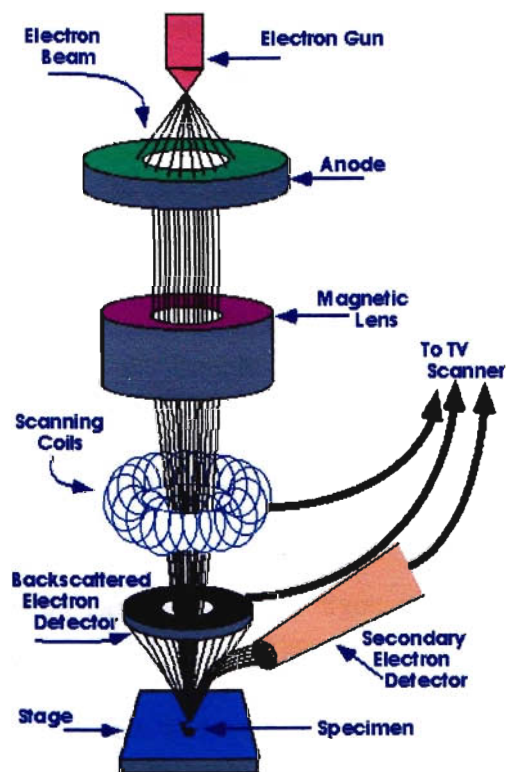
## Chapter 9

# Scanning Electron Microscope

SEM stands for scanning electron microscope. Scanning Electron Microscope is a microscope that uses electrons instead of light to form an image. Since their development in the early 1950's, scanning electron microscopes have developed new areas of study in the medical and physical science communities. The SEM has allowed researchers to examine a much bigger variety of specimens.

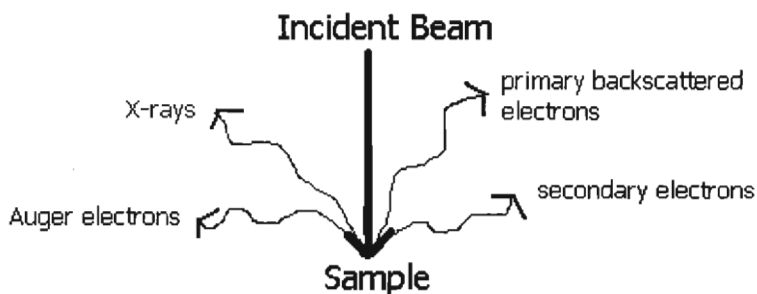
The Scanning Electron Microscope has many advantages over traditional microscopes. The SEM has a large depth of field, which allows more of a specimen to be in focus at one time. The SEM also has much higher resolution, so closely spaced specimens can be magnified at much higher levels. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today.

### 9.1 How does a SEM Work



**Fig 9.1: working procedure of SEM**

The SEM is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, which focus the beam down toward the sample. Once the beam hits the sample, electrons and X-rays are ejected from the sample.



**Fig 9.2:** Beams hit in the sample

Detectors collect these X-rays, backscattered electrons, and secondary electrons and convert them into a signal that is sent to a screen similar to a television screen. This produces the final image.

## 9.2 How is a Sample Prepared

Because the SEM utilizes vacuum conditions and uses electrons to form an image, special preparations must be done to the sample. All water must be removed from the samples because the water would vaporize in the vacuum. All metals are conductive and require no preparation before being used. All non-metals need to be made conductive by covering the sample with a thin layer of conductive material. This is done by using a device called a "sputter coater."

The sputter coater uses an electric field and argon gas. The sample is placed in a small chamber that is at a vacuum. Argon gas and an electric field cause an electron to be removed from the argon, making the atoms positively charged. The argon ions then become attracted to a negatively charged gold foil. The argon ions knock gold atoms from the surface of the gold foil. These gold atoms fall and settle onto the surface of the sample producing a thin gold coating.

### **9.3 Strengths and Limitations of Scanning Electron Microscopy (SEM)**

#### **Strengths**

There is arguably no other instrument with the breadth of applications in the study of solid materials that compares with the SEM. The SEM is critical in all fields that require characterization of solid materials. While this contribution is most concerned with geological applications, it is important to note that these applications are a very small subset of the scientific and industrial applications that exist for this instrumentation. Most SEM's are comparatively easy to operate, with user-friendly "intuitive" interfaces. Many applications require minimal sample preparation. For many applications, data acquisition is rapid (less than 5 minutes/image for SEI, BSE, spot EDS analyses.) Modern SEMs generate data in digital formats, which are highly portable.

#### **Limitations**

Samples must be solid and they must fit into the microscope chamber. Maximum size in horizontal dimensions is usually on the order of 10 cm, vertical dimensions are generally much more limited and rarely exceed 40 mm. For most instruments samples must be stable in a vacuum on the order of  $10^{-5}$  -  $10^{-6}$  torr. Samples likely to outgas at low pressures (rocks saturated with hydrocarbons, "wet" samples such as coal, organic materials or swelling clays, and samples likely to decrepitate at low pressure) are unsuitable for examination in conventional SEM's. However, "low vacuum" and "environmental" SEMs also exist, and many of these types of samples can be successfully examined in these specialized instruments. EDS detectors on SEM's cannot detect very light elements (H, He, and Li), and many instruments cannot detect elements with atomic numbers less than 11 (Na). Most SEMs use a solid state x-ray detector (EDS), and while these detectors are very fast and easy to utilize, they have relatively poor energy resolution and sensitivity to elements present in low abundances when compared to wavelength dispersive x-ray detectors (WDS) on most electron probe microanalyzers (EPMA). An electrically conductive coating must be applied to electrically insulating samples for study in conventional SEM's, unless the instrument is capable of operation in a low vacuum mode.

## 9.4 Result And Discussion

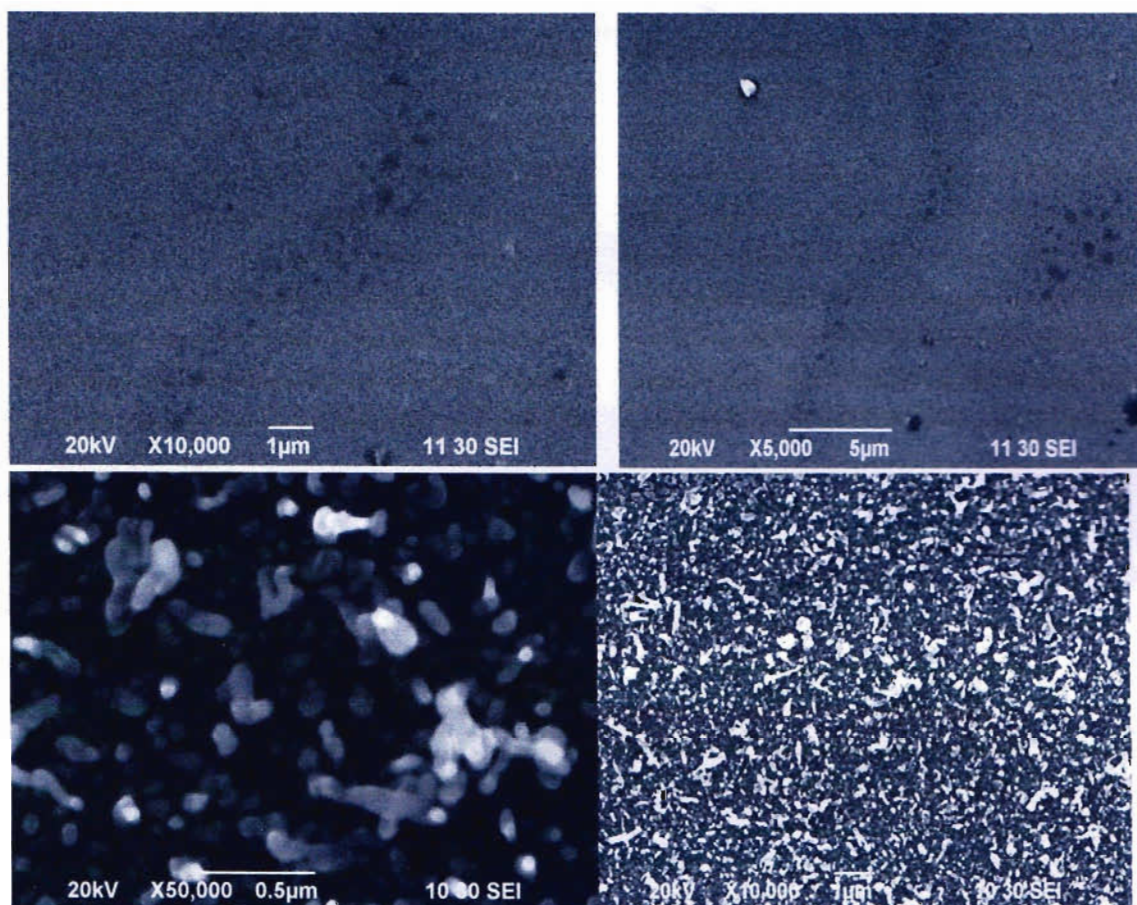


Fig 9.3:(a,b) view of SEM image for Al and (c,d) view of SEM image for ZnO

Figure 9.3(a,b) shows the SEM obtained for Al thin film.

Al powders were evaporated in thermal evaporation to set the thin film on glass substrate.

As obtained Al powder were very smooth morphological texture .It is very difficult to distinguish between the images taken respectively for magnification of 5000 and 1000.It could be assumed that higher resolution for the SEM images available at higher magnification that is  $> 50,000$ .

Figure 9.3(c,d) shows the SEM obtained for ZnO thin film

Grains of ZnO thin films are evident in both the images as shown in figure (c) and (d).From this SEM micrograph ,it has been seen that ZnO formed with excellent vertical alignment,uniformity and packing density.It is not so difficult to distinguish between the images taken respectively for magnification of 10000 and 50000.large grains are found on fig (c) and its very clear while in fig (d) grains are not clear.

## Chapter Ten

### Conclusion

#### 10.1 Summary

In this paper the facilities required for the micro chip fabrication for the semiconductor devices are described with specification. Silicon wafer has chosen for study and detailed fabrication process for metal-semiconductor contact has been developed at the semiconductor Technology Research Center, University of Dhaka and Bangladesh Atomic Energy commission, Savar.

The physical characterization of the contact was done very precisely using highly sophisticated Profilometer named the Dektak -150 auto Profilometer, which has a resolution of 4Å. Thus any structure of few thousand atomic layers was detected and measured successfully using this Profilometer.

The Profilometer analysis of metal semiconductor contact is studied using Dektak-150 Autoprofilometer. Different stripes of a section of a mask were analyze and studied along with their depths and widths. Protocols for Dektak -150 Autoprofilometer, thermal evaporator and autoscriber were also developed in this project along with specification and working principle.

In VLSI lab in Savar, it is seen that the left portion on the graph is almost on the zero line, which represents the smoothness of the wafer surface except the presence of some dust particle. On the otherhand in semiconductor lab the spike on the glass substrate represents the non-uniformity of the thin film and the presence of dust particles.

There is specific range of length on the film which gives the uniform; uncontaminated surface film. This provides the region of optimum performance of the film. The four point probe technique provides the resistivity at various points on the film surfaces.

Scanning electron microscope (SEM) is one of the most versatile instruments available for the examination and analysis of the microstructure morphology and chemical composition characterizations. This thesis paper gives a quick review of fundamentals of scanning electron microscopy using Al and ZnO as examples. Al and ZnO thin films were uniform and big grains were found in ZnO thin film. It can help SEM users and researchers to study about fundamental of SEM in a short time.

## 10.2 Future Work

Future fossil fuel scarcity and environmental degradation have demonstrated the need for renewable, low carbon sources of energy to power an increasingly industrialized world. Solar energy, with its extraordinary resource base is one of the most feasible long term options for satisfying energy demand. The present work aims to develop through characterization method for thin films fabricated through a physical vapor deposition (PVD) process. It provides a platform for material analysis and opportunity to adapt a typically high throughput manufacturing method to make high quality thin film. Future work may apply this procedure to fully characterize other materials and eventually lead to solar cell fabrication.



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